Part 2: Quality Assurance Project Plan

Remedial Investigation, UMore East
Dakota County, Minnesota

June 15, 2011

BARR

A Project Management
A1 Title and Approval Sheet

PART 2: Quality Assurance Project Plan
Remedial Investigation
UMore East
Dakota County, Minnesota

June 15th, 2011

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Principal-in-Charge — Date: 6-15-11

Jim Eidem, Barr Engineering Co.
Project Manager — Date: 6-15-11

Kristen Schimpke, Barr Engineering Co.
Field Team Leader, Site Safety Officer — Date: 6/17/11

Andrea Nord, Barr Engineering Co.
Quality Assurance Officer — Date: 6/15/11

Gary Krueger, Minnesota Pollution Control Agency
Project Manager — Date: 6/17/11

for Dave Scheer, Minnesota Pollution Control Agency
QA Officer/Project Hydrogeologist — Date: 6/17/11

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Laboratory Project Manager

6/15/11
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Laboratory QA Officer

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Date

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Laboratory QA Officer

6/14/2011
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06/14/2011
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Kristen Schimpke, Barr Engineering Co. – Consultant Field Team Leader

Andrea Nord, Barr Engineering Co. – Consultant Project Quality Assurance Officer

Gary Krueger, Minnesota Pollution Control Agency – Agency Project Manager

Dave Scheer, Minnesota Pollution Control Agency – Agency Project QA Officer/Hydrogeologist

William Scruton, Minnesota Pollution Control Agency – Agency QA Coordinator

Terri Olson, Legend Technical Services – Laboratory Project Manager

William Dahl, Legend Technical Services – Laboratory QA Officer

Karen Sellers, TestAmerica – Laboratory Project Manager

Douglas Weir, TestAmerica – Laboratory QA Officer

Acronym List

AES	Agricultural Experiment Station
AOC	Area of Concern
ARAR	Applicable or Relevant and Appropriate Requirements
AST	Above Ground Storage Tank
bgs	Below ground surface
COC	Constituent of Concern
DBP	Di-n-butyl Phthalate
DNT	Dinitrotoluene
DNR	Department of Natural Resources
DPA	Diphenylamine
DRO	Diesel Range Organics
DQO	Data Quality Objective
EIS	Environmental Impact Statement
EPA	Environmental Protection Agency
FSI	Focused Site Inspection
GPS	Global Positioning System
GOW	Gopher OrdnanceWorks
GRO	Gasoline Range Organics
HRL	Health Risk Limit
kg	Kilograms
MDA	Minnesota Department of Agriculture
MDH	Minnesota Department of Health
mg	Milligrams
MPCA	Minnesota Pollution Control Agency
msl	Mean sea level
PA	Preliminary Assessment
PACM	Potentially Asbestos Containing Material
PAH	Polycyclic Aromatic Hydrocarbon
PID	Photoionization Detector
PPM	Parts per Million
Phase I	Phase I Environmental Site Assessment
REC	Recognized Environmental Condition
RI	Remedial Investigation

SAP	Sampling and Analysis Plan
SSI	Supplemental Site Inspection
SLV	Soil Leaching Value
SOC	Site of Concern
SRV	Soil Reference Value
SVOC	Semi-volatile Organic Compound
TBC	To-be-considered Criteria
UMA	UMore Mining Area
UMore Park	University of Minnesota Outreach, Research and Experimentation Park
ug	Micrograms
USACE	U.S. Army Corps of Engineers
USDA	U.S. Department of Agriculture
UST	Under Ground Storage Tank
VOC	Volatile Organic Compound

A4 Introduction

This QAPP presents the organization, objectives, functional activities and specific Quality Assurance (QA) and quality control (QC) activities required for the Remedial Investigation (RI) that will be used to characterize soil and groundwater at sixty-nine Sites of Concern (SOCs) located in the UMore East Remedial Investigation (RI) project area in, Dakota County, Minnesota (the Site). This QAPP is intended to encompass the Site sampling and analysis activities associated with this investigation. This QAPP also describes the protocols that will be followed for sampling, sample handling and storage, chain of custody, laboratory analysis, and field analysis.

All QA/QC procedures will be in accordance with applicable professional technical standards, EPA requirements, government regulations and guidelines, and specific project goals and requirements. This QAPP was prepared by Barr Engineering Co. (Barr) in accordance with EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, Quality Assurance Division, United States Environmental Protection Agency, March 2001.

A5 Project Organization

The project organization is shown on Figure 1. The qualifications of the main project team members are included in Appendix A.

A5.1 University of Minnesota Project Manager

The University of Minnesota (University) is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. University Project Manager Janet Dalgleish will be responsible for reviewing all project deliverables and documents. She has overall authority and responsibility for technical aspects of the project. The University project manager will provide the major point of contact and control for matters concerning the project. The responsibilities of the University project manager include:

- Acquiring and applying resources as needed to ensure performance within budget and schedule constraints;
- Directing all project activities
- Reviewing all project deliverables, and oversee all project strategies
- Representing the project team at meetings and public hearings

The University project manager may delegate most of these responsibilities to competent individuals.

A5.2 Barr Engineering Co. (Barr)

At the direction of the University, Barr has responsibility for oversight of the site investigation. Overall project implementation management will be provided by Barr. The various quality assurance and management responsibilities of key project personnel are defined below.

A5.2.1 Barr Principal in Charge

Allan Gebhard is the Barr Principal in Charge. The Principal in Charge has overall responsibility for verifying that the project meets the established objectives and quality standards. The Principal in Charge is the primary contact for contractual issues and for resolving quality concerns. The Principal in Charge has responsibility for overall project implementation management and product quality.

Specific responsibilities of the Principal in Charge include:

- Leading and overseeing on behalf of Barr contract negotiations and development, including contract terms, scope, schedule, and budget
- Involvement with overall management, administration, and technical aspects of project
- Providing independent quality review and validation for technical and contractual issues
- Monitoring client satisfaction for contract work
- Resolving contractual or quality issues

A5.2.2 Barr Project Manager

Jim Eidem is the Barr Project Manager. Barr's Project Manger is the University's primary contact for technical issues and day-to-day communication of scope, schedule, and budget progress. Barr's Project Manager is the primary Barr contact for project direction. The Barr Project Manager has the day-to-day and overall responsibility for managing implementation of the project, including quality management and overall project quality. The Barr Project Manager is responsible to the University for implementing the project. The Barr Project Manager's primary function is to see that technical, financial, and scheduling objectives are achieved successfully. The Barr Project Manager will provide the major point of contact for the University on matters concerning implementation of the project. Specific responsibilities of the Barr Project Manager include:

- Involvement on behalf of Barr in contract negotiation of scope, schedule and budget
- Direct involvement in day-to-day administration, budgeting, coordination, scheduling, and other managerial tasks
- Matching project needs with staff abilities and informing all team members of the project requirements
- Overall direction of technical aspects of the project including defining project objectives and developing a detailed work plan and schedule
- Primary responsibility for project quality, including technical correctness and completeness, contract compliance, and budget and schedule compliance
- Notifying the University of necessary scope, schedule, or budget modifications
- Reviewing and recommending subcontractors

- Communicating directly with the University Project Manager

A5.2.3 Barr Quality Assurance (QA) Manager

Andrea Nord is the Barr Quality Assurance Manager. The role of the Quality Assurance Manager is to provide an independent review of the product and the process to see that the work meets quality standards. She is responsible for auditing the implementation of the QA program in conformance with the requirements of this quality assurance plan, and the demands of specific project tasks. Specific responsibilities of the QA Manager include:

- Providing QA technical assistance to project staff
- Reporting on the adequacy, status, and effectiveness of the QA program on a regular basis to the Barr Project Manager
- Data validation
- Laboratory audits
- Initiation, tracking and review of QA/QC corrective actions
- Distribution of the approved SAP and subsequent revisions

A5.2.4 Barr Field Manager

The role of the Barr Field Manager is to oversee the entire investigation and the collection of all analytical samples following the procedures outlined in this QAPP and associated work plans. The Barr Field Manager, in conjunction with the Barr project manager and with approval of the University project manager, has the authority to stop or change work activities to ensure compliance with project goals and data quality objectives.

Additional Barr Field Manager responsibilities include;

- Direct all field staff to ensure the data collection and field activities meet the objectives of the RI.
- Along with the Barr Project Manager, make field decisions related to the scope and schedule of the RI.

A5.2.5 Barr Field Staff

The role of the field staff is to collect all analytical samples following the procedures outlined in this QAPP and associated work plans. Additional field staff responsibilities include;

- Collect and calibrate all necessary field equipment prior to beginning an assessment
- Oversee investigation contractors to ensure proper techniques are being followed and the desired information is being collected
- Assure quality objectives are met during sample collection, packaging, documentation, and shipping
- Documenting field activities to assist subsequent data analysis interpretation and reporting
- Complete and submit all necessary paperwork and forms to the project team

A5.2.6 Barr Health and Safety

Karen Stoller, an industrial hygienist, is the Barr Health and Safety Manager. The role of the Health and Safety Manager is to oversee all aspects of job safety and develop Project Health and Safety Plans (PHASP) which provide guidelines, requirements, and procedures intended to help protect the health and safety of all employees of Barr and Barr's subcontractors who will participate in the field work in accordance with the provisions of 29 CFR 1910.120, Hazardous Waste Operations and Emergency Response.

A5.3 Legend Technical Services, Inc.

Legend Technical Services, Inc. (Legend) located in St. Paul, Minnesota will conduct the physical preparation and chemical analyses of the majority of the analytical samples specified in the associated work plan. Independent quality assurance will be provided by the Legend Project Manager and QA Officer prior to release of all data to Barr. A copy of Legend's Quality Assurance Manual (QAM) is provided in Appendix B.

Legend is certified though the Minnesota Department of Health's (MDH) Environmental Laboratory Certification Program (when applicable for the target analytical list in Table 1).The perchlorate and nitrocellulose are not certifiable tests under the MDH program. Any additional subcontracted laboratories will be certified by the MDH to perform analysis in Minnesota, where applicable, and will follow the processes and procedures as outlined in this QAPP.

All laboratory reports will be prepared and submitted to Barr following each sampling event electronically. Specific roles of the Legend personnel are outlined below.

A5.3.1 Legend Project Manager

Terri Olson is the Legend project manager. The Legend Project Manager is responsible for verifying that the assessment data meets the established objectives and Legend's quality standards. The Legend Project Manager is responsible for technical quality control and project oversight. The Legend Project Manager's primary function is to see that technical, financial and scheduling objectives are achieved successfully. The Legend Project Manager will be the primary laboratory contact for administrative, financial and scheduling considerations. Specific responsibilities include:

- Acquiring and applying technical and corporate resources as needed to perform the work within budget and schedule constraints
- Developing and meeting on-going project and staffing requirements
- Reviewing all work performed by Legend to verify its quality and completeness and review subcontractors data to verify its completeness, responsiveness and timeliness

A5.3.2 Legend Project QA Officer

William Dahl is the Legend QA Officer for the laboratory. The Legend project QA Officer will remain separate and distinct from other project-related duties. The QA Officer is responsible for maintaining conformance to project QA requirements, Legend's Corporate QA/QC Plan, EPA and related methodologies. The following lists several specific duties of the Legend QA Officer:

- Tracking validation data and ensuring adherence to published guidelines
- Determining if the levels of QA/QC are being achieved
- Certifying the level of QA/QC for each analytical project
- Maintaining QA/QC procedures
- Initiating and overseeing internal audits
- Initiation and implementation of corrective actions

A5.4 Test America, Inc.

Test America, Inc. (TestAmerica) located in West Sacramento, California will conduct the physical preparation and chemical analyses of nitrocellulose as specified in the associated work plan. Independent quality assurance will be provided by the TestAmerica Project Manager prior to release of all data to Barr. A copy of TestAmerica's Quality Assurance Manual (QAM) is provided in Appendix B. Specific roles of the TestAmerica personnel are outlined below.

A5.4.1 TestAmerica Project Manager

Karen Sellers is the TestAmerica project manager. The TestAmerica Project Manager is responsible for verifying that the assessment data meets the established objectives and TestAmerica's quality standards. The TestAmerica Project Manager is responsible for technical quality control and project oversight. The TestAmerica Project Manager's primary function is to see that technical, financial and scheduling objectives are achieved successfully. The TestAmerica Project Manager will be the primary laboratory contact for administrative, financial and scheduling considerations. Specific responsibilities include:

- Acquiring and applying technical and corporate resources as needed to perform the work within budget and schedule constraints
- Developing and meeting on-going project and staffing requirements
- Reviewing all work performed by TestAmerica to verify its quality and completeness and review subcontractors data to verify its completeness, responsiveness and timeliness

A5.3.3 TestAmerica Project QA Officer

Douglas Weir is the TestAmerica QA Officer for the laboratory. The TestAmerica project QA Officer will remain separate and distinct from other project-related duties. The QA Officer is responsible for maintaining conformance to project QA requirements, TestAmerica's Corporate QA/QC Plan, EPA and related methodologies. The following lists several specific duties of the TestAmerica QA Officer:

- Tracking validation data and ensuring adherence to published guidelines
- Determining if the levels of QA/QC are being achieved
- Certifying the level of QA/QC for each analytical project

- Maintaining QA/QC procedures
- Initiating and overseeing internal audits
- Initiation and implementation of corrective actions

A5.5 Minnesota Pollution Control Agency (MPCA)

The MPCA project manager and quality assurance reviewer must approve all quality documents prior to beginning any field work. Specific responsibilities for the MPCA project manager and the MPCA quality assurance reviewer are addressed in the following sections.

A5.5.1 MPCA Project Manager

Gary Krueger is the MPCA Project Manager. Specific responsibilities include;

- Direct review and approval of the QAPP and work plans
- Technical consultation with the University Project Manager and/or the Barr Project Manager
- Review all progress reports detailing completed work
- Review all final reports

A5.5.2 MPCA Quality Assurance Coordinator

William Scruton is the MPCA QA Coordinator. Specific responsibilities include;

- Review and approve QAPP
- Assist in review of all sampling protocols
- Conducting external performance and system audits of laboratory and field activities.
- Reviewing and evaluating analytical field and laboratory procedures

A6 Project Definition and Background

This QAPP has been prepared on behalf of the University by Barr to describe the environmental investigations of the eastern two-thirds of UMore Park, located in Dakota County, Minnesota (Figure 2). The project area is approximately 3,500 acres in size and includes the industrial portion of the Gopher Ordnance Works (GOW). The GOW was a 12,000 acre federal government –owned, contractor-operated facility constructed during World War II for the production of smokeless cannon powder and related products. The University acquired portions of the GOW from the Unites States in 1947 and 1948.

A6.1 Historical Site Assessments

A6.1.1 Assessment

A Phase I Environmental Site Assessment (Phase I) was prepared for UMore Park in 2011 (Barr, 2011). In the updated Phase I, seven Recognized Environmental Conditions (RECs) encompassing sixty-nine Sites of Concern (SOCs) were identified. The SOCs were associated with operations that occurred during or after operations of the GOW. In April 2011, Barr submitted a draft Remedial Investigation (RI) Work Plan (Work Plan) to the Minnesota Pollution Control Agency (MPCA) describing the proposed investigation of the SOCs. At the time this QAPP was published, the MPCA had not issued any comments to the Work Plan.

A7 Project Description

UMore Park is located approximately 15 miles southeast of the Twin Cities, west of US Highway 52 and bounded to the north by Dakota County Road 42 and to the south by County Road 58 (170th Street). The RI project area includes the eastern two-thirds of UMore Park. The western one-third of UMore Park, referred to as the UMore Mining Area (UMA), was the subject of a recently completed Environmental Impact Statement and associated environmental assessments for an aggregate mining development (University of Minnesota, 2010). The RI project area and UMore Park boundaries are shown on Figure 2.

The University is conducting this RI at the request of the MPCA to collect environmental data regarding RECs identified in the Phase I and to support the long-term planning of the future use of UMore Park. The Remedial Investigation Work Plan submitted (under separate cover) provides the background information and described the tasks that will be performed at SOCs identified through previous investigation of the project area.

The results of this RI will be used to assess the nature and extent of releases of hazardous substances or petroleum products from GOW and post-GOW activities, to develop strategies to address identified releases, to identify data gaps for consideration during future investigations, and to assist in the planning and implementation of future site activities. The planned investigation includes conducting an RI of the 69 SOCs identified in the Phase I. As described in the Phase I and the RI Work Plan, the project area has been divided into 7 subareas for the purpose of describing the past operational history. The subareas are listed below and are shown on Figure 13 of the Work Plan:

- GOW East
- ABC Line
- GOW Central
- DEF Line
- Navy/Burning Grounds
- GOW West
- GOW North

As described in the draft RI Work Plan, the primary objective of the RI is to provide the University with soil and groundwater data to evaluate the sites of greatest environmental concern in the RI project area. A decision tree illustrating the RI design is included in the RI Work Plan and the Field

Sampling Plan. As detailed in Section 3.0 of the Work Plan, the SOCs included in the RI have been divided up into the following categories:

- Category 1: SOCs where releases of hazardous substances or petroleum products have been identified above Tier I SRVs based upon previous environmental investigation results (Barr, 2011). Environmental data collected at the Category 1 SOCs in the RI will be used to delineate the magnitude and extent of the documented releases.

- Category 2: SOCs where RECs were identified in the Phase I (Barr, 2011) but environmental investigations have not been conducted and/or no releases have been identified. Environmental data collected as Category 2 SOCs will be used for release identification.

- Category 3: Includes portions of the RI project area where releases are less likely to have occurred based upon review of past land use (Barr, 2011). Environmental data collected in Category 3 areas will be used for release identification.

This project will involve both soil and groundwater sampling and characterization. The soil and groundwater samples will be analyzed for parameters that have been selected based on past Site uses. Sampling and analysis plan details, analytical methodologies, quality assurance sampling frequency, and sample container, preservative, and hold times are summarized in Tables 1 through 5 of the QAPP.

Field screening and analytical results will be used to determine if past land use has impacted soil or groundwater at the Site. Soil analytical results will be compared to Minnesota Pollution Control Agency Tier I and II Soil Reference Values (SRVs), considering the human-soil pathway for residential and industrial chronic risk scenarios. Groundwater results will be compared to Minnesota Department of Health Health Risk Limits (HRLs) and Health Based Values (HBVs), and EPA Maximum Contaminant Levels (MCLs). Soil and groundwater data will be compared to regulatory criteria to determine if, and the extent to which, past land use has impacted the site.

Sample collection is scheduled to begin in June 2011 and will take approximately eleven weeks. Laboratory analyses will be completed and data will be provided within 45 days of sample receipt at the laboratory. A report describing the results of the investigation will be prepared in December 2011.

A8 Special Training Requirements/Certification

A8.1 Field Personnel

All field personnel will be under the supervision of the Project Manager. The personnel conducting the on-site activities will be experienced in conducting proper quality procedures as outlined in this QAPP. All field personnel will be trained to follow all health and safety procedures as outlined in the project health and safety plan, as well as in the operation of all field monitoring equipment. All project field staff will have been 40 hour OSHA HAZWOPER trained.

A8.2 Laboratory

The laboratories utilized for this project will have all appropriate certifications necessary to perform analysis in the state of Minnesota, where applicable. A summary of the laboratories certification documentation is included in Appendix D. The laboratory personnel training will be conducted by appropriate trainers and monitored by the laboratory personnel as outlined in the Laboratory QAM included in Appendix B.

A8.3 Training Records

Barr's Health and Safety Manager, Karen Stoller, is responsible for maintaining the OSHA health and Safety Training Records.

A9 Data Quality Objectives and Criteria for Measurement Data

A9.1 Data Quality Objectives

A9.1.1 Project Quality Objectives

Project data quality objectives (DQOs) are qualitative and quantitative statements that specify the quality of the analytical data needed to support decisions made during project investigations. DQOs are established to ensure that the data collected are sufficient and of adequate level of quality for their intended uses. A summary of the project data quality objectives is included in Table 2.

The specific options for the program were developed in conformance with the U.S. EPA document QA/G5 guidance document (EPA, 2002). The following subsections describe the DQO process followed according to QA/G5.

The seven-step DQO process (EPA 2000a) was used to identify the adequacy of existing data and the need for additional data, to develop the overall approach to each study element, and ultimately to design the various field and laboratory investigations.

DQOs are designed to ensure that the type, quality, and quantity of environmental data used in decision-making are appropriate for their intended application. DQOs are qualitative and quantitative statements that: (1) clarify the study objective; (2) define the most appropriate type of data to collect; and (3) determine the most appropriate conditions under which to collect the data. The elements of the seven step DQO process for this sampling effort are described in the following sections and in Table 2 of this QAPP.

A9.1.1.1 Step 1: Identify the Problem

The first step of the DQO process is to develop a concise and complete description of the problem. This problem statement provides the basis for the rest of the DQO development. To do this, technical representatives from the University and Barr worked in consultation with representatives from the MPCA and laboratories. Concise problem statement descriptions are presented in Table 2, broken down by environmental media and area.

A9.1.1.2 Step 2: Identify the Decision

The purpose of this step is to define the decision statement and alternative actions that may be taken depending on the findings of the sampling program. Output from this step will be used to identify decision rules (Step 5) and define tolerable limits on decision errors (Step 6) later in the process. These statements are presented in Table 2.

A9.1.1.3 Step 3: Identify Inputs to the Decision

In this step, the different types of information needed to resolve the decision statement are identified. The inputs to the decisions defined in Step 2 are specified in Table 2 for each medium. In general, decision inputs will consist of historical sampling data, new data generated through the sampling program described in the associated Field Sampling Plan and work plan, background/reference area concentrations and screening levels. Project data needs were identified based on a review of available historical data and consideration of the type, quality, and quantity of data needed.

A9.1.1.4 Step 4: Define the Study Boundaries

Study boundaries are both spatial and temporal. The MPCA approved work plans describe the spatial and temporal boundaries (including overall study area boundaries, sampling areas, specific sampling locations, and project schedule) in sufficient detail to perform the investigation. Spatial and temporal boundaries are also described in general terms in Table 2.

A9.1.1.5 Step 5: Develop a Decision Rule

The decision rule is a synthesis of the output from the previous DQO steps into "if... then..." statements that define the response(s) to the study outcome. In this case, the "if" portion of the statements assesses the sampling results of hazardous constituents and other analytical parameters relative to background/reference area concentrations, human health screening levels, and/or ecological screening levels (whichever is specified as appropriate). The "then" portion of the statement indicates the further action, if required (e.g., further investigation or evaluation in a risk assessment). Decision rules for each element of the investigation are provided in Table 2.

A9.1.1.6 Step 6: Specify Limits on Decision Errors

Decision errors can arise from sampling design error and/or measurement error. It is important to limit the likelihood of decision errors so that risk management and remediation decisions will be

protective of public health and the environment and that project resources will be used appropriately and efficiently.

Sampling design error occurs when the data collection design does not capture the characteristics of the study area to the extent appropriate to answer the principal study questions. This error is influenced by the inherent variability of the population over space and time, the sample collection design, the number of samples, and the uncertainty that is inherent in using sample data to represent the characteristics of the entire target population or environmental medium of interest. It is usually impractical to measure the entire decision unit, and limited sampling may miss some features of the natural variation of the measurement of interest. Sampling design error can lead to random error (i.e., variability or imprecision) and systematic error (bias) in estimates of population parameters. This is reflected in the sampling design by: 1) appropriate selection of sampling locations and analytes, and 2) identification of appropriate sample collection methods.

Measurement errors are defined as the combination of random and systematic errors that inevitably arise during the various steps of the measurement process. This type of error is minimized at this site through the systematic uniform management of each of the steps of the measurement process. Each of the measurement process steps and the overall management plan are outlined in this QAPP for laboratory procedures and in the associated work plan for sampling protocols.

The sampling program and QA procedures for this project were designed based on site-specific information, MPCA guidance, and professional experience with the goal of providing a data set that will limit decision errors to acceptable levels. Potential sources of decision error, along with the potential consequences of any such error, will be identified on a case-by-case basis during the data evaluation phase of the project. The planning team will apply professional judgment to weigh the likelihood of potential decision errors against the risks of incorrect decisions. In the event that the risk of decision error is unacceptably high, the planning team will determine an appropriate course of action (e.g., additional sampling and analysis) to reduce the probability of decision error to an acceptable level.

A9.1.1.7 Step 7: Optimize the Design for Obtaining Data.

The study design for obtaining data to support the work plan objectives was developed through an intensive planning process. Key considerations in the study design were review of information on

site history and material disposal practices, review of previous environmental sampling results, and identification of data gaps.

The sampling program was developed using both quantitative and qualitative approaches to determine the number, type, and locations of sampling locations, to identify analytical parameters, and to establish QA standards and procedures for the project.

Details of the study design and its underlying rationale are provided in the MPCA approved work plans. Study design elements are also summarized under Step 7 in Table 2 of this QAPP.

A9.1.1.8 Project Data Quality Objectives

Project data quality objectives (DQOs) are qualitative and quantitative statements which specify the quality of the analytical data needed to support decisions made during project investigations. DQOs are established to ensure that the data collected are sufficient and of adequate level of quality for their intended uses. Four Site data quality objectives have been identified and are presented below along with brief descriptions of steps that will be taken to address these objectives. The data must satisfy the site data quality objectives presented below.

1. Analytical results must accurately represent groundwater and soil quality: Chemical analyses will be performed to confirm the target analytes present and their concentrations at each SOC.

2. Analytical results must satisfy quality control requirements for: accuracy, precision, representativeness, completeness and comparability (see the following section).

3. Field data requires an intermediate level of data quality compared to laboratory analysis done in a controlled environment: field data provides real-time data that may be necessary to make field decisions. Field data includes volatile organic headspace monitoring with a photo ionization detector (PID) (MPCA Method) and soil classification (ASTM D 2488).

4. The laboratory analyses will require a high level of data quality and will be used to determine the type and concentrations of chemical constituents present at the property. These analyses are characterized by established QA/QC protocols and documentation and provide qualitative and quantitative data. These methods are based on EPA or other regulatory method protocols and are presented in Table 1. Analytical and data review procedures must be in accordance with recognized protocols to ensure the data is valid.

A9.2 Quality Assurance Objectives and Criteria

The overall quality assurance objectives (QAOs) are to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide the level of data required for determining the characteristics of the various environmental media. Specific procedures for sampling, chain-of-custody, laboratory instruments calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability. The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols.

Quality control checks available for use in each project include the following measures:

- Field blank samples are analyzed to check for procedural contamination that may cause sample contamination.
- Duplicate samples are analyzed to check for sampling and analytical reproducibility.
- Matrix spikes (MS) provide information about the effect of the sample matrix on the digestion or preparation and measurement methodology. Matrix spikes are sometimes performed in duplicate and are referred to as MSD samples.

The general level of the QC effort will be a minimum of one field duplicate and field blank for each batch of 20 samples during the investigation. MS/MSD samples are analyzed as required by the methodology in accordance with the laboratory SOPs, but are typically analyzed with every batch of 20 samples, The level of QC effort provided by the laboratory will be equivalent to the level specified within the SOPs for the parameters to be tested (Appendix C).

The five individual QAOs are defined below, along with the means by which they are measured to monitor the compliance to the project needs.

A9.2.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. Precision of analytical laboratory data may be assessed by comparing the analytical results between matrix spike/matrix spike duplicates (MS/MSD), laboratory control sample/laboratory control sample duplicates, laboratory duplicates (non-spiked), or masked field duplicate samples. Duplicate

samples, when collected, processed and analyzed by the same organization, provide intra-laboratory precision information for the entire measurement system, including sample acquisition, handling, shipping, storage, preparation, and analysis. Field duplicate samples are submitted to the laboratory as blind or mask samples. Relative percent differences (%RPD) will be calculated for each pair of duplicate results using the following equation:

$$\%\,\mathrm{RPD} = \left| \frac{S - D}{(S + D)/2} \times 100 \right|$$

Where: S = First sample value

D = Second sample value

RPD calculations of MS/MSD, LCS/LCSD will be performed on the final concentration (not the percent recoveries). The RPD limits for MS/MSD, LCS/LCSD and non-spiked laboratory duplicates are set by the laboratory and are subject to change. For this investigation RPDs falling beyond the laboratory published for MS/MSD, LCS/LCSD and/or non-spiked laboratory duplicates will be evaluated as detailed in the data review SOPs included in Appendix F. The differences between duplicates must be less than the action level for evaluation. All duplicate concentrations greater than five times the reporting level must possess an RPD less than 25% for liquid samples and 50% for soil samples.

A9.2.1.1 Field Precision Objectives

Field precision is assessed through the collection and measurement of replicate field samples with the field equipment at a rate of one per 20 analytical samples to ensure the precision of the field equipment and to demonstrate precision in the field collection procedures. These replicates will be collected and analyzed in the field only. Table 3 outlines the field instrumentation's precision, accuracy limits and preventative maintenance procedures.

Field duplicate samples will be collected and sent to the laboratory at the frequency presented in Table 4. The RPD limits for field duplicate soil samples will be 40% and 30% for field duplicate groundwater samples. Native and duplicate sample results at or near the reporting limits can exaggerate RPD values therefore; these higher RPD values do not always indicate poor precision. Duplicate samples should be collected from locations where target analytes are expected to be present.

A9.2.1.2 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent difference (RPD) for laboratory duplicates, MS/MSD and LCS/LCSD samples. These quality control samples will be analyzed at a rate of one per twenty samples as required by the laboratory SOPs. This data allows for evaluation of the laboratory's ability to satisfactorily replicate specific sample results. The Laboratory precision and accuracy criteria are published in each laboratory report and will be used as the final acceptance criteria during data review.

A9.2.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy measures the bias in a measurement system. Accuracy of laboratory results may be assessed using the analytical results of method blanks, reagent/preparation blank, matrix spike/matrix spike duplicate samples and laboratory control samples. The percent recovery for (%R) matrix spikes and laboratory control samples will be calculated.

Percent recoveries for surrogate standards (for organic analyses only), LCS samples, and MS samples are established by the laboratory and are subject to change. In general, surrogate standard percent recovery limits for VOCs are 75-125%, for the semivolatile and/or PAH analyses the surrogate recoveries vary depending on the class of compound, but for purposes of this investigation acceptable limits will not exceed 30-150% (including pesticides). In general, for MS and LCS samples, percent recovery limits are 80-120% for VOCs and 75-125% metals, for semivolatile and/or PAH analyses, the recoveries vary widely depending on the class of compounds, but for purposes of this investigation, acceptable limits will not exceed 30-150%. Typical percent recoveries for pesticides in MS and LCS samples are 70-130%.

These percent recoveries are subject to change. The current limits will be present along with all sample results within the laboratory reports and will be used as the final acceptance criteria during data review.

Results of method blanks will be evaluated to determine the presence of any gross systematic contamination issues to identify potential false positive results.

A9.2.2.1 Field Accuracy Objectives

Accuracy in the field is assessed through the use of field and trip blanks (for VOC analyses only) and through the adherence to all sample handling, preservation and holding times.

SOPs for the field equipment to measure organic vapors, pH, conductivity, Eh, and temperature are outlined in Appendix E. Accuracy and precision requirements for field screening analyses are included in Table 3.

A9.2.2.2 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through determination of percent recoveries in the analysis of MS/MSD's, LCS/LCSDs and surrogate spikes (for organic analyses only). Accuracy control limits are included in each laboratory report and will be used as the final acceptance criteria during data review. The frequency of sample spikes being analyzed will be at least 5 percent as outlined in the laboratory SOPs and/or EPA or other regulatory methodology. Corrective actions are discussed in more detail in Section C2 of this QAPP for laboratory content, and Appendix F for potential data qualification.

A9.2.3 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Field completeness goals for each project will be greater than 95 percent. It is expected that Legend will provide data meeting QC acceptance criteria for 95 percent or more of all samples tested. However, other factors may affect the decision to resample for lost or otherwise invalid data, such as if the sample was collected for confirmation of an earlier detection, or if the same parameter at the same well was somehow invalidated during consecutive sampling events. Following completion of analytical testing, completeness will be calculated as a percent using the following equations:

$$Completeness\ (\%) = \frac{(Number\ of\ valid\ data)}{(Number\ of\ samples\ collected\ for\ each\ parameter\ analyzed)}\ x\ 100$$

A9.2.4 Representativeness

Representativeness is defined as a measure of the degree to which data accurately and precisely represents a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and proper laboratory protocol. As described in the work plans, the sampling network will be designed to provide samples representative of site conditions. During development of this network, consideration will be given to available

information regarding the site, and any future remedial action. The representativeness criteria will be satisfied by following the associated work plan and by the use of proper sampling techniques and appropriate analytical procedures. Sample collection procedures (included in Appendix E) will describe proper sample homogenization techniques for soil samples and stabilization procedures for water samples that will aid in ensuring a sample is representative of site conditions. This will be measured on this project through the use of matrix spikes, matrix spike duplicates, field blanks, method blanks, and field duplicates as described in Section A9.3.

A9.2.5 Comparability

Comparability is defined as the confidence with which one set of data can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. Comparability will be satisfied by ensuring that the sample plan is followed. This will be accomplished by the project team with the use of matrix spikes, field blanks, method blanks and field duplicates as described in Section A9.3.

A9.2.6 Sensitivity

Sensitivity expresses the methodology's and laboratory instrumentation's ability to meet or exceed the associated screening levels. In some cases, laboratory instrumentation limitations result in final reporting limits greater than the associated screening level. In these cases, the laboratory will report estimated concentrations below the final reporting limit but above the method detection limit. These results will be qualified with a "J" qualifier indicating an estimated concentration.

A9.3 Field Sampling QA/QC

Field blanks will be prepared and submitted to the analytical laboratories to check for procedural contamination at the site which may cause sample contamination.

Accuracy of the field measurements will be assessed using daily instrument calibration, calibration check, and analysis of blanks. Precision will be assessed on the basis of reproducibility by multiple analyses of a single sample. Data completeness may be determined upon project completion and receipt of all data. The quality control program consists of collecting and analyzing field blank and field duplicate samples.

A9.3.1 Field Blanks

Field blanks are defined as samples which are obtained by pouring analyte-free, deionized water into the appropriate sample containers for analysis.

Field blanks will be collected and submitted at the frequency of one field blank per 20 investigative samples. Field blank samples will be identified with the prefix FB followed by a sequential number (FB-1, FB-2....).

The results of field blanks will be evaluated to determine the presence of any potential false positive results. The results of the field blanks should not have reportable concentrations of any target analyte above its reporting limit. Data qualifications relating to field blanks are discussed in Appendix F.

A9.3.2 Field Duplicate Samples

Field duplicate samples are independent samples collected in such a manner that they are equally representative of the parameter(s) of interest at a given point in space and time. Field duplicate samples, when collected, processed, and analyzed by the same organization, provide intralaboratory precision information for the entire measurement system, including sample acquisition, homogeneity, handling, shipping, storage, preparation, and analysis. Field duplicate samples are submitted to the laboratory as blind or mask samples.

One out of every 20 investigative samples will be collected in duplicate, with a minimum of one per event. These samples should be collected at locations where contaminants are expected to be present. Field duplicate samples will be identified with the SOC number, a prefix Dup (Duplicate) followed by a sequential number (Dup-1, DUP-2 ....).

A10 Documentation and Records

The following is a list of information that must be documented and records that must be reported or available for review. The list is not intended to be a complete list of every item, rather general guidance on required information.

A10.1 Field Records

Field records should include:

- Sample collection records
- Chain of custody
- QC sample records, if applicable
- Field procedures
- Field measurement results
- Equipment calibration documentation
- Corrective action reports
- Observation notes
- Weather Conditions
- Results of field testing
- Names of all personnel on site

A10.2 Laboratory Records

Laboratory records should include:

- Date of sample analysis
- Sample management information (e.g., receipt, numbering, handling)
- Analytical procedures
- Notes of deviations from procedures
- Sample preparation and analysis information
- Results of analytical testing
- Detection limits and reporting limits
- QC criteria and results
- Data handling information

A10.3 Storage and Retention

Field files are stored in the Barr project files which are retained on or off-site indefinitely.

Laboratory report retention is discussed in Section B10.5.

B Measurement Data Acquisition

B1 Sampling Process Design

Samples locations, parameters, and rationale will be specified in the associated work plans.

Tables 1 present a summary of the analytical constituents and methods that may be required for laboratory analysis at the Site. The following table presents a summary of the laboratories and associated analyses to be performed.

Laboratory	Analyses **
Legend – St. Paul, MN	Soil, groundwater analyses of volatiles, semi-volatiles, metals and general chemistry parameters
TestAmerica – West Sacramento, CA	Nitrocellulose

**Specific methods for these analyses are contained in Table 1.

B2 Field Sampling Method Requirements

Sample collection procedures are described in the Field Sampling Plan and the Work plan. A short summary of the Site activities is described in the following paragraphs.

B2.1 Field Sampling Equipment and Procedures

Sample collection equipment and procedures are described in Appendix E of this QAPP.

The following is a brief overview of procedures related to the correct acquisition of surface and groundwater levels and samples. It is assumed that the reader has a firm knowledge of environmental sampling, and procedures related to environmental fieldwork.

There are four general types of sampling conducted at the Site; groundwater sample collection, surface soil collection, subsurface soil collection, and composite soil sample collection. Specific numbers of samples to be collected and locations are included in the Field Sampling Plan.

B2.1.1 Sample Collection

A direct push sampling unit, drilling rig, or hand sampling equipment will most likely be used to collect any required sub-surface soil samples using coring, split-spoon sampling and hand sampling gear. In addition, bailers and pumps (submersible or peristaltic) may be used to perform groundwater sampling.

Additional information on the Barr field sampling techniques can be found in the Barr SOPs located in Appendix E. All subcontractors performing work under the direction of Barr will adhere to these SOPs.

B2.2 Field Sample Handling and Analysis

All analytical samples will be collected in the field in accordance with an approved work plan and QAPP.

Each laboratory sample to be transported will be marked with a permanent marker directly on the container or on adhesive labels that will remain on the container. Each shipping container will be marked with a proper U.S. DOT transportation description, the sample designation and the names and addresses of the senders and receivers. Proper shipping papers will accompany each shipment of samples.

All samples will be shipped to the laboratory(s) at the following location except for nitrocellulose samples which may be shipped directly to TestAmerica:

Legend Technical Services, Inc.
Attn: Sample Receiving
88 Empire Drive
St. Paul, MN 55103
(651) 642-1150

TestAmerica – West Sacramento
Attn: Sample Receiving
880 Riverside Parkway
West Sacramento, CA 95605
(916) 374-4442

All samples will be shipped for delivery within four days of sample collection unless sample holding times dictate shorter delivery. All analytical samples will be shipped via an over-night delivery or messenger service.

B2.3 Field Logbooks/Documentation

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

Field logbooks will be bound, field survey books, or notebooks. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned
- Project name
- Project start date
- End date

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

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

Samples will be collected following the sampling procedures documented in the QAPP, the Field Sampling Plan and approved work plans. The equipment used to collect samples will be noted, along with the time of sampling, sample description, volume, and number of containers. A sample identification number will be assigned prior to sample collection. Duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

The following nomenclature will be followed for sample identification.

Samples will be represented by the site-specific building numbers where the sample is collected from, a letter designator representing the type of investigative method, a unique location number indicated in the Work Plan, and, in the case of soil samples, the sample depth. Standard investigative designators are as follows:

- **SS (Surface Soil)**: Surface soil samples will be collected beneath the surface vegetation and the rooting zone, approximately from an interval of 2 to 6 inches below the ground surface. (Example: 101C-SS1-0-0.5', etc.)
- **SB (Soil Boring)**: Represents any soil boring installed for the purpose of collecting information on the stratigraphy or for collecting soil samples with a geoprobe or drill rig. (Example: 101C-SB1-0-0.5", etc.)
- **TT (Test Trench):** Represents any test pit excavated for the purpose of observing subsurface conditions or for collecting soil samples. (Example: 101C-TT1-0-0.5', etc)

Groundwater samples collected from wells will be represented by the well identification number prefix and the date of sample collection.

QA/QC samples will be identified with the following prefixes as discussed below:

- **FB** (Field Blank): Represents a sample collected for QA/QC procedures.
- **DUP** (Duplicate): Represents a duplicate soil or groundwater sample collected for QA/QC procedures. (Example: 101C-DUP1, or for groundwater: GW-DUP1)

- **TB** (Trip Blank): Represents a blank container filled by the laboratory with ultra clean test water or methanol and are employed for VOC sample analysis.

B2.4 Sample Containers, Preservation Techniques, and Holding Times

The sample containers associated with the anticipated analytical tests are listed in Tables 1 and their proper preservation techniques are detailed in Table 5.

B3 Sample Handling and Custody Requirements

It is U.S. EPA Policy to follow the sample custody (chain-of-custody) protocols as described in "NEIC Policies and Procedures," EPA-330/9-78DDI-R, Revised June 1991. This custody is in three parts: sample collection, laboratory analysis, and final evidence files. A sample or evidence file is under your custody if they:

- Are in your possession;
- Are in your view, after being in your possession;
- Are in your possession and you place them in a secured location; or
- Are in a designated secure area.

Barr will follow this EPA policy for this project.

B3.1 Field Chain-of-Custody Procedures

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain-of-custody intact.

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.
2. All containers will be labeled with sample description and location.
3. Sample labels are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.
4. The Barr QA Manager will review field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required.

B3.2 Transfer of Custody and Shipment Procedures

Samples are accompanied by a properly completed chain-of-custody form. An example of the chain-of-custody form is provided on Figure 3. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to the laboratory, or to/from a secure storage area.

Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be sealed and secured with tape for shipment to the laboratory. The cooler is strapped shut with strapping tape in at least two locations. At least one custody seal will be signed and placed over the cooler opening to verify that the samples have not been disturbed.

Whenever samples are co-located with a source or split with a government agency, a separate chain-of-custody form is prepared for those samples and marked to indicate with whom the samples are being co-located. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

All shipments will be accompanied by the Chain of Custody Record identifying the contents. The original record will accompany the shipment, and the pink and gold copies will be retained by the sampler for returning to the sampling office.

B3.3 Chain-of-Custody Samples in the Laboratory

The laboratory sample custodian will be responsible for maintaining proper chain-of-custody from the time that the samples are received by the laboratory for the project. All facility entrances are secured or monitored at all times; all visitors to the laboratory portion of the facility are documented in the visitor's log book. The laboratories document receipt of samples into the laboratory using preprinted chain-of-custody records (client chain-of-custody forms are acceptable). When samples are received in the laboratory, the chain-of-custody documents are signed and dated by the sample custodian. The samples are then assigned an identification number by the sample custodian. Samples do not remain outside refrigeration for more than 4 hours from the time of receipt. Samples are transferred after log-in to the sample refrigerators by the Sample Custodian. The internal analytical request forms, chain-of-custody forms, and any related paperwork are put into the project

folder. The analysts are responsible for the custody of the samples until they are returned to the sample refrigerators.

B3.4 Custody of Evidence File

Until completion of the project, all correspondence, laboratory reports, and data will be maintained in Barr project files. All original laboratory reports and field data are maintained in their original format and stored separately from working copies of these reports. The Barr Project Manager will direct maintenance of the project file. Following completion of the project, the evidence file will be stored in the Barr project file storage area or transferred to a secure document storage facility. The files will be maintained for a minimum of 5 years.

B4 Analytical Methods Requirements

Trained Barr personnel (or Barr's subcontractor) will perform all field analytical methods. Table 1 presents the required methods for each of the target compounds identified for this project.

B4.1 Laboratory Samples

All laboratory samples will be collected following all applicable EPA and other regulatory methods as described in the laboratory SOPs included in Appendix C.

B4.2 Laboratory Analysis

Analytical methods will be selected to provide adequate detection limits for compounds of interest, and for the final intended data usage. A list of anticipated laboratory methods and their corresponding reporting limits and minimum detection limits can be found in Table 1. All solid sample results will be provided on a dry weight basis as the methodology specifies. SOPs have been prepared for all methods used for analysis of samples for this project. Laboratories project-required SOPs are included in Appendix C. Each of these SOPs is based on an analytical method published by the U.S. EPA, Standard Methods or other recognized sources as available.

A few compounds (including some explosives, pesticides, VOCs and SVOCs) listed in Table 1 have reporting limits that do not achieve regulatory criteria. For these compounds, the laboratories will quantitate down to the method detection limit to achieve the lowest possible levels. This will result in the majority of the compounds meeting their respective regulatory limits.

There are a relatively small number of compounds that will not be able to be quantified below the regulatory criteria using approved analytical methods. It is possible that these compounds will be present above regulatory criteria in samples with results reported as non-detect. Barr will evaluate each compound on an individual basis at each investigation area to determine what potential risks may be involved with not being able to quantify to the regulatory criteria.

B4.3 Field Analysis

Barr personnel will perform analytical screening in the field which may include soils identification, headspace, pH, Eh, temperature, conductivity. All field screening methods will be selected to allow for real time data, while meeting data quality objectives. SOPs for the field methods are included in Appendix E.

B5 Quality Control Requirements

B5.1 Field Quality Control Requirements

QC procedures for pH, Eh, specific conductance, temperature of water samples, flame ionization detector (FID), photoionization detector (PID), and organic vapor measurement for soils will include calibrating the instruments as described in Section B7.2 of the QAPP, measuring duplicate samples and checking the reproducibility of the measurements by taking multiple readings on a single sample or reference standard. The thermometer used will be compared to a NIST traceable thermometer (or equivalent). Assessment of field sampling precision and bias will be accomplished through collecting field duplicates and field blanks for laboratory analysis. Collection of the samples will be in accordance with the applicable procedures in the SOPs located in Appendix E. Frequency of the collection of quality assurance samples is presented in Table 4. Field collection techniques must be conducted to ensure that samples will not be field filtered or otherwise transferred from one sample container to another (with the exception of field filtered metal samples) and that whenever possible, samples will be collected from the dirtiest location to the cleanest whenever the nature of the contamination is known. Field collection techniques must also ensure that water samples for volatile analysis are not collected in a manner which allows for headspace within the sample vials.

B5.2 Laboratory Quality Control Requirements

The laboratories proposed for use on this project ensure the production of quality analytical data through the use of overall quality assurance systems that are supported by documented quality control checks.

B5.2.1 Quality Assurance Program

The main objectives of Legend and TestAmerica's QA Programs are to assure that the laboratory generates data of known quality, that data meets or exceeds all QA/QC criteria, and that records necessary to document laboratory performance are maintained. QA oversight is performed throughout sample processing from initial order/entry, through the analytical system, to the final report. The QA Officer is responsible for monitoring compliance with the laboratory Standard Operating Procedures, and established Good Laboratory Practices (GLPs). Additionally, the QA/QC Officer has the responsibility of providing feedback to management and identifying and implementing policies to improve quality.

All laboratory procedures are documented in writing as SOPs. Internal quality control procedures for analytical services will be conducted in accordance with their standard operating procedures and the individual method requirements in a manner consistent with appropriate U.S. EPA procedures, 40 CFR Part 136 and SW846. The analytical SOPs are presented in Appendix C.

B5.2.2 Quality Control Checks

The particular types and frequencies of quality control checks analyzed with each sample are defined in the laboratory SOPs and QAM. All analytical procedures are documented in writing as SOPs and each SOP includes a QC section, which addresses the minimum QC requirements for the procedure. The internal quality control checks might differ slightly for each individual procedure but in general the QC requirements include the following:

- Method blanks
- Reagent/preparation blanks (applicable to inorganic analysis)
- Instrument blanks
- Matrix spikes/matrix spike duplicates (MS/MSDs)
- Surrogate spikes (applicable to organic analysis)
- Field duplicates
- Laboratory duplicates
- Laboratory control standards
- Internal standard areas for GC/MS analysis
- Mass tuning for GC/MS analysis
- Proficiency Testing Blind Standard

Refer to the submitted SOPs (Appendix C), and the Laboratory Quality Assurance Manual in Appendix B for a description of the specific QC requirements and the frequency of internal and external audits.

All data obtained will be properly recorded. The data package will include summary QC data to allow the recipient to evaluate QC results and compare it to applicable criteria. All samples analyzed and appearing in nonconformance with the QC criteria, will be reanalyzed by the laboratory, if sufficient volume is available. It is expected that sufficient volumes/weights of samples will be collected to allow for reanalysis when necessary.

B5.3 Field Quality Control Requirements

Barr ensures the production of quality field data through the use of overall quality assurance systems that are supported by documented quality control checks. These checks include instrument calibration standards and field blanks.

B6 Instrument/Equipment Testing, Inspection and Maintenance Requirements

B6.1 Field Equipment

Barr staff and/or subcontractors perform routine preventive maintenance of instruments based on manufacturers' recommendations and schedules. Equipment usage and calibration standards are obtained from the manufacturer of that equipment or from a recognized standard source. Field equipment maintenance information is provided in the SOPs (Appendix E).

B6.2 Laboratory Equipment

Legend performs routine preventive maintenance of instruments based on manufacturer's recommendations. Maintenance of the laboratory instruments is the responsibility of the analyst. Laboratory equipment maintenance information is provided in Section 8 in Legend's QAM and in Section 20 of TestAmerica's QAM.

B7 Instrument Calibration and Frequency

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment which are used for conducting field and laboratory analyses. These instruments and equipment are calibrated prior to each use or on a scheduled, periodic basis.

B7.1 Laboratory Instruments

Procedures for initial calibration and continuing calibration verification are in place for all instruments within the laboratory. The calibrations generally involve checking instrument response to standards for each target compound to be analyzed. The source and accuracy of standards used for this purpose are integral to obtaining the best quality data. Standards used at the laboratories are prepared from pure standard materials or purchased. All standards in solution are stored in a discrete freezer or refrigerator in the applicable laboratory section. Each standard is discretely designated. The information is stored in a standards book and/or electronically within the laboratory database.

Instruments are calibrated and recalibrated at regular intervals as specified in the applicable SOP, and consistent with EPA or Standard Methods methodology.

The frequency of calibration and calibration verification, number of points calibrated, and acceptance criteria for each of the instruments to be used are provided in the SOPs.

Additional information on laboratory calibration procedures is included in laboratory SOPs located in Appendix C.

B7.2 Field Equipment

All field equipment is tested and maintained when needed using manufacturers' recommendations and labeled with most recent calibration date.

B7.3 Field Instrument Calibration

The field instruments will be calibrated as described in the manual provided by the manufacturer. Field instruments include an organic vapor analyzer (PID), water quality meter (to measure pH, temperature, conductance, and dissolved oxygen), and a balance. As a rule, instruments will be calibrated daily prior to use.

The calibration procedures performed will be documented in the field report and will include the date/time of calibration, name of person performing the calibration, reference standard used, and readings taken on the standard. Multiple readings on one sample or standard or on replicate samples will also be documented.

B8 Inspection/Acceptance Requirements for Supplies and Consumables

Supplies and consumables that will be used for the projects include, sample jars, sampling equipment and various analytical reagents and gasses.

All sample jars and analytical reagents will be supplied by each laboratory and be acquired from approved vendor sources. The laboratory will acquire only pre-cleaned, certified sample jars approved for the analytes/methods cited in Table 1 per EPA specifications. Trip Blanks for volatile analysis will be provided by the laboratory. All pre-preserved sample jars will be shipped to the site in accordance with federal shipping guidelines. All gasses and reagents will be supplied by approved vendors or be traceable to standard lots, and if any variation in method performance occurs, this will be compared to the change of an analytical reagent. If there is any correlation between a reagent lot and the method variations, that reagent lot may be isolated for further analysis.

Sample jars will not be accepted at any site if there is more than 10% breakage of the jars upon receipt. If the sample jars contain preservative and are broken in the receiving container, none of the sample jars in that container will be used for sampling.

All sampling equipment will be examined upon receipt from various vendors. In the case of sampling gloves, if any physical tears or discoloration exists on the gloves, they should not be used. Sampling scoops that have obvious physical damage should also not be used.

All other consumable equipment will be examined on-site and a determination as to its usability will be made based upon the product's physical appearance.

B9 Data Acquisition Requirements for Non-Direct Measurements

Existing chemical data from previous investigations at this site were used to design the scope for this investigation. Historical data were reviewed for quality assurance.

B10 Data Management

All data generated through field activities or by the laboratory shall be reduced and validated prior to reporting. No data will be disseminated by the laboratory until it has been subjected to the procedures summarized in subsections below:

B10.1 Data Collection

Most outputs are generated through computer programs that have been validated by the manufacturer prior to laboratory purchase of the instrumentation. The instruments have programs available for the analysts to manually verify integrations and quantitations as part of the manufacturer's software package. Manual verification is routinely performed annually.

B10.2 Data Reduction

Data reduction includes all processes that change either the instrument/computer-generated values, quantity of data values or numbers of data items, and frequently includes computation of summary statistics. In most cases, a programmable calculator, computer spreadsheet or computer program is used to generate statistical information. The documentation allows the reviewer to verify the validity of the data reduction process.

An extra significant figure (may be more than one) is carried through all calculations until the final, reportable result is generated. Analytical results are never corrected for blank (background) contamination.

In the data review process, the data produced are compared to information concerning the sample processing history, sample preparations, sample analysis, and associated QA data to evaluate the validity of the results. In addition, any project-specific requirements are reviewed for data compliance.

B10.2.1 Field Data Reduction Procedures

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. The use of pH meters, thermometers, FIDs/PIDs, and specific conductance probes will generate measurements directly read from the meters following calibration per manufacturer's recommendations as outlined in Section B7.2 of this QAPP. Such data will be written into field data sheets immediately after measurements are taken. If errors are made, results will be legibly crossed

out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, when the results forms required for this study are being filled out, the Barr QA manager and project manager, will proof the forms to determine whether any transcription errors have been made by the field crew.

B10.2.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures will be followed according to the following protocol: All data are generated by the analyst and either manually entered or electronically transferred into an electronic report from the software used to process the original data set. Copies of printouts (such as gas chromatograms) will be maintained on file.

Errors are noted, corrections are made, but the original notations are crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry-weight basis (if sufficient volume has been submitted for the percent moisture measurements). One hundred percent of the analytical data is peer reviewed.

Quality control data (e.g., laboratory duplicates, surrogates, MS/MSDs) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Final data packages will be sent to the Laboratory Project Manager for review. Upon approval the data packages will be sent to Barr. Unacceptable data shall be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits, and any other anomalous conditions encountered during sample analysis. After reported by the laboratory, they are considered ready for third-party data validation. More information on laboratory data reduction can be found in the individual analytical SOPs located in Appendix C.

B10.3 Data Validation

Data validation procedures shall be performed for all laboratory data following the Barr SOPs included as Appendix F.

B10.3.1 Procedures Used to Evaluate Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of field notebooks, on the part of field crew members. This task will be the responsibility

of the Barr Field Manager, who will otherwise not participate in making any of the field measurements, or in adding notes, data or other information to the notebook.

B10.3.2 Procedures to Review Laboratory Data

The data will be reviewed in accordance with Barr's Data Validation SOPs, located in Appendix F, which are based on the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review, 2008/2010.

Barr data assessment will be accomplished by the joint efforts of the QA Manager and Project Manager. The data assessment by the project manager will be based on the criteria that the sample was properly collected and handled according to the associated work plan and QAPP.

The Barr QA Manager will conduct a systematic review of the data for compliance with the established QC criteria based on the spike, duplicate and blank results provided by the laboratory. Essentially, all technical holding times shall be reviewed; results of all blanks, surrogate spikes, MS/MSDs, laboratory control samples, and system performance checks shall be reviewed. One hundred percent of the data shall be reviewed.

The data reviewer will identify any out-of-control data points and data omissions and interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the project manager based on the extent of the deficiencies and their importance in the overall context of the project.

All data generated for the projects will be computerized in a format organized to facilitate data review and evaluation. The computerized data set will include the data flags applied by the laboratory, as well as any additional data flags by the Barr QA Manager following the data validation process (Appendix F). The laboratory-provided data flags will include such items as when a concentration below required reporting limit and concentration of chemical(s) were found in laboratory blank. The data reviewer comments will indicate that the data are: (1) usable as a quantitative concentration, (2) usable with caution as an estimated concentration, or (3) unusable due to out-of-control QC results.

The overall completeness of the data package will also be evaluated by the Barr QA manager. Completeness checks will be administered on all data to determine whether deliverables specified in the QAPP are present. At a minimum, deliverables will include sample chain-of-custody forms,

analytical results, and QC summaries. The QA Manager will determine whether all required items are present and request copies of missing deliverables.

B10.4 Data Reporting

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

B10.4.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field. Field documentation of field instrument calibrations, well logs, boring logs, sample identifications, etc. will be contained in the final field reports. Examples of field forms used for final field reports are included in Appendix G.

B10.4.2 Laboratory Data Reporting

Laboratory analyses reports will generally be submitted to Barr Engineering Co. within four weeks of the receipt of samples. The Laboratory Project Manager performs a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the record of chain-of-custody, the report format shall consist of the following:

- Date of issuance
- Any deviations from intended analytical strategy (in case narrative)
- Laboratory batch number
- Quality control procedures utilized and also references to the acceptance criteria
- Project name and number
- Condition of samples 'as-received'
- Discussion of if holding times were not met
- Discussion of technical problems or other observations which may have created analytical difficulties (in case narrative)
- Discussion of any laboratory quality control checks which failed to meet project criteria(in case narrative)

- Signature of the Laboratory Project Manager and Report Reviewer
- Sample collection and receipt date
- Extraction /digestion and analysis dates
- Cross referencing of laboratory sample to project sample identification numbers
- Sample preparation and analyses date for samples
- Sample data (including units and percent moisture / solid data used in dry weight corrections – if applicable)
- MS/MSD, LCS/LCSD and method blank data (percent recoveries and RPDs)
- QC data summary
- Laboratory reporting limit and method detection limits for each analyte
- Method used for analysis
- All sample results and their associated raw data for samples, quality control samples, method blanks
- Percent recovery of surrogate compounds.
- Electronic data deliverable
- Data qualifier description

Data will be received in an electronic format compatible to the Barr laboratory information management system (LIMS). Any data received in non-electronic form will be entered into the Barr LIMS database and output in spreadsheet format to be used in reports.

B10.5 Data Retention

Raw data generated for this project will be stored by the laboratory for five years. Final laboratory reports are kept in archive files by Barr Engineering Co. indefinitely.

B11 Data Acquisition Requirements

B11.1 Previous Data Collection

Data previously generated for this site will be utilized for decisions in accordance to the level of quality control performed for each event.

C Assessment and Oversight

C1 Performance and System Audits

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the work plan and QAPP. The audits of field and laboratory activities may include two separate independent parts: Internal and External audits.

C1.1 Field Audits

Internal audits of field activities (sampling and measurements) are conducted by the Barr QA Manager. The audits will include examination of field sampling records, field instrument operating records, maintenance of QA procedures, sample collection, handling and packaging in compliance with the established procedures. The audit will also include examination of QA procedures and chain-of-custody procedures to ensure they are being followed correctly. A copy of the field audit checklist is included as Appendix I. While the QA Officer may perform field audits, no field audit for this project is anticipated.

C1.2 Laboratory Audits

Many of the objectives of a routine audit are similar to those a client or independent auditor would hope to accomplish during an on-site laboratory evaluation and data audit. These goals include the following:

- Documented quality control and quality assurance procedures, including necessary corrective actions, are being applied.
- Adequate facilities and equipment area are available to perform the client's required scope of work.
- The personnel are qualified to perform the assigned tasks.
- Complete documentation is available, including sample chain-of-custody.
- Proper analytical methodology is being applied.
- Acceptable data-handling techniques are being used.

- Corrective actions identified in any previous on-site visits have been implemented.
- The laboratory management continues to demonstrate a commitment to quality.

In response to an audit, any corrective actions taken are noted with reference to the auditor's deficiency report and the laboratory's SOPs.

C1.2.1 Internal Audits

Internal audits of laboratory activities are conducted by the Laboratory QA Manager or their qualified designee (internal auditor). The audit may be either scheduled or unannounced before it is conducted. A system audit is an on-site inspection and review of one system in the QA/QC program for the laboratory. A performance audit could include the evaluation of one individual or procedure performed in the laboratory. While performance audits are a quantitative appraisal, system audits are for the most part qualitative in nature. The auditor may: (1) review the laboratories' SOPs to verify compliance with EPA procedures; (2) review hands-on procedures to ensure compliance with written SOPs; and (3) verify that proper corrective action has been taken. Personnel and facilities may also be evaluated during an audit.

If deficiencies are observed during an audit, and if deemed necessary, a findings report will be initiated. A findings report will include sufficient detail as to all remedial actions taken. The findings report indicates the proposed implementation date and the individual(s) responsible for the corrective action. A follow-up audit or other documentation may be needed to conclude the corrective action.

C1.2.2 External Audits

Laboratory performance will be evaluated by reviewing the QC procedures, SOPs, and qualifications of the laboratory. In addition to the document review, an on-site laboratory visit and evaluation is included to evaluate the audit items indicated above. Legend has participated in Barr's independent QA audit program for over 10 years, is audited on a biennial schedule and participates in Barr's blind sample program. All audit results are on file at Barr. Legend's last Barr audit occurred in February 2009 with favorable findings. No non-conformance issues were identified.

C1.2.3 Preventative Maintenance

Routine preventative maintenance is performed on laboratory(s) equipment as scheduled by laboratory personnel in accordance with the laboratories QAMs and SOPs included in Appendices B and C.

C2 Corrective Actions

Corrective actions may be required for two classes of problems: (1) a deficiency that does not adversely affect data; and (2) a deficiency that does affect data. A problem could occur from the time samples are collected up until data is reviewed, including: sampling and sample handling, sample preparation, laboratory instrumental analysis, and data review.

For any problem, a corrective action will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Project Manager.

Any nonconformance with the established quality control procedures in the QAPP will be identified and corrected in accordance with the QAPP.

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

C2.1 Sample Collection

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Barr Field Manager. The Barr Field Manager will be responsible for assessing the suspected problems, in consultation with the Barr Project QA Manager and the Barr Project Manager, and making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the Barr Project Manager.

The Barr Project Manager will be responsible for verifying that corrective action for nonconformances are initiated by:

- Evaluating all reported nonconformances
- Controlling additional work on nonconforming items
- Determining disposition or action to be taken
- Maintaining a log of nonconformances
- Reviewing nonconformance reports and corrective actions taken

- Verifying inclusion of nonconformance in the final site documentation in project files

If appropriate, the Barr Project Manager will see that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

The Barr Project Manager or his designee is responsible for all site activities. In this role, the designee at times is required to adjust the site programs to accommodate site-specific needs. When it becomes necessary to modify a program, the responsible person notifies the Barr Project Manager of the anticipated change and implements the necessary changes after obtaining the approval of the Barr Project Manager. The Barr Project Manager must approve the change in writing or verbally prior to field implementation, if feasible. If unacceptable, the action taken during the period of deviation will be evaluated in order to determine the significance of any departure from the established practices, and determine action to be taken.

The Barr Field Manager for the Site is responsible for the controlling, tracking, and implementation of the identified changes. Reports on all changes will be distributed to all affected parties.

C2.2 Laboratory Analyses

When nonconformances occur, analysts notify their immediate supervisor. The laboratory supervisor will evaluate the problem and decide what corrective action is required. The following guidelines are used to validate data, and determine what, if any, corrective action is necessary.

- Verify all calculations which use raw laboratory data, including sample aliquots, dilution factors, linear regression calculations, etc.
- Verify that method specific matrix interference procedures were followed. Check the analytical data which was generated for other field samples in the same analytical batch in order to determine whether the problem is unique to a single sample (a possible matrix problem).
- Review the analytical procedure with the analyst to make certain that the required procedures and sample preparation techniques were performed correctly.
- Check the initial calibration data to verify that instrumental operating requirements were met prior to starting sample analysis.
- Verify that quality control sample checks were analyzed at the proper frequency and that quality control sample performance criteria requirements were met.

- Determine if an alternative method would be more appropriate for sample analysis.
- Review log-in and chain-of-custody information to determine if sample conditions may have been affected between sampling and receipt of sample.

When a definitive explanation for the problem cannot be determined, sample reanalysis is required. All nonconformances and corrective action procedures taken to correct the problem must be documented and included in the job file.

If the nonconformance has not been corrected and the validity of the data is in question, the laboratory director, laboratory QA Officer, or Laboratory Project Manager must contact Barr. All actions will be documented in the applicable work order file.

The laboratory quality assurance department is also responsible for implementing the internal audit protocol which verify compliance with laboratory SOPs and assist in identifying and correcting any deficiencies. Follow-up audits verify that proper corrective action has been taken for the identified discrepancy.

Barr may request corrective action for any nonconformance identified by audits or data validation. Corrective action may include:

- Reanalyzing the samples, if holding time criteria permit;
- Resampling and analyzing;
- Evaluating and amending sampling procedures and/or evaluating and amending analytical procedures; and/or
- Accepting data and acknowledging the level of uncertainty.

C3 Quality Assurance Reports to Management

The final report will contain QC sections that summarize data quality information collected during the project. Included in this report will be a discussion of the field activities during sample collection, a brief discussion of the QA/QC activities conducted by the laboratory, a summary of the data validation procedures performed by Barr on the laboratory data, and tabulated results of analytical data.

D Data Validation and Usability

D1 Data Review, Validation and Verification

D1.1 Data Review and Validation

For the purposes of this document, data validation is defined as the evaluation of the technical usability of the data. Data verification is defined as the determination of adherence to SOPs, the field sampling plan, the QAPP, and the laboratory(s) quality assurance plan.

Data review and validation will be performed as presented below. Verification is accomplished through laboratory audits and review of QC data.

D1.2 Laboratory Data Review and Validation

Data validation takes place on two levels. The first level of review occurs "at the bench." Analysts are charged with the responsibility of monitoring all laboratory QA/QC activities, and verifying that systems are in control. Data validation also occurs on a sample-by-sample basis. The initial review is performed by the instrument operator or analyst who is responsible for assessing the following:

- Cross-checking all sample identification numbers on work sheets, extract vials/digestate bottles, and instrument outputs.
- Calculation of surrogate recoveries and internal standard responses (when applicable), and verification that QA acceptance criteria are met.
- Verification that all calibration, tuning, linearity, and retention time drift checks are within QA acceptance criteria.
- Determination that peak chromatography and other instrument performance characteristics are acceptable.
- Confirmation that chain-of-custody is intact based on accompanying paperwork.
- Verification of all preparative and analytical procedures was conducted within method suggested holding times.

The area supervisor and/or technical supervisor perform the second level of validation and review. The analyst, technical reviewer, and/or the Laboratory Project Manager are responsible for the QC and data review of analyses and reports. The QC review of QC analyses and applicable calibrations is completed and includes the following:

- Confirmation that all quality control blanks meet QA requirements for contamination, and that associated sample data are appropriately qualified when necessary.
- Calculation of matrix spike recoveries and duplicate RPDs, and confirmation that accuracy and precision QA criteria are met or appropriately flagged when necessary.
- Comparison of all injections of a sample and comparison of matrix spikes with the original unspiked sample for acceptable replication.

After QC review the data are sent to report preparation. The final report review includes both data review and a review of report accuracy. The data review includes confirmation of all assessments previously made by the operator/analyst, and includes an evaluation of the qualitative identification of all target analytes using specific SOP interpretation criteria.

Data generated by the analyst is reviewed by a technical reviewer for data completeness and accuracy.

The final report review will assess the complete data report for completeness, accuracy of reported hits, comparison to target analyte lists, and comparison with project QC requirements. The Laboratory Project Manager generates and reviews the final report and reviews as summarized below:

- Making a comparative evaluation of data from individual fractions of a sample, and of samples from the same sit for consistency of analytical results and resolution of discrepancies.
- Checking data report or case narrative for completeness.
- Verifying QAPP specific requests have been met.

D1.3 Field Data Review and Verification

Field data is reviewed by both the QA Manager and the Field Manager. Additionally, during preparation of the final field report, technical field staff verifies their documentation for accuracy and

completeness. The QA Manager and the Barr Project Manager additionally check for completeness, representativeness and any transcription errors. If any errors are detected, the field personnel will be contacted and corrective action will be initiated.

D1.4 Barr Data Review and Validation

The data will be reviewed in accordance with Barr's Data Validation SOPs, located in Appendix F, which are based on the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review, 2008/2010. Data validation procedures will use the method-specific QC acceptance limits specified in the EPA SW-846 methods and SOPs.

The specific requirements which will be checked during data validation are:

1. Holding times
2. Method blank data
3. Surrogate recovery
4. Laboratory Control Sample data
5. Matrix spike data
6. Duplicate analyses data
7. Overall data assessment

Upon completing the validation procedure for all data, a quality control review report will be compiled and submitted. The Barr SOP for data review is included as Appendix F.

D1.5 Data Verification

Data verification is defined as the determination of adherence to SOPs, the field sampling plan, the work plan, the QAPP, and the laboratory QAMs. Internal and external laboratory audits measure adherence to these elements. In addition, internal and external verification of adherence to these elements will be completed through the evaluation of field and laboratory documentation.

D2 Validation and Verification Methods

Data validation methods to be used are based on the following documents:

- EPA, 2010. *USEPA Contract Laboratory Program, National Functional Guidelines for Inorganic Superfund Data Review.* OSWER 9240.1-51. USEPA-540-R-10-011, January 2010.

- EPA, 2008. *USEPA Contract Laboratory Program, National Functional Guidelines for Superfund Organic Methods Data Review.* OSWER 9240.1-48, USEPA-540-R-08-01. June 2008

A brief overview of procedures for evaluating and reviewing the data are included below:

Holding Times: Compare the time and date the sample was collected (on the chain-of-custody) to the date analyzed in the laboratory data package. Verify the dates are within the SW-846 recommended holding times for the particular method.

Method Blank Data: Verify through the method blank sample data results that no significant laboratory contamination issues exist.

Surrogate Recovery: Verify the percent recovery of each surrogate falls within acceptable laboratory quality control limits included in each laboratory report, or the Barr SOP presented in Appendix F.

Laboratory Control Sample Data: Verify the percent recovery of the spiked compounds is within acceptable laboratory criteria included in each laboratory report, or the Barr SOP presented in Appendix F.

Matrix Spike Data: Verify the percent recovery of the spiked compounds is within acceptable laboratory criteria included in each laboratory report, or the Barr SOP presented in Appendix F.

Field Duplicate Analysis Data: Calculate the relative percent difference for all detections of target compounds above the laboratory reporting or minimum detection limits, and compare them to the acceptance criteria included in each laboratory report, or the Barr SOP presented in Appendix F.

Overall Data Assessment: Examine the data package as a whole and compare it to (1) the chain-of-custody to verify completeness, (2) the historical data to verify representativeness (3) the other site data to verify comparability is being achieved.

Qualification of the data may result if the evaluation criteria for data validation are not met. All data qualification will be presented on the tabulated form of the data, and in the QA review sections all site reports.

D3 Reconciliation with Data Quality Objectives

D3.1 Specific Procedures to Assess Data Precision, Accuracy and Completeness

D3.1.1 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, completeness and sensitivity as follows:

D3.1.1.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD and LCS/LCSD for organic analysis, and laboratory duplicate analyses for inorganic analysis. The relative percent difference (%RPD) will be calculated for each pair of duplicate analyses using the following equation:

$$\%RPD = \left| \frac{S - D}{(S + D)/2} \, x\, 100 \right|$$

Where: S = First sample value (original or MS value)
D = Second sample value (duplicate or MSD value)

D3.1.1.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in the specific SOPs using the analytical results of method blanks, reagent/preparation blank, matrix spike/matrix spike duplicate samples, field blank, and bottle blanks. The percent recovery (%R) of matrix spike samples and LCS will be calculated using the following equation:

$$\%R = \frac{A - B}{C} \, x\, 100$$

Where: A = The analyte concentration determined experimentally from the spiked sample;

B = The background level determined by a separate analysis of the unspiked sample; and

C = The amount of the spike added.

D3.1.1.3 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. The completeness is calculated as described previously.

D3.2 Data Quality Assessment

The data will be compiled from each investigation phase and summarized in tabular and/or graphical form.

The data quality assessment process will involve multiple steps depending on the results of the data validation process. Data that has been qualified (by the laboratory or by Barr) will be assessed for the particular circumstances surrounding the sample. For example, if multiple compounds are detected in a method, field or trip blank and in the associated samples at comparable levels (as defined in Appendix F), the data result will likely be treated as a false positive; however, if the sample location is critical (i.e., compliance boundary), the data may be treated as non-false positive or rejected and resampled. This also applies to qualifications based on failure to meet matrix spike/matrix spike duplicate criteria if the sample or contaminant affected is critical to the project decision-making, in which case corrective actions may result. Corrective actions may include resampling and/or reanalysis of the sample. Detection limits may be elevated above appropriate criteria due to dilutions or matrix interferences. In this case, the necessity of the data will be evaluated as with the previous examples and potential corrective actions may include (a) reporting the data result as equal to the method detection limits and using the qualified data, or (b) resampling of critical samples.

Additional factors that may be considered when evaluating the data include:

- Data time-series or historical trends.
- Spatial distributions of results such as similar and dissimilar results from adjacent sample locations.
- Outlier analysis (when statistical sampling protocols are used).
- Statistical interpretation of large data sets (sample sizes) when statistical sampling protocols are used.

- The relationship of detected results to known site history information. For example, soil results indicating a possible chemical release beneath a bulk chemical storage and loading area or beneath a former storage tank location.
- The relationship of detected results to other transient site conditions such as dynamic contaminant migration through vadose zone soils or as a solute plume in migrating groundwater.
- The relationship of detected results to site conditions such as geologic stratigraphy, historic site development (filling, previous demolition), proximity to neighboring contamination sources.

The results will be compared to the project quality objectives that are summarized in Section A9.1.1.8 of this QAPP and summarized in Table 2.

D3.2.1 Sensitivity

Laboratory sensitivity will be assessed by comparing the analytical reporting or minimum detection limits to the applicable site standard criteria (Table 1). If the analytical detection limits presented are greater than the listed site criteria, the following procedures will be applied and a decision on the site data will be made;

- Verify the laboratory cannot achieve lower detection limits for the parameter of interest.
- Examine other matrices at the site for detections of parameter of interest.
- Establish historical likelihood that the parameter in question is a contaminant of concern.

Examine all positive detections in the samples of interest to identify if like-compounds are present.

Tables

Table 1
Analytical Parameters, Methods and Quantitation Limits
Stage 1
UMore East Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	CAS Number	Matrix	Method (EPA unless noted otherwise)	Method Detection Limit	Reporting Limit	Test Unit	MN GW Values	Minnesota Tier SLV [4]	Minnesota SRV [5]	Minnesota Tier II Industrial SRV [6]
RCRA Metals										
Arsenic	7440-38-2	Soil/Solid	6010B	0.055	0.50	mg/kg	NA	15.1	9	20
Barium	7440-39-3	Soil/Solid	6010B	0.060	1.00	mg/kg	NA	842	1100	18000
Cadmium	7440-43-9	Soil/Solid	6010B	0.033	0.25	mg/kg	NA	4.4	25	200
Chromium	7440-47-3	Soil/Solid	6010B	0.012	0.50	mg/kg	NA	18 CR	87 CR	650 CR
Lead	7439-92-1	Soil/Solid	6010B	0.14	1.0	mg/kg	NA	525	300	700
Selenium	7782-49-2	Soil/Solid	6010B	0.095	1.0	mg/kg	NA	1.5	160	1300
Silver	7440-22-4	Soil/Solid	6010B	0.016	0.25	mg/kg	NA	3.9	160	1300
Mercury	7439-97-6	Soil/Solid	7471A	0.019	0.10	mg/kg	NA	1.6 MC	0.5	1.5
RCRA Metals										
Arsenic, Dissolved	7440-38-2	Water/Liquid	6010B	0.0011	0.010	mg/L	--	NA	NA	NA
Barium, Dissolved	7440-39-3	Water/Liquid	6010B	0.0012	0.020	mg/L	2 HRL93	NA	NA	NA
Cadmium, Dissolved	7440-43-9	Water/Liquid	6010B	0.00013	0.0010	mg/L	4 HRL93	NA	NA	NA
Chromium, Dissolved	7440-47-3	Water/Liquid	6010B	0.00024	0.010	mg/L	0.1 CR	NA	NA	NA
Lead, Dissolved	7439-92-1	Water/Liquid	6010B	0.00042	0.0030	mg/L	--	NA	NA	NA
Selenium, Dissolved	7782-49-2	Water/Liquid	6010B	0.0019	0.020	mg/L	0.03 HRL93	NA	NA	NA
Silver, Dissolved	7440-22-4	Water/Liquid	6010B	0.00031	0.0050	mg/L	0.03 HRL93	NA	NA	NA
Mercury, Dissolved	7439-97-6	Water/Liquid	7470A	0.000037	0.00020	mg/L	--	NA	NA	NA
Major Cations and Anions										
Calcium, Dissolved	7440-70-2	Water/Liquid	6010B	0.030	1.0	mg/L	--	NA	NA	NA
Magnesium, Dissolved	7439-95-4	Water/Liquid	6010B	0.0021	1.0	mg/L	--	NA	NA	NA
Potassium, Dissolved	7440-09-7	Water/Liquid	6010B	0.0016	1.0	mg/L	--	NA	NA	NA
Sodium, Dissolved	7440-23-5	Water/Liquid	6010B	0.0053	1.0	mg/L	--	NA	NA	NA
Chloride	16887-00-6	Water/Liquid	9056A	0.0029	0.10	mg/L	--	NA	NA	NA
Sulfate	14808-79-8	Water/Liquid	9056A	0.024	0.10	mg/L	--	NA	NA	NA
Total Alkalinity	NA	Water/Liquid	SM2320B	5.3	20	mg/L	--	NA	NA	NA
Nitrate+Nitrite Nitrogen as N										
Nitrate + Nitrite Nitrogen as N [3]	NA	Water/Liquid	9056A	0.0068	0.053	mg/L	10 MCL	NA	NA	NA
Perchlorate										
Perchlorate	NA	Water/Liquid	9056A Mod	0.019	0.10	mg/L	--	NA	NA	NA
Nitrocellulose (TestAmerica)										
Nitrocellulose as N	NA	Soil/Solid	WS-WC-0050	0.78	5	mg/kg	NA	--	--	--
Flashpoint										
Flashpoint	NA	Soil/Solid	1010A	NA	NA	degrees F	--	--	--	--
Glycols										
Ethylene Glycol	107-21-1	Soil/Solid	8015B Mod	0.23	10	mg/kg	NA	10	50000	100000
Propylene Glycol	57-55-6	Soil/Solid	8015D Mod	0.38	10	mg/kg	NA	--	--	--
Organochlorine Pesticides										
4,4´-DDD	72-54-8	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	56	125
4,4´-DDE	72-55-9	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	40	80
4,4´-DDT	50-29-3	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	15	88
a-Chlordane	5103-71-9	Soil/Solid	8081A	0.0021	0.040	mg/kg	NA	--	--	--
Aldrin	309-00-2	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	1	2
alpha-BHC	319-84-6	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	2	3.5
beta-BHC	319-85-7	Soil/Solid	8081A	0.0022	0.040	mg/kg	NA	--	7	15
delta-BHC	319-86-8	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	--	--
Dieldrin	60-57-1	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	0.8	2
Endosulfan I	959-98-8	Soil/Solid	8081A	0.0022	0.040	mg/kg	NA	--	--	--
Endosulfan II	891-86-1	Soil/Solid	8081A	0.0021	0.040	mg/kg	NA	--	--	--
Endosulfan sulfate	1031-07-8	Soil/Solid	8081A	0.0021	0.040	mg/kg	NA	--	--	--
Endrin	72-20-8	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	8	56
Endrin aldehyde	7421-93-4	Soil/Solid	8081A	0.0014	0.040	mg/kg	NA	--	--	--
Endrin ketone	53494-70-5	Soil/Solid	8081A	0.0021	0.040	mg/kg	NA	--	--	--
gamma-BHC (Lindane)	58-89-9	Soil/Solid	8081A	0.0021	0.040	mg/kg	NA	--	9	15
gamma-Chlordane	5566-34-7	Soil/Solid	8081A	0.0021	0.040	mg/kg	NA	--	--	--
Heptachlor	76-44-8	Soil/Solid	8081A	0.0021	0.040	mg/kg	NA	--	2	3.5
Heptachlor epoxide	1024-57-3	Soil/Solid	8081A	0.0020	0.040	mg/kg	NA	--	0.4	3
Methoxychlor	72-43-5	Soil/Solid	8081A	0.0021	0.040	mg/kg	NA	--	11	50
Toxaphene	8001-35-2	Soil/Solid	8081A	0.016	0.080	mg/kg	NA	--	13	28
Organochlorine Pesticides										
4,4´-DDD	72-54-8	Water/Liquid	8081A	0.031	0.40	ug/L	1 HRL93	NA	NA	NA
4,4´-DDE	72-55-9	Water/Liquid	8081A	0.031	0.40	ug/L	1 HRL93	NA	NA	NA
4,4´-DDT	50-29-3	Water/Liquid	8081A	0.031	0.40	ug/L	1 HRL93	NA	NA	NA
a-Chlordane	5103-71-9	Water/Liquid	8081A	0.033	0.40	ug/L	--	NA	NA	NA
Aldrin	309-00-2	Water/Liquid	8081A	0.028	0.40	ug/L	--	NA	NA	NA
alpha-BHC	319-84-6	Water/Liquid	8081A	0.032	0.40	ug/L	--	NA	NA	NA
beta-BHC	319-85-7	Water/Liquid	8081A	0.031	0.40	ug/L	--	NA	NA	NA
delta-BHC	319-86-8	Water/Liquid	8081A	0.030	0.40	ug/L	--	NA	NA	NA
Dieldrin	60-57-1	Water/Liquid	8081A	0.031	0.40	ug/L	0.006 HRL08(1)	NA	NA	NA
Endosulfan I	959-98-8	Water/Liquid	8081A	0.032	0.40	ug/L	--	NA	NA	NA
Endosulfan II	891-86-1	Water/Liquid	8081A	0.032	0.40	ug/L	--	NA	NA	NA
Endosulfan sulfate	1031-07-8	Water/Liquid	8081A	0.031	0.40	ug/L	--	NA	NA	NA
Endrin	72-20-8	Water/Liquid	8081A	0.033	0.40	ug/L	--	NA	NA	NA
Endrin aldehyde	7421-93-4	Water/Liquid	8081A	0.040	0.40	ug/L	--	NA	NA	NA
Endrin ketone	53494-70-5	Water/Liquid	8081A	0.031	0.40	ug/L	--	NA	NA	NA
gamma-BHC (Lindane)	58-89-9	Water/Liquid	8081A	0.029	0.40	ug/L	--	NA	NA	NA
gamma-Chlordane	5566-34-7	Water/Liquid	8081A	0.032	0.40	ug/L	--	NA	NA	NA

Table 1
Analytical Parameters, Methods and Quantitation Limits
Stage 1
UMore East Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	CAS Number	Matrix	Method (EPA unless noted otherwise)	Method Detection Limit	Reporting Limit	Test Unit	MN GW Values	Minnesota Tier SLV [4]	Minnesota SRV [5]	Minnesota Tier II Industrial SRV [6]
Heptachlor	76-44-8	Water/Liquid	8081A	0.031	0.40	ug/L	0.08 HRL93	NA	NA	NA
Heptachlor epoxide	1024-57-3	Water/Liquid	8081A	0.031	0.40	ug/L	0.04 HRL93	NA	NA	NA
Methoxychlor	72-43-5	Water/Liquid	8081A	0.029	0.40	ug/L	--	NA	NA	NA
Toxaphene	8001-35-2	Water/Liquid	8081A	0.29	1.0	ug/L	0.3 HRL93	NA	NA	NA
PCBs										
Aroclor 1016	12674-11-2	Soil/Solid	8082	0.0079	0.20	mg/kg	NA	--	--	--
Aroclor 1221	11104-28-2	Soil/Solid	8082	0.020	0.20	mg/kg	NA	--	--	--
Aroclor 1232	11141-16-5	Soil/Solid	8082	0.023	0.20	mg/kg	NA	--	--	--
Aroclor 1242	53469-21-9	Soil/Solid	8082	0.010	0.20	mg/kg	NA	--	--	--
Aroclor 1248	12672-29-6	Soil/Solid	8082	0.040	0.20	mg/kg	NA	--	--	--
Aroclor 1254	11097-69-1	Soil/Solid	8082	0.040	0.20	mg/kg	NA	--	--	--
Aroclor 1260	11096-82-5	Soil/Solid	8082	0.0059	0.20	mg/kg	NA	--	--	--
Polychlorinated Biphenyls	1336-36-3	Soil/Solid	8082	NA	NA	mg/kg	NA	2.1	1.2	8
VOCs - Soil/Solid										
1,1,1,2-Tetrachloroethane	630-20-6	Soil/Solid	8260B	0.059	0.25	mg/kg	NA	1.4	31	51
1,1,1-Trichloroethane	71-55-6	Soil/Solid	8260B	0.032	0.25	mg/kg	NA	3.5	140	472
1,1,2,2-Tetrachloroethane	79-34-5	Soil/Solid	8260B	0.026	0.25	mg/kg	NA	0.005	3.5	6.5
1,1,2-Trichloroethane	79-00-5	Soil/Solid	8260B	0.034	0.25	mg/kg	NA	0.010	9	14
1,1,2-Trichlorotrifluoroethane	76-13-1	Soil/Solid	8260B	0.068	0.25	mg/kg	NA	2580	3745	5430
1,1-Dichloroethane	75-34-3	Soil/Solid	8260B	0.023	0.25	mg/kg	NA	0.18	34	55
1,1-Dichloroethene	75-35-4	Soil/Solid	8260B	0.025	0.25	mg/kg	NA	0.025	20	60
1,1-Dichloropropene	563-58-6	Soil/Solid	8260B	0.021	0.25	mg/kg	NA	--	--	--
1,2,3-Trichlorobenzene	87-61-6	Soil/Solid	8260B	0.061	0.50	mg/kg	NA	--	--	--
1,2,3-Trichloropropane	96-18-4	Soil/Solid	8260B	0.024	0.25	mg/kg	NA	0.35	--	--
1,2,4-Trichlorobenzene	120-82-1	Soil/Solid	8260B	0.027	0.50	mg/kg	NA	0.31	200	985
1,2,4-Trimethylbenzene	95-63-6	Soil/Solid	8260B	0.018	0.25	mg/kg	NA	--	8	25
1,2-Dibromo-3-chloropropane	96-12-8	Soil/Solid	8260B	0.069	0.50	mg/kg	NA	0.001	--	--
1,2-Dibromoethane (EDB)	106-93-4	Soil/Solid	8260B	0.022	0.25	mg/kg	NA	0.00001	0.3	0.5
1,2-Dichlorobenzene	95-50-1	Soil/Solid	8260B	0.020	0.25	mg/kg	NA	8.1	26	75
1,2-Dichloroethane	107-06-2	Soil/Solid	8260B	0.027	0.25	mg/kg	NA	0.010	4	6
1,2-Dichloropropane	78-87-5	Soil/Solid	8260B	0.031	0.25	mg/kg	NA	0.011	4	6
1,3,5-Trimethylbenzene	108-67-8	Soil/Solid	8260B	0.020	0.25	mg/kg	NA	--	3	10
1,3-Dichlorobenzene	541-73-1	Soil/Solid	8260B	0.026	0.25	mg/kg	NA	4.2	26	200
1,3-Dichloropropane	142-28-9	Soil/Solid	8260B	0.022	0.25	mg/kg	NA	--	--	--
1,4-Dichlorobenzene	106-46-7	Soil/Solid	8260B	0.017	0.25	mg/kg	NA	0.13	30	50
2,2-Dichloropropane	594-20-7	Soil/Solid	8260B	0.060	0.50	mg/kg	NA	--	--	--
2-Butanone	78-93-3	Soil/Solid	8260B	0.088	2.0	mg/kg	NA	6.4	5500	19000
2-Chlorotoluene	95-49-8	Soil/Solid	8260B	0.014	0.25	mg/kg	NA	--	436	436
4-Chlorotoluene	106-43-4	Soil/Solid	8260B	0.021	0.25	mg/kg	NA	--	--	--
Acetone	67-64-1	Soil/Solid	8260B	0.19	2.0	mg/kg	NA	0.7	340	1000
Allyl chloride	107-05-1	Soil/Solid	8260B	0.058	0.50	mg/kg	NA	0.032	--	--
Benzene	71-43-2	Soil/Solid	8260B	0.015	0.25	mg/kg	NA	0.034	6	10
Bromobenzene	108-86-1	Soil/Solid	8260B	0.027	0.25	mg/kg	NA	--	--	--
Bromochloromethane	74-97-5	Soil/Solid	8260B	0.037	0.25	mg/kg	NA	0.15	--	--
Bromodichloromethane	75-27-4	Soil/Solid	8260B	0.027	0.25	mg/kg	NA	0.013	10	17
Bromoform	75-25-2	Soil/Solid	8260B	0.027	0.50	mg/kg	NA	0.14	370	650
Bromomethane	74-83-9	Soil/Solid	8260B	0.11	0.50	mg/kg	NA	0.5	0.7	2
Carbon tetrachloride	56-23-5	Soil/Solid	8260B	0.034	0.25	mg/kg	NA	0.023	0.3	0.9
Chlorobenzene	108-90-7	Soil/Solid	8260B	0.019	0.25	mg/kg	NA	1.1	11	32
Chloroethane	75-00-3	Soil/Solid	8260B	0.037	0.25	mg/kg	NA	--	1000	3000
Chloroform	67-66-3	Soil/Solid	8260B	0.021	0.25	mg/kg	NA	0.17	2.5	4
Chloromethane	74-87-3	Soil/Solid	8260B	0.061	0.25	mg/kg	NA	0.006	8	23
cis-1,2-Dichloroethene	156-59-2	Soil/Solid	8260B	0.026	0.25	mg/kg	NA	0.14	8	22
cis-1,3-Dichloropropene	10061-01-5	Soil/Solid	8260B	0.022	0.25	mg/kg	NA	0.005	--	--
Dibromochloromethane	124-48-1	Soil/Solid	8260B	0.034	0.25	mg/kg	NA	0.03	12	20
Dibromomethane	74-95-3	Soil/Solid	8260B	0.029	0.25	mg/kg	NA	--	260	1860
Dichlorodifluoromethane	75-71-8	Soil/Solid	8260B	0.099	0.50	mg/kg	NA	38	16	50
Dichlorofluoromethane	75-43-4	Soil/Solid	8260B	0.038	0.25	mg/kg	NA	--	--	--
Ethyl ether	60-29-7	Soil/Solid	8260B	0.038	0.50	mg/kg	NA	1.2	--	--
Ethylbenzene	100-41-4	Soil/Solid	8260B	0.011	0.25	mg/kg	NA	4.7	200	200
Hexachlorobutadiene	87-68-3	Soil/Solid	8260B	0.069	1.0	mg/kg	NA	25	6	37
Isopropylbenzene	98-82-8	Soil/Solid	8260B	0.019	0.25	mg/kg	NA	18	30	87
m,p-Xylene	108-38-3 / 106-42-3	Soil/Solid	8260B	0.047	0.50	mg/kg	NA	45 M	45 M	130 M
Methyl isobutyl ketone	108-10-1	Soil/Solid	8260B	0.082	0.50	mg/kg	NA	0.42	1700	9000
Methyl tert-butyl ether	1634-04-4	Soil/Solid	8260B	0.016	0.25	mg/kg	NA	0.027	--	--
Methylene chloride	75-09-2	Soil/Solid	8260B	0.058	1.0	mg/kg	NA	0.068	97	158
Naphthalene	91-20-3	Soil/Solid	8260B	0.055	0.50	mg/kg	NA	7.5	10	28
n-Butylbenzene	104-51-8	Soil/Solid	8260B	0.027	0.25	mg/kg	NA	--	30	92
n-Propylbenzene	103-65-1	Soil/Solid	8260B	0.021	0.25	mg/kg	NA	--	30	93
o-Xylene	95-47-6	Soil/Solid	8260B	0.025	0.25	mg/kg	NA	45 M	45 M	130 M
p-Isopropyltoluene	99-87-6	Soil/Solid	8260B	0.014	0.25	mg/kg	NA	--	--	--
sec-Butylbenzene	135-98-8	Soil/Solid	8260B	0.017	0.25	mg/kg	NA	--	25	70
Styrene	100-42-5	Soil/Solid	8260B	0.020	0.25	mg/kg	NA	1.9	210	600
tert-Butylbenzene	98-06-6	Soil/Solid	8260B	0.018	0.25	mg/kg	NA	--	30	90
Tetrachloroethene	127-18-4	Soil/Solid	8260B	0.028	0.25	mg/kg	NA	0.068	72	131
Tetrahydrofuran	109-99-9	Soil/Solid	8260B	0.12	2.0	mg/kg	NA	0.16	--	--
Toluene	108-88-3	Soil/Solid	8260B	0.016	0.25	mg/kg	NA	6.4	107	305
trans-1,2-Dichloroethene	156-60-5	Soil/Solid	8260B	0.030	0.25	mg/kg	NA	0.27	11	33

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trans-1,3-Dichloropropene	10061-02-6	Soil/Solid	8260B	0.023	0.25	mg/kg	NA	0.005	--	--
Trichloroethene	79-01-6	Soil/Solid	8260B	0.041	0.25	mg/kg	NA	0.14	29	46
Trichlorofluoromethane	75-69-4	Soil/Solid	8260B	0.035	0.25	mg/kg	NA	22	67	195
Vinyl chloride	75-01-4	Soil/Solid	8260B	0.065	0.25	mg/kg	NA	0.001	0.8	2.2
VOCs - Water/Liquid										
1,1,1,2-Tetrachloroethane	630-20-6	Water/Liquid	8260B	0.29	1.0	ug/L	70 HRL93	NA	NA	NA
1,1,1-Trichloroethane	71-55-6	Water/Liquid	8260B	0.16	1.0	ug/L	9000 HRL08(1)	NA	NA	NA
1,1,2,2-Tetrachloroethane	79-34-5	Water/Liquid	8260B	0.16	1.0	ug/L	2 HRL94	NA	NA	NA
1,1,2-Trichloroethane	79-00-5	Water/Liquid	8260B	0.19	1.0	ug/L	3 HRL93	NA	NA	NA
1,1,2-Trichlorotrifluoroethane	76-13-1	Water/Liquid	8260B	0.25	1.0	ug/L	200000 HRL93	NA	NA	NA
1,1-Dichloroethane	75-34-3	Water/Liquid	8260B	0.15	1.0	ug/L	100 RAA(1)	NA	NA	NA
1,1-Dichloroethene	75-35-4	Water/Liquid	8260B	0.24	1.0	ug/L	200 HBV09(1)	NA	NA	NA
1,1-Dichloropropene	563-58-6	Water/Liquid	8260B	0.14	1.0	ug/L	--	NA	NA	NA
1,2,3-Trichlorobenzene	87-61-6	Water/Liquid	8260B	0.35	5.0	ug/L	--	NA	NA	NA
1,2,3-Trichloropropane	96-18-4	Water/Liquid	8260B	0.22	2.5	ug/L	40 HBV10(1)	NA	NA	NA
1,2,4-Trichlorobenzene	120-82-1	Water/Liquid	8260B	0.30	5.0	ug/L	--	NA	NA	NA
1,2,4-Trimethylbenzene	95-63-6	Water/Liquid	8260B	0.072	1.0	ug/L	100 RAA10(1,2)	NA	NA	NA
1,2-Dibromo-3-chloropropane	96-12-8	Water/Liquid	8260B	0.40	5.0	ug/L	--	NA	NA	NA
1,2-Dibromoethane (EDB)	106-93-4	Water/Liquid	8260B	0.15	2.5	ug/L	0.004 HRL93	NA	NA	NA
1,2-Dichlorobenzene	95-50-1	Water/Liquid	8260B	0.12	1.0	ug/L	600 HRL93	NA	NA	NA
1,2-Dichloroethane	107-06-2	Water/Liquid	8260B	0.26	1.0	ug/L	1 HBV11(1)	NA	NA	NA
1,2-Dichloropropane	78-87-5	Water/Liquid	8260B	0.19	1.0	ug/L	5 HRL94	NA	NA	NA
1,3,5-Trimethylbenzene	108-67-8	Water/Liquid	8260B	0.11	1.0	ug/L	100 HRL08(1,2)	NA	NA	NA
1,3-Dichlorobenzene	541-73-1	Water/Liquid	8260B	0.15	1.0	ug/L	--	NA	NA	NA
1,3-Dichloropropane	142-28-9	Water/Liquid	8260B	0.16	1.0	ug/L	2 HRL94	NA	NA	NA
1,4-Dichlorobenzene	106-46-7	Water/Liquid	8260B	0.081	1.0	ug/L	10 HRL94	NA	NA	NA
2,2-Dichloropropane	594-20-7	Water/Liquid	8260B	0.66	5.0	ug/L	--	NA	NA	NA
2-Butanone	78-93-3	Water/Liquid	8260B	0.65	20	ug/L	4000 HRL94	NA	NA	NA
2-Chlorotoluene	95-49-8	Water/Liquid	8260B	0.081	1.0	ug/L	--	NA	NA	NA
4-Chlorotoluene	106-43-4	Water/Liquid	8260B	0.11	1.0	ug/L	--	NA	NA	NA
Acetone	67-64-1	Water/Liquid	8260B	3.8	20	ug/L	4000 HBV10(1)	NA	NA	NA
Allyl chloride	107-05-1	Water/Liquid	8260B	0.51	5.0	ug/L	30 HRL94	NA	NA	NA
Benzene	71-43-2	Water/Liquid	8260B	0.071	1.0	ug/L	2 HRL08(1)	NA	NA	NA
Bromobenzene	108-86-1	Water/Liquid	8260B	0.082	1.0	ug/L	--	NA	NA	NA
Bromochloromethane	74-97-5	Water/Liquid	8260B	0.20	1.0	ug/L	--	NA	NA	NA
Bromodichloromethane	75-27-4	Water/Liquid	8260B	0.23	1.0	ug/L	6 HRL93	NA	NA	NA
Bromoform	75-25-2	Water/Liquid	8260B	0.26	5.0	ug/L	40 HRL93	NA	NA	NA
Bromomethane	74-83-9	Water/Liquid	8260B	0.32	5.0	ug/L	10 HRL93	NA	NA	NA
Carbon tetrachloride	56-23-5	Water/Liquid	8260B	0.15	1.0	ug/L	1 HBV10(1)	NA	NA	NA
Chlorobenzene	108-90-7	Water/Liquid	8260B	0.20	1.0	ug/L	100 HRL93	NA	NA	NA
Chloroethane	75-00-3	Water/Liquid	8260B	0.27	2.5	ug/L	--	NA	NA	NA
Chloroform	67-66-3	Water/Liquid	8260B	0.15	1.0	ug/L	30 HRL08(1,2)	NA	NA	NA
Chloromethane	74-87-3	Water/Liquid	8260B	0.24	2.5	ug/L	--	NA	NA	NA
cis-1,2-Dichloroethene	156-59-2	Water/Liquid	8260B	0.12	1.0	ug/L	50 HRL08(1)	NA	NA	NA
cis-1,3-Dichloropropene	10061-01-5	Water/Liquid	8260B	0.18	1.0	ug/L	--	NA	NA	NA
Dibromochloromethane	124-48-1	Water/Liquid	8260B	0.26	2.5	ug/L	--	NA	NA	NA
Dibromomethane	74-95-3	Water/Liquid	8260B	0.24	2.5	ug/L	--	NA	NA	NA
Dichlorodifluoromethane	75-71-8	Water/Liquid	8260B	0.25	5.0	ug/L	700 HBV09(1)	NA	NA	NA
Dichlorofluoromethane	75-43-4	Water/Liquid	8260B	0.21	1.0	ug/L	--	NA	NA	NA
Ethyl ether	60-29-7	Water/Liquid	8260B	0.27	5.0	ug/L	200 RAA10(1)	NA	NA	NA
Ethylbenzene	100-41-4	Water/Liquid	8260B	0.28	1.0	ug/L	50 HBV10(2)	NA	NA	NA
Hexachlorobutadiene	87-68-3	Water/Liquid	8260B	0.42	10	ug/L	1 HRL93	NA	NA	NA
Isopropylbenzene	98-82-8	Water/Liquid	8260B	0.12	1.0	ug/L	300 HRL93	NA	NA	NA
m,p-Xylene	108-38-3 / 106-42-3	Water/Liquid	8260B	0.57	2.0	ug/L	300 HRL10(1,2)	NA	NA	NA
Methyl isobutyl ketone	108-10-1	Water/Liquid	8260B	0.40	5.0	ug/L	300 HRL94	NA	NA	NA
Methyl tert-butyl ether	1634-04-4	Water/Liquid	8260B	0.16	1.0	ug/L	--	NA	NA	NA
Methylene chloride	75-09-2	Water/Liquid	8260B	0.85	5.0	ug/L	5 MCL	NA	NA	NA
Naphthalene	91-20-3	Water/Liquid	8260B	0.30	5.0	ug/L	300 HRL94	NA	NA	NA
n-Butylbenzene	104-51-8	Water/Liquid	8260B	0.15	2.5	ug/L	--	NA	NA	NA
n-Propylbenzene	103-65-1	Water/Liquid	8260B	0.094	1.0	ug/L	--	NA	NA	NA
o-Xylene	95-47-6	Water/Liquid	8260B	0.19	1.0	ug/L	300 HRL10(1,2)	NA	NA	NA
p-Isopropyltoluene	99-87-6	Water/Liquid	8260B	0.14	2.5	ug/L	--	NA	NA	NA
sec-Butylbenzene	135-98-8	Water/Liquid	8260B	0.11	1.0	ug/L	--	NA	NA	NA
Styrene	100-42-5	Water/Liquid	8260B	0.21	1.0	ug/L	--	NA	NA	NA
tert-Butylbenzene	98-06-6	Water/Liquid	8260B	0.091	1.0	ug/L	--	NA	NA	NA
Tetrachloroethene	127-18-4	Water/Liquid	8260B	0.28	1.0	ug/L	5 MCL	NA	NA	NA
Tetrahydrofuran	109-99-9	Water/Liquid	8260B	0.75	20	ug/L	--	NA	NA	NA
Toluene	108-88-3	Water/Liquid	8260B	0.10	1.0	ug/L	1000 HBV09	NA	NA	NA
trans-1,2-Dichloroethene	156-60-5	Water/Liquid	8260B	0.056	1.0	ug/L	100 HBV11(1)	NA	NA	NA
trans-1,3-Dichloropropene	10061-02-6	Water/Liquid	8260B	0.14	1.0	ug/L	--	NA	NA	NA
Trichloroethene	79-01-6	Water/Liquid	8260B	0.18	1.0	ug/L	5 MCL	NA	NA	NA
Trichlorofluoromethane	75-69-4	Water/Liquid	8260B	0.29	1.0	ug/L	2000 HRL93	NA	NA	NA
Vinyl chloride	75-01-4	Water/Liquid	8260B	0.21	1.0	ug/L	0.2 HRL08(1)	NA	NA	NA

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SemiVolatile Organics										
1,2,4-Trichlorobenzene	120-82-1	Soil/Solid	8270C	0.031	0.33	mg/kg	NA	0.31	200	985
1,2-Dichlorobenzene	95-50-1	Soil/Solid	8270C	0.033	0.33	mg/kg	NA	8.1	26	75
1,2-Diphenylhydrazine as Azobenzene	103-33-3	Soil/Solid	8270C	0.036	0.33	mg/kg	NA	--	--	--
1,3-Dichlorobenzene	541-73-1	Soil/Solid	8270C	0.031	0.33	mg/kg	NA	4.2	26	200
1,4-Dichlorobenzene	106-46-7	Soil/Solid	8270C	0.029	0.33	mg/kg	NA	0.13	30	50
2,3,4,6-Tetrachlorophenol	58-90-2	Soil/Solid	8270C	0.078	0.67	mg/kg	NA	--	636	3700
2,4,5-Trichlorophenol	95-95-4	Soil/Solid	8270C	0.037	0.67	mg/kg	NA	--	1920	10600
2,4,6-Trichlorophenol	88-06-2	Soil/Solid	8270C	0.080	0.67	mg/kg	NA	0.21	595	1060
2,4-Dichlorophenol	120-83-2	Soil/Solid	8270C	0.075	0.67	mg/kg	NA	0.076	48	230
2,4-Dimethylphenol	105-67-9	Soil/Solid	8270C	0.080	0.67	mg/kg	NA	0.34	390	1925
2,4-Dinitrophenol	51-28-5	Soil/Solid	8270C	0.032	0.67	mg/kg	NA	0.014	--	--
2,4-Dinitrotoluene	121-14-2	Soil/Solid	8270C	0.035	0.33	mg/kg	NA	0.001	50	355
2,6-Dichlorophenol	87-65-0	Soil/Solid	8270C	0.069	0.67	mg/kg	NA	--	--	--
2,6-Dinitrotoluene	606-20-2	Soil/Solid	8270C	0.039	0.33	mg/kg	NA	0.001	25	175
2-Chloronaphthalene	91-58-7	Soil/Solid	8270C	0.034	0.33	mg/kg	NA	--	--	--
2-Chlorophenol	95-57-8	Soil/Solid	8270C	0.060	0.67	mg/kg	NA	0.26	--	--
2-Methylnaphthalene	91-57-6	Soil/Solid	8270C	0.036	0.33	mg/kg	NA	--	100	369
2-Methylphenol	95-48-7	Soil/Solid	8270C	0.034	0.67	mg/kg	NA	0.064	75	352
2-Nitroaniline	88-74-4	Soil/Solid	8270C	0.038	0.33	mg/kg	NA	--	--	--
2-Nitrophenol	88-75-5	Soil/Solid	8270C	0.085	0.67	mg/kg	NA	0.60	--	--
3&4-Methylphenol	108-39-4	Soil/Solid	8270C	0.032	0.67	mg/kg	NA	0.033 MP	10 MP	59 MP
3,3´-Dichlorobenzidine	91-94-1	Soil/Solid	8270C	0.15	1.6	mg/kg	NA	0.36	25	50
3-Nitroaniline	99-09-2	Soil/Solid	8270C	0.027	0.33	mg/kg	NA	--	--	--
4,6-Dinitro-2-methylphenol	534-52-1	Soil/Solid	8270C	0.034	0.67	mg/kg	NA	--	--	--
4-Bromophenyl phenyl ether	101-55-3	Soil/Solid	8270C	0.033	0.33	mg/kg	NA	--	--	--
4-Chloro-3-methylphenol	59-50-7	Soil/Solid	8270C	0.066	0.67	mg/kg	NA	--	--	--
4-Chloroaniline	106-47-8	Soil/Solid	8270C	0.027	0.67	mg/kg	NA	--	--	--
4-Chlorophenyl phenyl ether	7005-72-3	Soil/Solid	8270C	0.036	0.33	mg/kg	NA	--	--	--
4-Nitroaniline	100-01-6	Soil/Solid	8270C	0.034	0.33	mg/kg	NA	--	--	--
4-Nitrophenol	100-02-7	Soil/Solid	8270C	0.093	0.67	mg/kg	NA	--	--	--
Acenaphthene	83-32-9	Soil/Solid	8270C	0.038	0.33	mg/kg	NA	50	1200	5260
Acenaphthylene	208-96-8	Soil/Solid	8270C	0.041	0.33	mg/kg	NA	--	--	--
Aniline	62-53-3	Soil/Solid	8270C	0.031	0.67	mg/kg	NA	--	--	--
Anthracene	120-12-7	Soil/Solid	8270C	0.036	0.33	mg/kg	NA	942	7880	45400
Benzidine	92-87-5	Soil/Solid	8270C	0.19	2.5	mg/kg	NA	--	--	--
Benzo (a) anthracene	56-55-3	Soil/Solid	8270C	0.041	0.33	mg/kg	NA	10.2 T	2 T	3 T
Benzo (a) pyrene	50-32-8	Soil/Solid	8270C	0.042	0.33	mg/kg	NA	10.2 T	2 T	3 T
Benzo (b) fluoranthene	205-99-2	Soil/Solid	8270C	0.043	0.33	mg/kg	NA	10.2 T	2 T	3 T
Benzo (g,h,i) perylene	191-24-2	Soil/Solid	8270C	0.043	0.33	mg/kg	NA	--	--	--
Benzo (k) fluoranthene	207-08-9	Soil/Solid	8270C	0.048	0.33	mg/kg	NA	10.2 T	2 T	3 T
Benzoic acid	65-85-0	Soil/Solid	8270C	0.024	0.33	mg/kg	NA	30	50000	100000
Benzyl alcohol	100-51-6	Soil/Solid	8270C	0.095	0.67	mg/kg	NA	--	8700	56000
Bis(2-chloroethoxy)methane	111-91-1	Soil/Solid	8270C	0.034	0.33	mg/kg	NA	--	--	--
Bis(2-chloroethyl)ether	111-44-4	Soil/Solid	8270C	0.035	0.33	mg/kg	NA	0.001	2.5	5
Bis(2-chloroisopropyl)ether	39638-32-9	Soil/Solid	8270C	0.034	0.33	mg/kg	NA	--	--	--
Bis(2-ethylhexyl)phthalate	117-81-7	Soil/Solid	8270C	0.048	0.33	mg/kg	NA	40	570	2100
Butyl benzyl phthalate	85-68-7	Soil/Solid	8270C	0.042	0.33	mg/kg	NA	28	580	3700
Carbazole	86-74-8	Soil/Solid	8270C	0.035	0.33	mg/kg	NA	--	700	1310
Chrysene	218-01-9	Soil/Solid	8270C	0.044	0.33	mg/kg	NA	10.2 T	2 T	3 T
Dibenz (a,h) anthracene	53-70-3	Soil/Solid	8270C	0.043	0.33	mg/kg	NA	10.2 T	2 T	3 T
Dibenzofuran	132-64-9	Soil/Solid	8270C	0.033	0.33	mg/kg	NA	--	104	810
Diethyl phthalate	84-66-2	Soil/Solid	8270C	0.031	0.33	mg/kg	NA	18	--	--
Dimethyl phthalate	131-11-3	Soil/Solid	8270C	0.030	0.33	mg/kg	NA	172	--	--
Di-n-butyl phthalate	84-74-2	Soil/Solid	8270C	0.044	0.33	mg/kg	NA	23	2440	16300
Di-n-octyl phthalate	117-84-0	Soil/Solid	8270C	0.048	0.33	mg/kg	NA	--	520	3700
Fluoranthene	206-44-0	Soil/Solid	8270C	0.039	0.33	mg/kg	NA	295	1080	6800
Fluorene	86-73-7	Soil/Solid	8270C	0.035	0.33	mg/kg	NA	47	850	4120
Hexachlorobenzene	118-74-1	Soil/Solid	8270C	0.036	0.33	mg/kg	NA	0.32	5	9
Hexachlorobutadiene	87-68-3	Soil/Solid	8270C	0.030	0.33	mg/kg	NA	25	6	37
Hexachlorocyclopentadiene	77-47-4	Soil/Solid	8270C	0.024	0.33	mg/kg	NA	4.4	2	6
Hexachloroethane	67-72-1	Soil/Solid	8270C	0.030	0.33	mg/kg	NA	0.050	--	--
Indeno (1,2,3-cd) pyrene	193-39-5	Soil/Solid	8270C	0.040	0.33	mg/kg	NA	10.2 T	2 T	3 T
Isophorone	78-59-1	Soil/Solid	8270C	0.031	0.33	mg/kg	NA	0.16	--	--
Naphthalene	91-20-3	Soil/Solid	8270C	0.033	0.33	mg/kg	NA	7.5	10	28
Nitrobenzene	98-95-3	Soil/Solid	8270C	0.033	0.33	mg/kg	NA	--	--	--
N-Nitrosodimethylamine	62-75-9	Soil/Solid	8270C	0.022	0.33	mg/kg	NA	0.82	--	--
N-Nitrosodi-n-propylamine	621-64-7	Soil/Solid	8270C	0.033	0.33	mg/kg	NA	--	0.7	1.2
N-Nitrosodiphenylamine ***	86-30-6	Soil/Solid	8270C	0.026	0.33	mg/kg	NA	0.88	1950	3720
Diphenylamine ***	122-39-4	Soil/Solid	8270C	--	--	--	NA	--	--	--
Pentachlorophenol	87-86-5	Soil/Solid	8270C	0.078	0.67	mg/kg	NA	0.034	80	120
Phenanthrene	85-01-8	Soil/Solid	8270C	0.036	0.33	mg/kg	NA	--	--	--
Phenol	108-95-2	Soil/Solid	8270C	0.069	0.67	mg/kg	NA	7.8	1500	20203
Pyrene	129-00-0	Soil/Solid	8270C	0.041	0.33	mg/kg	NA	272	890	5800

Table 1
Analytical Parameters, Methods and Quantitation Limits
Stage 1
UMore East Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	CAS Number	Matrix	Method (EPA unless noted otherwise)	Method Detection Limit	Reporting Limit	Test Unit	MN GW Values	Minnesota Tier SLV [4]	Minnesota SRV [5]	Minnesota Tier II Industrial SRV [6]
SemiVolatile Organics										
1,2,4-Trichlorobenzene	120-82-1	Water/Liquid	8270C	0.30	10	ug/L	--	NA	NA	NA
1,2-Dichlorobenzene	95-50-1	Water/Liquid	8270C	0.33	10	ug/L	600 HRL93	NA	NA	NA
1,2-Diphenylhydrazine as Azobenzene	103-33-3	Water/Liquid	8270C	0.26	10	ug/L	--	NA	NA	NA
1,3-Dichlorobenzene	541-73-1	Water/Liquid	8270C	0.31	10	ug/L	--	NA	NA	NA
1,4-Dichlorobenzene	106-46-7	Water/Liquid	8270C	0.29	10	ug/L	10 HRL94	NA	NA	NA
2,3,4,6-Tetrachlorophenol	58-90-2	Water/Liquid	8270C	0.96	10	ug/L	--	NA	NA	NA
2,4,5-Trichlorophenol	95-95-4	Water/Liquid	8270C	0.59	10	ug/L	--	NA	NA	NA
2,4,6-Trichlorophenol	88-06-2	Water/Liquid	8270C	0.59	10	ug/L	30 HRL93	NA	NA	NA
2,4-Dichlorophenol	120-83-2	Water/Liquid	8270C	0.63	10	ug/L	20 HRL93	NA	NA	NA
2,4-Dimethylphenol	105-67-9	Water/Liquid	8270C	0.57	10	ug/L	100 HRL93	NA	NA	NA
2,4-Dinitrophenol	51-28-5	Water/Liquid	8270C	1.2	10	ug/L	10 HRL94	NA	NA	NA
2,4-Dinitrotoluene	121-14-2	Water/Liquid	8270C	0.45	10	ug/L	--	NA	NA	NA
2,6-Dichlorophenol	87-65-0	Water/Liquid	8270C	0.50	10	ug/L	--	NA	NA	NA
2,6-Dinitrotoluene	606-20-2	Water/Liquid	8270C	0.53	10	ug/L	--	NA	NA	NA
2-Chloronaphthalene	91-58-7	Water/Liquid	8270C	0.60	10	ug/L	--	NA	NA	NA
2-Chlorophenol	95-57-8	Water/Liquid	8270C	0.39	10	ug/L	30 HRL93	NA	NA	NA
2-Methylnaphthalene	91-57-6	Water/Liquid	8270C	0.88	10	ug/L	--	NA	NA	NA
2-Methylphenol	95-48-7	Water/Liquid	8270C	0.48	10	ug/L	30 HRL93	NA	NA	NA
2-Nitroaniline	88-74-4	Water/Liquid	8270C	0.60	10	ug/L	--	NA	NA	NA
2-Nitrophenol	88-75-5	Water/Liquid	8270C	0.93	10	ug/L	--	NA	NA	NA
3&4-Methylphenol	108-39-4/106-44-5	Water/Liquid	8270C	0.41	10	ug/L	3 MP	NA	NA	NA
3,3´-Dichlorobenzidine	91-94-1	Water/Liquid	8270C	7.2	25	ug/L	0.8 HRL93	NA	NA	NA
3-Nitroaniline	99-09-2	Water/Liquid	8270C	1.1	10	ug/L	--	NA	NA	NA
4,6-Dinitro-2-methylphenol	534-52-1	Water/Liquid	8270C	1.0	10	ug/L	--	NA	NA	NA
4-Bromophenyl phenyl ether	101-55-3	Water/Liquid	8270C	0.42	10	ug/L	--	NA	NA	NA
4-Chloro-3-methylphenol	59-50-7	Water/Liquid	8270C	0.48	10	ug/L	--	NA	NA	NA
4-Chloroaniline	106-47-8	Water/Liquid	8270C	1.3	10	ug/L	--	NA	NA	NA
4-Chlorophenyl phenyl ether	7005-72-3	Water/Liquid	8270C	0.31	10	ug/L	--	NA	NA	NA
4-Nitroaniline	100-01-6	Water/Liquid	8270C	0.90	10	ug/L	--	NA	NA	NA
4-Nitrophenol	100-02-7	Water/Liquid	8270C	0.78	10	ug/L	--	NA	NA	NA
Acenaphthene	83-32-9	Water/Liquid	8270C	0.60	10	ug/L	400 HRL93	NA	NA	NA
Acenaphthylene	208-96-8	Water/Liquid	8270C	0.55	10	ug/L	--	NA	NA	NA
Aniline	62-53-3	Water/Liquid	8270C	1.6	10	ug/L	--	NA	NA	NA
Anthracene	120-12-7	Water/Liquid	8270C	0.56	10	ug/L	2000 HRL93	NA	NA	NA
Benzidine	92-87-5	Water/Liquid	8270C	7.6	100	ug/L	--	NA	NA	NA
Benzo (a) anthracene	56-55-3	Water/Liquid	8270C	0.50	10	ug/L	--	NA	NA	NA
Benzo (a) pyrene	50-32-8	Water/Liquid	8270C	0.41	10	ug/L	--	NA	NA	NA
Benzo (b) fluoranthene	205-99-2	Water/Liquid	8270C	0.44	10	ug/L	--	NA	NA	NA
Benzo (g,h,i) perylene	191-24-2	Water/Liquid	8270C	0.46	10	ug/L	--	NA	NA	NA
Benzo (k) fluoranthene	207-08-9	Water/Liquid	8270C	0.49	10	ug/L	--	NA	NA	NA
Benzoic acid	65-85-0	Water/Liquid	8270C	0.52	10	ug/L	30000 HRL93	NA	NA	NA
Benzyl alcohol	100-51-6	Water/Liquid	8270C	1.2	10	ug/L	--	NA	NA	NA
Bis(2-chloroethoxy)methane	111-91-1	Water/Liquid	8270C	0.27	10	ug/L	--	NA	NA	NA
Bis(2-chloroethyl)ether	111-44-4	Water/Liquid	8270C	0.22	10	ug/L	0.3 HRL93	NA	NA	NA
Bis(2-chloroisopropyl)ether	39638-32-9	Water/Liquid	8270C	0.23	10	ug/L	--	NA	NA	NA
Bis(2-ethylhexyl)phthalate	117-81-7	Water/Liquid	8270C	0.81	10	ug/L	6 MCL	NA	NA	NA
Butyl benzyl phthalate	85-68-7	Water/Liquid	8270C	0.45	10	ug/L	100 HRL93	NA	NA	NA
Carbazole	86-74-8	Water/Liquid	8270C	0.33	10	ug/L	--	NA	NA	NA
Chrysene	218-01-9	Water/Liquid	8270C	0.61	10	ug/L	--	NA	NA	NA
Dibenz (a,h) anthracene	53-70-3	Water/Liquid	8270C	0.55	10	ug/L	--	NA	NA	NA
Dibenzofuran	132-64-9	Water/Liquid	8270C	0.59	10	ug/L	--	NA	NA	NA
Diethyl phthalate	84-66-2	Water/Liquid	8270C	0.30	10	ug/L	6000 HRL93	NA	NA	NA
Dimethyl phthalate	131-11-3	Water/Liquid	8270C	0.33	10	ug/L	70000 HRL94	NA	NA	NA
Di-n-butyl phthalate	84-74-2	Water/Liquid	8270C	0.39	10	ug/L	700 HRL	NA	NA	NA
Di-n-octyl phthalate	117-84-0	Water/Liquid	8270C	0.67	10	ug/L	--	NA	NA	NA
Fluoranthene	206-44-0	Water/Liquid	8270C	0.61	10	ug/L	300 HRL93	NA	NA	NA
Fluorene	86-73-7	Water/Liquid	8270C	0.52	10	ug/L	300 HRL93	NA	NA	NA
Hexachlorobenzene	118-74-1	Water/Liquid	8270C	0.40	10	ug/L	0.2 HRL93	NA	NA	NA
Hexachlorobutadiene	87-68-3	Water/Liquid	8270C	0.41	10	ug/L	1 HRL93	NA	NA	NA
Hexachlorocyclopentadiene	77-47-4	Water/Liquid	8270C	0.35	10	ug/L	--	NA	NA	NA
Hexachloroethane	67-72-1	Water/Liquid	8270C	0.33	10	ug/L	--	NA	NA	NA
Indeno (1,2,3-cd) pyrene	193-39-5	Water/Liquid	8270C	0.39	10	ug/L	--	NA	NA	NA
Isophorone	78-59-1	Water/Liquid	8270C	0.26	10	ug/L	100 HRL93	NA	NA	NA
Naphthalene	91-20-3	Water/Liquid	8270C	0.52	10	ug/L	300 HRL94	NA	NA	NA
Nitrobenzene	98-95-3	Water/Liquid	8270C	0.37	10	ug/L	--	NA	NA	NA
N-Nitrosodimethylamine	62-75-9	Water/Liquid	8270C	0.48	10	ug/L	--	NA	NA	NA
N-Nitrosodi-n-propylamine	621-64-7	Water/Liquid	8270C	0.25	10	ug/L	--	NA	NA	NA
N-Nitrosodiphenylamine ***	86-30-6	Water/Liquid	8270C	0.38	10	ug/L	70 HRL93	NA	NA	NA
Diphenylamine ***	122-39-4	Water/Liquid	8270C	--	--	--	--	NA	NA	NA
Pentachlorophenol	87-86-5	Water/Liquid	8270C	0.90	10	ug/L	1 MCL	NA	NA	NA
Phenanthrene	85-01-8	Water/Liquid	8270C	0.35	10	ug/L	--	NA	NA	NA
Phenol	108-95-2	Water/Liquid	8270C	0.41	10	ug/L	4000 HRL93	NA	NA	NA
Pyrene	129-00-0	Water/Liquid	8270C	0.66	10	ug/L	200 HRL93	NA	NA	NA

Table 1
Analytical Parameters, Methods and Quantitation Limits
Stage 1
UMore East Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	CAS Number	Matrix	Method (EPA unless noted otherwise)	Method Detection Limit	Reporting Limit	Test Unit	MN GW Values	Minnesota Tier SLV [4]	Minnesota SRV [5]	Minnesota Tier II Industrial SRV [6]
Explosives										
1,3,5-Trinitrobenzene	99-35-4	Soil/Solid	8330 Mod	0.043	1.0	mg/kg	NA	--	610	3760
1,3-Dinitrotoluene	99-65-0	Soil/Solid	8330 Mod	0.047	1.0	mg/kg	NA	0.002	2	13
2,4,6-Trinitrotoluene	118-96-7	Soil/Solid	8330 Mod	0.13	1.0	mg/kg	NA	0.014	10	63
2,4-Dinitrotoluene	121-14-2	Soil/Solid	8330 Mod	0.039	1.0	mg/kg	NA	0.001	50	355
2,6-Dinitrotoluene	606-20-2	Soil/Solid	8330 Mod	0.089	1.0	mg/kg	NA	0.001	25	175
2-Amino-4,6-Dinitrotoluene	35572-78-2	Soil/Solid	8330 Mod	0.072	1.0	mg/kg	NA	--	--	--
2-Nitrotoluene	88-72-2	Soil/Solid	8330 Mod	0.16	1.0	mg/kg	NA	--	--	--
3-Nitrotoluene	99-08-1	Soil/Solid	8330 Mod	0.24	1.0	mg/kg	NA	--	--	--
4-Amino-2,6-Dinitrotoluene	1946-51-0	Soil/Solid	8330 Mod	0.075	1.0	mg/kg	NA	--	--	--
4-Nitrotoluene	99-99-0	Soil/Solid	8330 Mod	0.23	1.0	mg/kg	NA	--	--	--
HMX	2691-41-0	Soil/Solid	8330 Mod	0.11	1.0	mg/kg	NA	--	1360	9560
Nitrobenzene	98-95-3	Soil/Solid	8330 Mod	0.066	1.0	mg/kg	NA	--	--	--
RDX	121-82-4	Soil/Solid	8330 Mod	0.078	1.0	mg/kg	NA	0.050	35	75
Tetryl	479-45-8	Soil/Solid	8330 Mod	0.17	1.0	mg/kg	NA	--	--	--
Explosives										
1,3,5-Trinitrobenzene	99-35-4	Water/Liquid	8330 Mod	0.19	4	ug/L	0.3 HRL93	NA	NA	NA
1,3-Dinitrotoluene	99-65-0	Water/Liquid	8330 Mod	0.38	4	ug/L	--	NA	NA	NA
2,4,6-Trinitrotoluene	118-96-7	Water/Liquid	8330 Mod	0.39	4	ug/L	--	NA	NA	NA
2,4-Dinitrotoluene	121-14-2	Water/Liquid	8330 Mod	0.34	4	ug/L	--	NA	NA	NA
2,6-Dinitrotoluene	606-20-2	Water/Liquid	8330 Mod	0.57	4	ug/L	--	NA	NA	NA
2-Amino-4,6-Dinitrotoluene	35572-78-2	Water/Liquid	8330 Mod	0.43	4	ug/L	--	NA	NA	NA
2-Nitrotoluene	88-72-2	Water/Liquid	8330 Mod	0.81	4	ug/L	--	NA	NA	NA
3-Nitrotoluene	99-08-1	Water/Liquid	8330 Mod	0.61	4	ug/L	--	NA	NA	NA
4-Amino-2,6-Dinitrotoluene	1946-51-0	Water/Liquid	8330 Mod	0.51	4	ug/L	--	NA	NA	NA
4-Nitrotoluene	99-99-0	Water/Liquid	8330 Mod	1.1	4	ug/L	--	NA	NA	NA
HMX	2691-41-0	Water/Liquid	8330 Mod	0.38	4	ug/L	--	NA	NA	NA
Nitrobenzene	98-95-3	Water/Liquid	8330 Mod	0.62	4	ug/L	--	NA	NA	NA
RDX	121-82-4	Water/Liquid	8330 Mod	0.48	4	ug/L	--	NA	NA	NA
Tetryl	479-45-8	Water/Liquid	8330 Mod	0.62	4	ug/L	--	NA	NA	NA

Notes:

M the values with this notation indicate the limit is for all combined isomers of this compound

T the values with this notation represent the limit for the total carcinogenic PAHs as BaP

MC Mercury as mercuric chloride

MP Value represents the criteria for p-cresol (4-Methylphenol)

(TA) Legend Technical Services, Inc. will subcontract this analysis to Test America, West Sacramento, California.

CR Value represents the criteria for Chromium, hexavalent.

HRL Minnesota Health Risk Limits

HBV Minnesota Health Based Values

RAA Minnesota Risk Assessment Advice

MCL EPA Maximum Contaminant Levels

(1) Value is representative of the lowest exposure duration published in the Minnesota Department of Health Groundwater Values Table.

(2) Set at short term HRL.

[3] Nitrate + nitrite nitrogen as N result was calculated by analyzing nitrate as N and Nitrite as N separately then calculating a combined nitrate + nitrite nitrogen as N value.

[4] - Minnesota Pollution Control Agency's Risk-based guidance for Soil - Soil Leaching Value (SLV)

[5] - Minnesota Pollution Control Agency's Risk-based guidance for Soil - Soil Reference Value (SRV)

[6] - Minnesota Pollution Control Agency's Risk-based guidance for Soil - Tier II Industrial SRV.

*** Diphenylamine cannot be separated from n-nitrosodiphenylamine under current lab instrumentation conditions.
If detections of n-nitrosodiphenylamine occurr, confirmational analysis may be considered.

Table 2

Data Quality Objectives
Quality Assurance Project Plan,
UMore East Remedial Investigation
Dakota County, MN

Step 1: Identify the Problem	Step 2: Identify the Decisions	Step 3: Identify Inputs to the Decisions	Step 4: Define the Study Boundaries	Step 5: Develop a Decision Rule	Step 6: Specify Limits on Decision Errors	Step 7: Optimize the Design for Obtaining Data
Category 1: Characterization is needed to determine the extent and magnitude of hazardous substances or petroleum products released to the soil.	**Study Question:** What is the extent of soil impacts that exceed TCBs? **Alternative actions:** - No further investigation - Additional investigation **Decision Statement:** Determine the extent and magnitude COCs present in soil.	1. New and existing validated measurements of COC concentrations in soil samples collected from the Remedial Investigation project area.	The SOC boundaries are included in the Work Plan (Barr, 2011).	If concentrations of COCs in soil samples exceed TCBs, further sampling and analysis will be conducted to determine magnitude and extent of the COC-impacted soils.	Potential sampling errors include sampling design errors and measurement errors. Design errors are countered with an adequate number of sampling points and measurement errors are minimized through systematic uniform management of each of the steps of measurement using SOPs and by following the QAPP.	Samples of soil will be collected and analyzed for COCs listed in the work plan. Surface and subsurface soils will be field screened for organic vapors. If elevated organic vapors are detected or other evidence of a release of hazardous substances are observed, samples for COCs will be collected from the interval demonstrating the most significant indications of the release. Sampling locations, depths, and methods are discussed in the Work Plan and FSP.
Category 2 and 3: Characterization is needed to determine if hazardous substances or petroleum products have been released to the soil.	**Study Question:** Are COCs present in soil at concentrations above TCBs? **Alternative actions:** - No further investigation - Additional investigation **Decision Statement:** Determine if COCs are present in soil and at what concentrations.	1. New and existing validated measurements of COC concentrations in soil samples collected from the Remedial Investigation project area.	The SOC boundaries are included in the Work Plan (Barr, 2011).	If concentrations of COCs in soil samples exceed TCBs, further sampling and analysis will be conducted to determine magnitude and extent of the COC-impacted soils.	Potential sampling errors include sampling design errors and measurement errors. Design errors are countered with an adequate number of sampling points and measurement errors are minimized through systematic uniform management of each of the steps of measurement using SOPs and by following the QAPP.	Samples of soil will be collected from test trenches, soil borings and on surface soil and analyzed for COCs listed in the work plan. Surface and subsurface soils will be field screened for organic vapors. If elevated organic vapors are detected or other evidence of a release of hazardous substances are observed, samples for COCs will be collected from the interval demonstrating the most significant indications of the release. Sampling locations, depths, and methods are discussed in the Work Plan and FSP.
Groundwater:	**Study Question:** Are COCs present in groundwater at concentrations above ARARs and TCBs? **Alternative actions:** - No further investigation - Additional investigation **Decision Statement:** Determine if COCs are present in groundwater and at what concentrations.	1. New and existing validated measurements of COC concentrations in samples collected from monitoring wells downgradient of the Remedial Investigation project area.	The Remedial Investigation project area boundary is included in the Work Plan (Barr, 2011).	If concentrations of COCs in groundwater from the nearby water supply well samples exceed ARARs and TCBs, further sampling and analysis may be conducted to determine magnitude and extent of the COC-impacted groundwater.	Potential sampling errors include sampling design errors and measurement errors. Design errors are countered with an adequate number of sampling points and measurement errors are minimized through systematic uniform management of each of the steps of measurement using SOPs and by following the QAPP.	Samples of groundwater will be collected from monitoring wells and analyzed for COCs listed in the work plan. Sampling locations, depths, and methods are discussed in the Work Plan and FSP.

Notes: Category 1 Sites = Sites of concern (SOCs) where releases of hazardous substances pr petroleum products have been identified above Tier I SRVs based upon previous environmental investigations.
Category 2 Sites = SOCs where Recognized Environmental Conditions (RECs) have been identified but environmental investigations have not been conducted and/or no releases have been identified.
Category 3 Sites = Portions of the RI project area where releases are less likely to have occurred based on a review of past land use.

Table 3
Field Instrument Precision, Accuracy and Preventative Maintenance
Stage 1
Quality Assurance Project Plan
UMore East Remedial investigation
Dakota County, Minnesota

Field Instrument	Precision Limits	Accuracy Limits	Preventive Maintenance
OVM-Thermo 580B	±35% RPD on duplicate soil samples	95-105%	If detected value is < or >10% of the true value submit for repair. Replace batteries as needed
MiniRAE 2000	±35% RPD on duplicate soil samples	95-105%	If detected value is < or >10% of the true value submit for repair. Replace batteries as needed
YSI Model 556 MPS	±25% RPD on duplicate water samples.	Calibrated daily	Change batteries when gauge is low. Replace sensors as needed. Replace/recharge batteries as needed
Ohaus Scout Pro Field Balance	50±3 g	Tare each time it is turned on.	Replace batteries as needed

Table 4
Frequency of Quality Assurance Samples
Quality Assurance Project Plan
UMore East Remedial Investigation
Dakota County, Minnesota

Parameter	Frequency	Comments
Field Blanks	1 collected every 20 samples	
Field Replicates	1 collected every 20 samples	Analyzed with field equipment only (i.e., replicate temp, pH or headspace readings to confirm instrument precision)
Field Duplicates	1 collected every 20 samples	Blind laboratory sample submittal
Trip Blanks- Soil (Methanol)	1 placed in every shipping container containing VOC soil samples.	Made up in the laboratory, only analyzed with associated VOCs soil samples. (a soil trip blank)
Trip Blanks-Water (HCl)	1 placed in every shipping container containing VOC water samples.	Made up in the laboratory, only analyzed with associated VOCs water samples.
Matrix Spike, Matrix Spike Duplicates	1 collected every 20 samples to provide the laboratory with necessary QA/QC volume.	Batch MS/MSD samples are required for this project and will performed on each matrix sampled. Since these batches should be representative of each matrix, project specific MS/MSD samples are not required for this project. Extra volume will be provided to the laboratory so that project samples may be used as MS/MSD samples.

Table 5
Sample Containers, Preservation and Holding Times
Stage 1
UMore East Remedial Investigation
Quality Assurance Project Plan
Dakota County, Minnesota

Parameter	Preservative/Container Type & Volume	EPA Recommended Hold Time
Soil		
Metals	Cool to ≤ 6°C, glass (4-oz and additional volume for moisture analysis)	180 days Mercury = 28 days
SVOCs	Cool to ≤ 6°C, glass (4-oz and additional volume for moisture analysis)	14 days to extraction; 40 days to analysis
VOCs	Cool to ≤ 6°C, 1:1 ratio soil:methanol (MeOH), glass (10 g to 10 ml solvent and additional volume for moisture analysis) 40 ml vial	14 days
Organochlorine Pesticides	Cool to ≤ 6°C, glass (one, 4oz container and additional volume for moisture analysis)	14 days to extraction; 40 days to analysis
Nitrocellulose	Cool to ≤ 6°C, glass (two, 4-oz containers)	28 days
PCBs	Cool to ≤ 6°C, glass (4-oz and additional volume for moisture analysis)	none
Explosives	Cool to ≤ 6°C, amber glass (4-oz and additional volume for moisture analysis)	14 days to extraction; 40 days to analysis
Flashpoint	Glass, 4 oz.	14 Days
Glycols (Ethylene and Propylene)	Cool to < 6°C, glass (one, 4oz container and additional volume for moisture analysis)	14 days to extraction; 40 days to analysis
Water		
Dissolved Metals	HNO_3 to pH<2; Plastic (500 ml)[1]	180 days Mercury = 28 days
Total Alkalinity	Cool to ≤ 6°C,unpreserved plastic (250 mL)	14 days
SVOCs	Cool to ≤ 6°C, amber glass (1-liter)	7 days to extraction; 40 days to analysis
VOCs	Cool to ≤ 6°C, HCl to pH <2, glass (set of 3-40 ml vials)	14 days
Nitrate + Nitrite as N	Cool to ≤ 6°C, plastic (100 mL)	48 Hours
Organochlorine Pesticides	Cool to ≤ 6°C, amber glass (1-liter)	7 days to extraction; 40 days to analysis
Chloride, Sulfate, Perchlorate	Cool to ≤ 6°C, plastic (100 mL)	28 days
Explosives	Cool to ≤ 6°C, amber glass (1-liter)	7 days to extraction; 40 days to analysis

1 All water samples collected for metals analysis from temporary wells or monitoring wells will be filtered in the field.

Figures

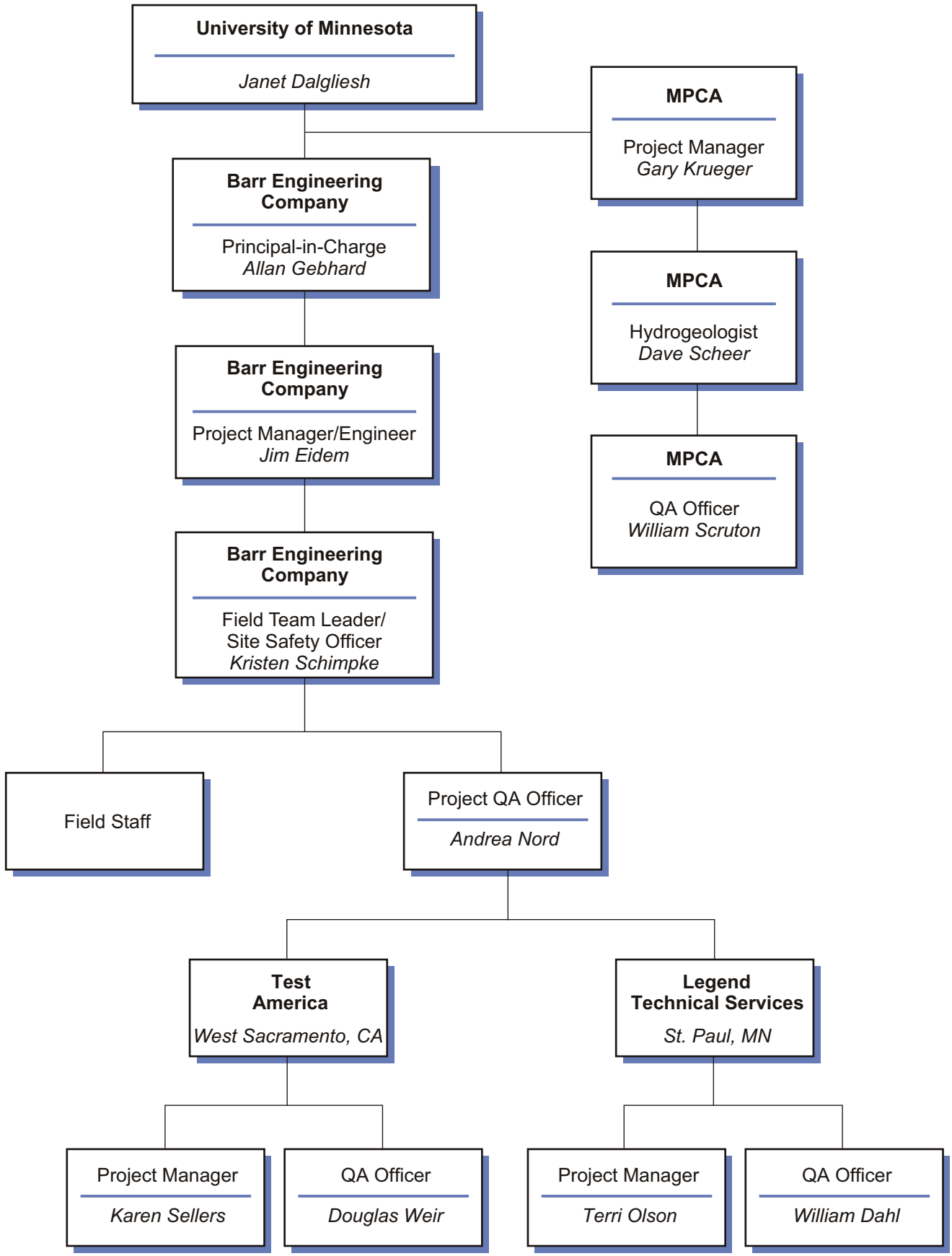

Figure 1

PROJECT ORGANIZATION
Quality Assurance Project Plan
UMore East Remedial Investigation
Dakota County, MN

UMP012461

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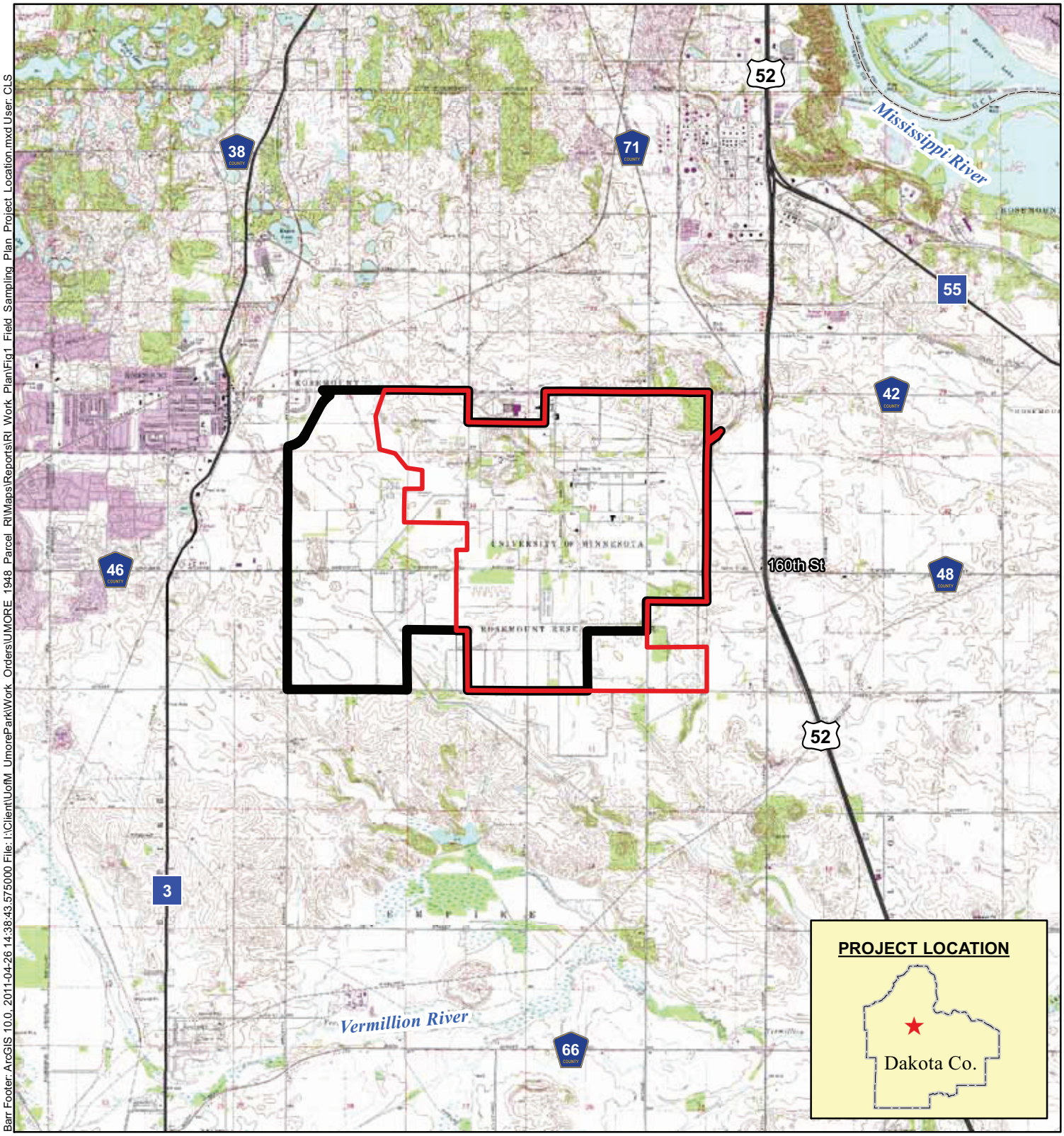

Barr Footer: ArcGIS 10.0, 2011-04-26 14:38:43.575000 File: I:\Client\UofM_UmorePark\Work_Orders\UMORE_1948_Parcel_RI\Maps\Reports\RI_Work_Plan\Fig1_Field_Sampling_Plan_Project_Location.mxd User: CLS

UMore East Project Area

UMore Park Boundary

Figure 2

PROJECT AREA LOCATION
Quality Assurance Project Plan
UMore East
Remedial Investigation
Dakota County, MN

Feet
6,000 0 6,000

Source: MnDOT, MN DNR, Dakota County, Barr, HKGi, University of Minnesota.
USGS topographic map background downloaded from the U.S.
Department of Agriculture, Natural Resources Conservation Service.

BARR

UMP012462

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BARR

Chain of Custody

4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Number of Containers/Preservative

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil	Matrix:	Type: Grab	Type: Comp.	Type: QC	Water: VOCs (HCl) #1	Water: SVOCs (unpreserved) #2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (unpreserved) #3	Water: Diesel Range Organics (HCl)	Water: Nutrients (H_2SO_4) #4	Water:	Water:	Water:	Soil: VOCs (tared MeOH) #1	Soil: GRO, BTEX (tared MeOH) #1	Soil: DRO (tared unpreserved)	Soil: Metals (unpreserved)	Soil: SVOCs (unpreserved) #2	Soil: % Solids (plastic vial, unpres.)	Soil:	Soil:	Total Number Of Containers
1.																													
2.																													
3.																													
4.																													
5.																													
6.																													
7.																													
8.																													
9.																													
10.																													

COC _____ of _____

Project Manager: _____

Project QC Contact: _____

Sampled by: _____

Laboratory: _____

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other: _____

Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Figure 3

CHAIN OF CUSTODY
Quality Assurance Project Plan
UMore East Remedial Investigation
Dakota County, MN

Appendices

Appendix A

Project Team Qualifications

Experience

Allan Gebhard has more than 40 years of experience as an engineering consultant at Barr, including serving as president from 1985 to 2000. In his career, he has directed a wide range of projects, with emphasis in the field of environmental engineering. Al has directed or otherwise participated in several hundred groundwater-contamination projects throughout the United States. He has managed remedial investigations that assessed human health and environmental impacts of wood preserving, oil refining, coke production, chemical manufacturing, industrial manufacturing, and mining facilities and has conducted feasibility studies that evaluated the effectiveness, cost, ability to implement, and secondary impacts of remedial actions. Al has also participated in remedial action projects at uncontrolled waste disposal sites, participated in property assessments and site cleanups pursuant to the buying and selling of real estate, and played a key role in designing environmental monitoring programs for a number of large industrial facilities.

Al has worked extensively with:

- Regulatory agency negotiations to define the extent of appropriate site remediation.
- CERCLA and RCRA regulations.
- Complex technical projects involving multi-jurisdictional regulatory agency involvement.
- Multi-disciplined teams of specialists evaluating technical solutions to complex waste management and site remediation problems.
- Steering committees and policymaking groups.
- Advising clients on technical issues at more than 100 cleanup sites in Illinois, Iowa, Florida, Georgia, Michigan, Minnesota, Montana, New York, Ohio, Oregon, South Dakota, Utah, Vermont, Washington, and Wisconsin.

His specific project experience has included:

- Directing a project for the Minneapolis-St. Paul metropolitan-area counties and the State of Minnesota in the late 1970s that surveyed hazardous waste generation in the Twin Cities metro area and recommended a “cradle to grave” management system for hazardous wastes. The work predated RCRA and became the basis for Minnesota’s early hazardous waste management legislation.
- Directing Barr’s work for the University of Minnesota on a contamination investigation needed to prepare a 1,722-acre area at UMore Park for mining.
- Directing the remedial investigation of an abandoned coal tar distillation and wood-treating facility in St. Louis Park, Minnesota, that had contaminated several aquifers, including the one supplying drinking water to that part of the

Twin Cities. The investigation was one of the first in the country to use groundwater modeling to track the movement of contaminants and to predict the future impact of the contamination on groundwater quality. This facility was one of the sites used by the U.S. EPA to demonstrate the need for CERCLA.

- Directing early remedial actions at Joslyn Manufacturing's former wood-treating facility, a Superfund site in Brooklyn Center, Minnesota. Remediation of the site included on-site biological treatment of contaminated soils, DNAPL recovery, and groundwater containment.

- Directing removal actions in compliance with multiple CERCLA orders issued for a closed wood-treating site. The work included a time-critical removal site evaluation and a time-critical removal action by order of the EPA. The removal site evaluation was completed within the stipulated timeframe. The removal action work plan properly characterized the waste stream (with EPA concurrence) as a RCRA Subtitle D waste. This characterization resulted in a $2 million savings. Concurrent with the time-critical removal action, a human health and ecological risk assessment work plan was developed by Barr and a partner consultant. This project also involves management and monitoring of remediation systems including product recovery, groundwater treatment with granular activated carbon, and post-closure activities for a RCRA-type containment vault.

- Directing the remedial investigation, feasibility study, and remedial action design for three industrial-waste-disposal sites in Oakdale, Minnesota. The Oakdale sites are included on the United States Environmental Protection Agency's National Priority List. Site remediation was overseen by a consent order with the state of Minnesota and U.S.EPA Region V. The entire cleanup project, from first notification to the final remedial construction, was completed in five years.

- Directing remediation and regulatory assistance for the Arrowhead Refinery Site near Duluth, Minnesota, a site on the U.S. EPA's National Priority List. Barr assisted in responding to U.S. EPA's administrative orders to implement the remedies in the record of decision (ROD) for the site. Source materials included high-lead-content sludge, filter cake, and oil-saturated peat. The EPA had selected on-site incineration as the appropriate remedial action in the ROD, but Barr conducted treatability studies and successfully assisted the potentially responsible party group in negotiating an amendment to the ROD that provided for implementation of a re-refining technology and subsequent disposal of process residues at an off-site Subtitle D landfill. Barr designed the more cost-effective remedial action and worked with a contractor to get cost estimates for completing the work. We also provided construction observation services during implementation of the project. Barr also managed development of the design and plans and specifications for the construction of a water-main extension connecting neighbors of the site to a municipal water

supply, and the installation and operation of a groundwater extraction system. In addition, we were responsible for construction observation of both these tasks, which were carried out by a subcontractor.

- Directing technical assistance to a responsible party in a large RCRA corrective action at a chemical plant in Florida. The corrective actions are being implemented by the present owner of the plant. Our client was a former owner of the facility. Barr became involved at this site when the draft RCRA facility-investigation/contamination-assessment report and baseline-risk-assessment report were being completed. We reviewed the available information and provided input about the identification, evaluation, and selection of the appropriate corrective measures and the implementation strategy. This input was incorporated into the feasibility study/corrective measures study (FS/CMS). Barr also assisted with complex PRP-cost-allocation issues on our client's behalf. Our technical approach to the cost-allocation negotiations led to what Barr's client believes was a fair and reasonable assignment of responsibility for future corrective measures costs.

- Directing the remedial investigation and remedial action design at the 180-acre National Pole and Treating Company site in Fridley, Minnesota, a site on the U.S. EPA's National Priority List. The remedial investigation involved collecting and analyzing soil samples; placing monitoring wells; and collecting and analyzing groundwater, surface water, and sediment samples. Barr also prepared plans and specifications for the selected remedial actions, and observed implementation of the remedial actions. Approximately 12,000 cubic yards of soil contaminated with polyaromatic hydrocarbons were excavated. Less-contaminated soil and contaminated groundwater were contained by a 35-foot-deep, 2,000-foot long slurry wall, a synthetic membrane cap, and interior and exterior groundwater collection systems.

- Directing the remedial investigation at a site once used to burn solvent waste from a specialty paper-production mill in northern New York. A preliminary assessment had been completed at the site by a consultant to the New York Department of Environmental Conservation. Barr placed a number of soil borings and test trenches across the site and analyzed representative soil samples for a wide range of volatile organics and metals. We concluded that soils above shallow bedrock were contaminated with lead. Barr completed a focused feasibility study that study concluded that stabilization and disposal at a RCRA Subtitle D facility was the most cost-effective remedy for the contaminated soils.

- Directing an environmental investigation at a site with former wastewater lagoons on a low island in the Willamette River across from a paper mill in Oregon. The Oregon Department of Environmental Quality (DEQ) had proposed listing the site on their confirmed release list (CRL) based on groundwater quality data from the mid-1980s that they found in their files. Barr located five existing monitoring wells that had been installed 15 to 25

years prior and were now concealed by dense vegetation. Barr accessed the wells and reviewed the resulting data and relevant results from prior investigations and evaluated them relative to current DEQ risk-based standards. These evaluations led to our conclusion that the site did not warrant listing on the CRL and that that no further investigation or actions were needed at the site. The DEQ agreed with our conclusions and stated that it would issue a no-further-action letter just 6 months after the site was proposed for listing on the CRL.

- Directing assistance to an ordnance manufacturer with the closure of several sites in Minneapolis that had been used for the disposal of munitions waste. The waste materials disposed at the sites consisted of explosive powder residues with small amounts of fuel oil and solvents. Contaminants of concern included petroleum products, volatile organics, and metals. Investigation activities included the placement of soil borings and monitoring wells and the collection of soil and groundwater samples. The work was done in conformance with RCRA since the facility was permitted under RCRA. Corrective measures studies were conducted at two of the sites and included excavation of contaminated soil, removal of contaminated groundwater, and clean closure of the sites.

- Directing an investigation and cleanup for an aerospace company in Bloomington, Minnesota. An electrical-transformer explosion resulted in PCB-containing oil contaminating surrounding soil and the inside and outside of a concrete electrical vault. Barr prepared a cleanup plan to comply with the U.S. EPA's self-implementing disposal option for PCB remediation waste and to meet the Minnesota Pollution Control Agency's requirements for contaminated soil remediation. Barr completed an investigation to assess the extent of contamination and oversaw cleanup activities, including excavation of contaminated soil and decontamination of the concrete vault according to U.S. EPA protocol.

- Directing a remedial investigation and feasibility study (RI/FS) at a former waste-oil-refinery site in Minneapolis, Minnesota. Barr prepared a quality assurance project plan (QAPP), conducted a remedial investigation to determine the nature and extent of soil and groundwater contamination, and performed a feasibility study for potential remedial options at the site.

- Directing groundwater remedial actions at a metal-plating facility in northeast Minneapolis with an apparent trichloroethylene release that had seeped into a fractured bedrock aquifer and was flowing along fractures toward the Mississippi River. Barr conducted a remedial investigation, including placement of several monitoring wells to map contaminant fate and transport in a complex fractured-bedrock setting. We then performed a feasibility study that considered a range of passive and active groundwater remedial actions. The selected remedial action consisted of a groundwater extraction system for plume migration control and an ultraviolet/peroxide treatment system with

subsequent discharge to the sanitary sewer. The treatment system was the first application of this technology to treat contaminated groundwater in Minnesota. Results indicate that greater than 99 percent removal of trichloroethylene is being achieved.

- Directing the investigation and remediation of a large shopping center in Wayzata, Minnesota. Contaminated soil and groundwater was discovered under the property from a former dry-cleaning tenant. Barr worked with the Minnesota Pollution Control Agency's Voluntary Investigation and Cleanup (VIC) program to conduct a soil and groundwater investigation, prepare a response action plan, and install a soil-vapor extraction system to remove residual contaminants. Barr also took the lead in preparing a reimbursement application from Minnesota Dry Cleaner Fund.

- Directing the investigation and remediation at a former research facility in Minneapolis, at which waste solvents were disposed. Barr investigated groundwater impacts, evaluated several remedial actions, conducted an air-stripping pilot test, and prepared plans and specifications for shallow and deep groundwater pump-out systems. Barr also provided long-term maintenance and monitoring to determine effectiveness of the systems.

- Directing the later stages of redevelopment assistance to the St. Paul Port Authority for the Maxson Steel Foundry property in St. Paul, Minnesota. The property had been used for over 100 years by heavy industry. Barr compiled the information from several previous investigations, conducted a Phase II investigation for areas requiring additional investigation, coordinated work with the MPCA's VIC program, prepared response-action cost estimates, helped prepared grant applications for the cleanup work, designed the response actions, provided construction observation, and assisted with a media-day and neighborhood meetings related to the redevelopment. The property is now called the Great Northern Business Center.

- Directing early investigations and remedial design and implementation efforts at an oil refinery in the Minneapolis-St. Paul metropolitan area. Barr's work has included conducting site-wide and release-specific investigations using innovative field analytical and geophysical techniques; collecting sediment and subsurface samples from the Mississippi River and the facility's wastewater treatment lagoons; designing and overseeing construction of groundwater and petroleum product extraction drains and wells; designing and installing bio-venting and bio-pile systems to handle contaminated soil; and designing ventilation systems and lining sewers to prevent entry and buildup of explosive vapors.
- Advising a potentially responsible parties group on the design and implementation of a $60 million RCRA corrective action in EPA Region IV. As part of this project, he led a Barr team that conducted a "value engineering" effort to improve the cost-effectiveness of the remedial action

design for one of the operable units. Barr's work identified approximately $2 million in potential cost savings.

- Advising a municipal client whose water-supply wells had been affected by chlorinated-solvent releases from a large military facility. The agreement with the military led to the use of the city's wells to control migration of the contaminants. Barr designed granular-activated-carbon treatment plants for the city's water supply. One of the plants was the second largest GAC plant for municipal supply in the United States.

Education

MS, Civil Engineering, University of Minnesota, 1967

BS, Civil Engineering, University of Minnesota, 1965

Registration

Civil Engineer: Minnesota and Michigan

Memberships

American Society of Civil Engineers

American Water Resources Association

Presentations/ Publications

"Cleanup Standards and Costs." Minnesota State Bar Association Superfund Forum. February 1994.

"Technologies for Groundwater State of the Art Remediation." Land Recycling: Minnesota Voluntary Investigation and Cleanup Program, Minnesota Environmental Initiative, Minnesota Pollution Control Agency, Minnesota Ground Water Association. St. Paul, MN, January, 1994.

"Implementation of Cleanup Remedies." CLG International Hazardous Waste Cleanup Seminar. October 22, 1993, and October 2, 1992.

"Using Contaminated Groundwater for Potable Supply." Presented at "Water 90" Conference, St. Paul, MN. April 1990 (with Mark Deady, P.E., and Greg Keil, P.E.).

"Control of Environmental Cleanup Costs." Minnesota Institute of Legal Education Environmental Law Institute. April 1990.

"Environmental Liability in Commercial Real Estate Lending." Minnesota Bankers Association, 1989 Real Estate Seminar. March 1989.

"How Clean Is Clean in Minnesota: A Private Perspective." Environmental Law Institute. October 1986.

"Groundwater Cleanup Standards." Environmental Law Institute. March 1985.

"Remedial Actions to Alleviate Groundwater Pollution from a Former Industrial Waste Disposal Site." Water Pollution Control Federation Annual Conference. October 1984.

"Hydrogeologic Assessments." Third Annual Midwest Conference on Environmental Laboratory Technology. December 1981.

"Hazardous Waste Management Facilities: Ownership, Financing, Siting and Liability." Water Pollution Control Federation Annual Conference. October 1979.

"Monitoring the Water Resource Impacts of Mining Activities." American Institute of Mining Engineers Symposium. February 1976.

"Recycling/Reclaiming Dumps—Old Beltline Dump Case Study," Minnesota Solid Waste Seminar, St. Paul, MN, February 1996.

"Technical Aspects of Site Cleanup." Land Recycling: The Redevelopment and Management of Previously Used Property, Minnesota Environmental Initiative, St. Cloud, Rochester, and Duluth, MN, September 1994.

Technical Reports Authored

While serving as project manager and principal of many Barr's projects, he authored or coauthored the following technical reports:

Phase I/Phase II Investigation Report, Slip 7 at Georgia-Pacific Corporation's Duluth, MN Facility. Prepared for the Georgia-Pacific Corporation, Duluth, MN.

Groundwater and Soils Investigation Report. Prepared for the Central Co-Operative Oil Association, Medford, MN.

Closure Investigation Report for the Noerenberg Burn Site. Prepared for Technical Ordnance, Incorporated, Carver County, MN.

Closure Investigation Report for the Polingo Burn Site. Prepared for Technical Ordnance, Incorporated, Wright County, MN.

Groundwater Investigation Report for Dan's Diner Eveleth, MN. Prepared for Hanft, Fride, O'Brien, Harries, Swelbar and Burns, P.A., Eveleth, MN.

Impact Investigation Report, Dairy Whey Disposal, Topp Farm, Dakota County, MN. Prepared for Marigold Foods, Inc., Farmington, MN.

Evaluation Report and Remedial Investigation Work Plan, Dairy Whey Disposal Site Topp Farm Dakota County, MN. Prepared for Hart, Bruner & O'Brien, Farmington, MN.

Groundwater Investigation. Prepared for the Central Co-Operative Oil Association, Medford, MN.

Soil and Groundwater Investigation, Final Report, Whitewood Wood Treating Facility. Prepared for Champion International, Whitewood, SD.

Evaluation Report for the Burnsville Landfill. Prepared for Edward Kraemer and Sons, Inc., Burnsville, MN.

Remedial Investigation/Feasibility Study. Prepared for Superior Plating, Inc., Minneapolis, MN.

Remedial Investigation/Alternatives Report, Brooklyn Center Wood Treating Site. Prepared for Joslyn Corporation, Brooklyn Center, MN.

Supplemental Alternatives Report, Brooklyn Center Wood Treating Site. Prepared for Joslyn Corporation, Brooklyn Center, MN.

Remedial Investigation Report for the USS Duluth Works Site. Prepared for USS, a Division of USX Corporation, Duluth, MN.

Initial Investigation, Final Report, Tonka Corporation Main Plant Site. Prepared for Tonka Corporation, Mound, MN.

Phase I and Phase II Summary Report: Ironwood Landfill Groundwater Investigation/Remedial Action Recommendations. Prepared for Advance Transformer Co., Spring Valley, MN.

Site Characterization Study and Remedial Action Plan, General Mills Solvent Disposal Site. Prepared for General Mills, Inc., Minneapolis, MN.

Feasibility Study of Remedial Actions, General Mills Solvent Disposal Site. Prepared for General Mills, Inc., Minneapolis, MN.

The Influence of Geologic Features on Land Disposal Facility Design. Prepared for the Minnesota Waste Management Board, Minneapolis, MN.

Conceptual Design & Preliminary Operating Plan: Accelerated Decomposition Landfill for Anoka County. Prepared for Anoka County Environmental Health Division, Anoka County, MN.

Soil and Groundwater Investigation, National Pole and Treating Company Site/Onan Property. Prepared for Boise Cascade Corporation, Fridley, MN.

Impact of Seepage from Freeway Sanitary Landfill on the Minnesota River. Prepared for Freeway Sanitary Landfill, Burnsville, MN.

Report on Hydrogeologic Investigation of the Freeway Landfill Site, Burnsville, MN. Prepared for Richard B. McGowan, Owner, and A.C. Godward, Consulting Engineer, Burnsville, MN.

215329v5

Experience

Jim Eidem has 13 years of experience as a hydrogeologist. He has served as project manager and lead hydrogeologist on a variety of projects, including soil and groundwater investigations, brownfields redevelopments, and solid waste facility expansions. Jim's clients have included real estate developers, solid waste facilities, chemical distribution companies, universities and industrial manufacturing companies. He has developed and negotiated investigative scopes with the U.S. Environmental Protection Agency and with state agencies in Minnesota, Illinois, and North Dakota.

Jim has extensive experience investigating hydrogeologic conditions of glacial deposits in the Midwestern U.S. His expertise includes designing and implementing investigation plans, developing hydrogeologic conceptual models and preparation of response action plans in support of site development. Jim's field experience includes implementing operations that include soil boring logging, monitoring well installation and decommissioning, aquifer testing, soil and groundwater sampling, and vertical groundwater-quality profiling.

Brownfields Redevelopment

Jim's project experience on complex brownfields redevelopment sites within the Minnesota Pollution Control Agency's Voluntary Investigation and Cleanup (VIC) Program includes assisting in the preparation of Phase I environmental site assessment reports, preparation of remedial investigation work plans and reports, development of response action plans, response action and construction oversight, contractor management, geotechnical/soil correction oversight, health and safety management, and preparation of implementation reports. Former uses at these sites included wood-treating facilities, an agricultural manufacturing facility, a lead-smelting facility, a street-sweepings disposal site, a firefighter-training/missile site, an automobile repair shop, a sand-blasting/foundry site, and a closed foundry landfill. His project work includes:

- Preparing a Phase I environmental assessment report, scoping and directing a Phase II investigation, and preparing a response action plan (RAP) for a phased redevelopment project along an urban creek corridor in the Twin Cities. Participated in meetings with the client (a watershed district), city, and attorneys.
- Preparing a remedial investigation (RI) work plan for a former agrichemical manufacturing and distributing site in Brooklyn Center, Minnesota. The site had soil and groundwater impacts from past operations and a fire at the facility. Ten "high risk" areas of concern were delineated for investigation with rotasonic drilling and composite sampling. The plan was submitted to two regulatory agencies for review and approval.
- Preparing a RAP detailing site-wide soil management actions for preparing a site in Minneapolis for the construction of a commercial facility. Environmental impacts at the site included street-sweepings, buried debris, and off-site

groundwater impacts. As part of RAP development, a phased-site investigation was conducted to fill "data gaps" in regards to environmental impact and geotechnical aspects. Impacted soils were managed on site by consolidating impacted soils beneath site pavements, constructing clean utility corridors, and vapor-barrier construction.

- Preparing a soil management plan (SMP) that detailed the methods by which soils impacted by heavy metals, organic compounds, and petroleum products would by excavated, managed, and used during response action implementation at a site in St. Louis Park, Minnesota. The SMP summarized the pre-development subsurface conditions with multiple cross-section and map-view figures, identified site soil-usage restrictions, and described soil stabilization and management methods to be employed by the earthwork contractor. The SMP was reviewed and approved by the MPCA prior to RAP implementation.

- Serving as staff hydrogeologist responsible for ensuring construction practices were conducted in accordance with the RAP at former wood-treating sites in Brooklyn Center and Fridley, Minnesota, both de-listed state Superfund sites. Also responsible for health and safety oversight, RAP implementation documentation, and weekly meetings with site contractors and project stakeholders. In addition to soil correction and consolidation, groundwater response actions involved decommissioning and relocating components of a groundwater pump-and-treat system and a DNAPL recovery system.

- Preparing the RAP implementation report for a site in St. Louis Park, Minnesota. Documented restricted waste abatement, building demolition, heavy metal stabilization, soil excavation/consolidation/capping, off-site disposal of impacted soil, export of non-impacted soil, investigation of former site utilities, well decommissioning, off-site disposal of liquid wastes, vapor-barrier construction, utility-corridor construction, utility installation, groundwater dewatering, and post-development subsurface conditions. The implementation report was approved by the regulatory agency.

- Serving as senior hydrogeologist for site visits at a windpower generation facility in Minnesota and one in Iowa as part of an effort to update existing Phase I environmental site assessments. Prepared historical site use, historic map review, city record review, and environmental database report review sections for multiple Phase I reports.

Groundwater Investigation

Jim has extensive experience evaluating the hydrostratigraphy of, and groundwater flow through, glacial units in the Upper Midwest. His graduate school research entailed developing a conceptual model to describe the glacial stratigraphy and groundwater flow throughout a 5,600-hectacre watershed in central Iowa. His project work includes:

- Serving as the field team manager for ten site characterization/remedial actions at the University of Minnesota's UMore Park since 2008 and understands the challenges and importance of scoping, budgeting, scheduling, and following through on projects at UMore Park. Participated in meetings and coordinated scopes of work with MPCA, Dakota County, City of Rosemount, Empire Township, Department of Natural Resources, and UMore Park staff. Worked with University staff to develop a streamlined approach to developing investigation plans for submittal to regulatory stakeholders. Responsibilities at UMore Park include:
  - Preparing a work plan for and directed the field efforts for the site-wide groundwater assessment to support the EIS for the UMore Mining Area (UMA). The scope of work included installation of monitoring wells and evaluating existing wells located throughout UMore Park and the former GOW for use in the assessment.
  - Preparing detailed quality assurance project plans (QAPPs) for use in environmental investigations throughout the UMA and greater UMore Park area. The QAPPs have been approved by the MPCA for use during the environmental investigations and remedial actions.
  - Planning, coordinating, and managing field investigations for the environmental investigation of eight sites of concern within the UMA. Field operations included drilling, test trenching, surface soil and groundwater sampling and occurred in active research areas, former GOW operational areas, and AES operation support areas without disruption to University and UMore Park staff.
  - Scoping, planning, and directing environmental investigations of five University-identified sites to evaluate the suitability of proposed future land use or ownership changes.
- Serving as project manager and hydrogeologist for hydrogeologic permitting for solid-waste-facility expansions in Elk River and Glencoe, Minnesota. Developed hydrogeologic evaluation work plans and project budgets, conducted field investigations, and prepared evaluation reports. The proposed facilities included a construction and demolition (C&D) debris facility, a municipal-solid-waste (MSW) facility, and the first inward-gradient design for a landfill in the state. Detailed geologic logging and hydrogeologic data were used to develop site conceptual models that focused on identifying the uppermost permeable groundwater-flow pathways.
- Scoping and conducting a series of hydrogeologic investigations to demonstrate groundwater discharge to a large wetland/floating bog in Elk River, Minnesota. Results of the investigation demonstrated the occurrence of groundwater discharge to the wetland complex and served as the basis for a proposal to implement surface-water compliance standards rather than conventional groundwater standards.

- Serving as project hydrogeologist for a Resource Conservation and Recovery Act (RCRA) facility investigation and corrective-measures-study pre-design investigation at a site in Omaha, Nebraska. Conducted an environmental investigation focused on determining the geologic/hydrogeologic conditions at the site and assessment of chlorinated solvent and pesticide impacts to soil and groundwater. Installation of monitoring wells up to three miles off-site required detailed logging of a sequence of loess and till deposits. Conducted data analysis and prepared numerous work plans, data transmittals, and reports documenting field activities and investigation results.

- Implementing a field investigation to determine the vertical and horizontal extent of two co-mingled chlorinated solvent plumes migrating through outwash deposits in Ringwood, Illinois. Detailed geologic logging was the key to unraveling the complex glacial stratigraphy. Continuous monitoring of groundwater stabilization parameters, along with the precise hydraulic head measurements, allowed for the collection of representative groundwater samples from the low-yield glacial deposits.

- Serving as project hydrogeologist for aquifer testing in Omaha, Nebraska. Coordinated and implemented a step-drawdown test and a five-day aquifer test to estimate groundwater flow parameters, which were used to refine a numerical groundwater-flow model and to design a long-term groundwater-extraction and treatment system. Coordination for the aquifer test included gaining approval for groundwater discharge to the combined storm/sanitary sewer, submersible pump sizing, developing internal data collection and QA/QC plans, and constructing purge-water transmission, metering, and pretreatment systems for the test.

- Serving as field team leader responsible for directing two field geologists and rotasonic drilling and sewer cleaning subcontractors. The facility was an active chemical manufacturing plant situated along a river. The project included detailed description of glacial deposits, installation of numerous monitoring wells, well development and sampling, and geophysical investigation.

Solid Waste Facilities

Jim's experience with solid-waste facilities includes nine years of groundwater investigations and compliance monitoring at a solid-waste facility in Minnesota. He has completed groundwater investigations involving geochemical, isotopic, hydraulic, and hydrogeologic components. Jim has designed long-term groundwater, surface-water, and landfill-gas monitoring systems and also has experience in developing plans for the operation and monitoring of a leachate-recirculation system. His project work includes:

- Serving as project manager and hydrogeologist for groundwater-monitoring-network optimization, design, and installation at Elk River Landfill in Elk River, Minnesota. A review of site geologic data indicated that the existing monitoring system targeted separate hydrostratigraphic units, which led to an erroneous interpretation of groundwater-flow direction at the facility. The hydrogeologic

conceptual model was revised and a new groundwater-monitoring network was installed. Monitoring results confirmed the appropriateness of the new monitoring network.

- Developing a monitoring protocol and performing down-hole and direct measurements of the moisture content of landfilled waste at two MSW facilities permitted to conduct leachate recirculation on a pilot-project basis. The monitoring protocol summarized the leachate recirculation procedures, provided specific data-quality objectives, and detailed specific monitoring and documentation methods.

- Serving as project manager and hydrogeologist for environmental monitoring program administration and reporting at Elk River Landfill in Elk River, Minnesota. Responsibilities included reviewing operating permits, developing sampling and analysis plans, coordinating laboratory and sampling crew personnel, and preparing quarterly and annual environmental monitoring reports. The quarterly monitoring reports were developed for the purpose of data transmission with a comparison of results to regulatory standards. The annual reports provided in-depth trend analyses and geochemical analysis using stiff and piper diagram interpretation.

- Serving as project manager and hydrogeologist for a hydrogeologic evaluation and well installation at Jahner Landfill in Wishek, North Dakota. The site geology consisted of fine-grained glacial deposits overlying weakly cemented bedrock units of inter-bedded shales, sandstones, and lignite deposits. Detailed geologic logging was performed in conjunction with careful observation of hydrogeologic conditions to determine effective monitoring intervals within the saturated bedrock units. The project resulted in a better understanding of the site stratigraphy and the installation of two effective network-monitoring wells.

- Serving as project hydrogeologist for a closure pre-design evaluation at a partially lined paper-sludge facility in Eau Claire, Wisconsin. Conducted a reconnaissance site visit and performed document review, a borrow study investigation, and waste-characterization sampling. Document review and site reconnaissance focused on developing an understanding of the construction and operation of the facility to develop an engineer's cost estimate for closure and post-closure care. Waste samples were collected with a lightweight/low-ground-pressure ATV drill rig using a piston-driven sampling apparatus. The borrow study was designed and implemented to determine the quality and volume of on-site fill suitable for landfill closure.

- Serving as project manager and lead hydrogeologist responsible for the design and installation of a bedrock water supply well at Elk River Landfill in Elk River, Minnesota. The well was designed and constructed with a target production rate of 50 gallons per minute and to provide compliance monitoring and water supply. Mud and air rotary drilling methods were used to construct the well to a total depth of 365 feet underground.

Site Remediation

- Serving as project hydrogeologist for the cost estimate of a groundwater corrective action at a solid waste landfill in Chicago, Illinois. Responsibilities included reviewing site hydrogeologic data, defining a rudimentary groundwater treatment system, and estimating costs for implementing the system. The client used the cost estimate for quantifying long-term liabilities for setting limits for closure and post closure costs.

- Serving as project hydrogeologist for the environmental response to three uncontrolled release sites in Geneva, Nebraska; Braham, Minnesota; and Brooklyn Center, Minnesota. Responsibilities included determining the extent of the released materials, collecting characterization samples, overseeing soil excavation and other remedial activities, and providing documentation. One project entailed responding to a water main break at a brownfields site, resulting in the transport of soils impacted with wood-treating chemicals through a sewer pipe and into a stormwater pond. Response actions included establishing site control measures, communicating with the developer and regulatory personnel, and removing soils from the sewer pipe and stormwater pond.

- Overseeing the removal of approximately 25 underground storage tank systems at U.S. Postal Service and filling station sites throughout Minnesota. Responsibilities included documenting the tank removal procedures, screening excavated soils for impacts, and collecting confirmation soil and groundwater samples. Reporting included the completion of a worksheet-style documentation report for submittal to the state.

Education

MS, Geology, Iowa State University, 1996

BA, Geology, University of Minnesota-Morris, 1993

Registration

Registered Professional Geologist: MN

Memberships

Minnesota Groundwater Association

Publications

Eidem, J.M., W.W. Simpkins, and M.R. Burkart. "Geology, Groundwater Flow, and Water Quality in the Walnut Creek Watershed." *Journal of Environmental Quality.* V. 28, p. 60-88. 1999.

Simpkins, W.W., M.F. Helmke, M.R. Burkart, and J.M. Eidem. "Effect of Agriculture on Groundwater Quality and Recharge in a Till-Dominated Watershed in Central Iowa." *Geological Society of America, Abstracts with Programs.* v. 30, no.7, p. A-173. 1998.

Simpkins, W.W., J.M. Eidem, B.L. Johnson, H.H. Seo, M.F. Helmke, T.B. Parkin, and M.R. Burkart. "Applications of Stratigraphic and Paleo-landscape Analysis to Hydrogeologic Studies of a Regional Quaternary-age Confining Unit in Central Iowa." *Geological Society of Canada Annual Meeting Abstracts* A-137. 1997.

Eidem, J.M. and W.W. Simpkins. "Quaternary Stratigraphy of the Walnut Creek Watershed." *Hydrogeology and Water Quality of the Walnut Creek Watershed.* Geological Survey Bureau, Guidebook Series no. 20, p. 19-29. 1996.

Eidem, J.M., W.W. Simpkins, and M.R. Burkart. "Groundwater Flow and Water Quality in the Walnut Creek Watershed." *Hydrogeology and Water Quality of the Walnut Creek Watershed.* Geological Survey Bureau, Guidebook Series no. 20, p. 37-44. 1996.

Seo, H.H., J.M. Eidem, and W.W. Simpkins. "Hydraulic Properties of Quaternary Units in the Walnut Creek Watershed." *Hydrogeology and Water Quality of the Walnut Creek Watershed.* Geological Survey Bureau, Guidebook Series no. 20, p. 59-67. 1996.

Simpkins, W.W., J.M. Eidem, H.H. Seo, B.L. Johnson, M.F. Helmke, M.R. Weis, T.B. Parkin, M.R. Burkart, and T.B. Moorman. "Confining Units Are Not Created Equal: Quaternary History and Its Effect on Physical and Biogeochemical Processes." *Geological Society of America, Abstracts with Programs.* v. 28, no. 7, p. A-72. 1996.

Eidem, J.M., W.W. Simpkins, and M.R. Burkart. "Geological Controls on Groundwater Flow and Water Quality in the Walnut Creek Watershed of Central Iowa." *Geological Society of America, Abstracts with Programs.* v. 28 no. 6, p. 37. 1996.

Eidem, J.M., W.W. Simpkins, and M.R. Burkart. "Quaternary Geology and Groundwater Flow in the Walnut Creek Watershed, Central Iowa." *Abstracts of the Iowa Academy of Science.* 1996.

Eidem, J., W.W. Simpkins, and M.R. Burkart. "Hydrogeology of the Walnut Creek Watershed." *Abstracts of the Iowa Academy of Science.* 1995.

Simpkins, W.W., B.L. Johnson, J.M. Eidem, H. Van Iten, M.R. Weis, and T.B. Parkin. "Hydrogeology of Central and Northeast Iowa Till." *Iowa MSEA Water Quality Colloquium Proceedings and Research Updates*, M.A. Smith, ed. USDA-Agricultural Research Service and ISU Extension. p. 17-22. 1994.

Simpkins, W.W., B.L. Johnson, J.M. Eidem, and M.R. Weis. "Use of Non-Traditional Piezometer Installation Techniques for Hydrogeological Studies in the Walnut Creek Watershed." *Water, Water Everywhere… Guidebook for the 57th Annual Tri-State Geological Field Conference and Geological Society of Iowa Field Trip Guidebook 58,* Ames, IA. p. 83-89. 1993.

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Experience

Andrea Nord has 15 years of experience providing technical support for analytical measurements and services. She has a strong familiarity with U.S. Environmental Protection Agency (USEPA) methodology and safety and hazardous waste compliance. Andrea's responsibilities at Barr include reviewing and updating all of Barr's standard operating procedures for data management, conducting technical laboratory and field-sampling audits, and selecting and pre-qualifying laboratories. Andrea's project work at Barr has included:

- Establishing a quality-management system for a clients' internal National Pollutant Discharge Elimination System laboratory.
- Performing on-site and off-site laboratory regulatory compliance audits
- Preparing analytical cost estimates, quality assurance project plans, sampling and analysis plans, and quality assurance/quality control reports.
- Examining data from routing routine data-quality-control measures.
- Following method-specific quality assurance criteria.
- Following the USEPA's Contract Laboratory Program National Functional Guidelines for data validation.
- Coordinating sample events for a variety of environmental remediation and investigation sites, including former manufactured-gas-plant sites, Superfund sites, Resource Conservation and Recovery Act (RCRA) facilities, mining sites, voluntary investigation and cleanup (VIC) program sites.
- Serving as project manager for a federal drinking-water monitoring program for a major airline.

Prior to joining Barr, Andrea's work experience includes:

- Serving as a hazardous waste coordinator, project manager, and database development liaison at an environmental laboratory in Minneapolis. Her responsibilities included:
  - Serving as the primary technical and administrative liaison between clients and the laboratory through daily coordination of activities, including preparing quotes and reporting data and analytical results.
  - Managing multi-million-dollar federal-contract clients with very stringent laboratory-quality requirements.
  - Maintaining clients' data records to ensure data defensibility.
  - Developing and launching an internal waste program by evaluating internal waste streams and negotiating contracts with transportation and disposal companies.

  - Directing the safety officer by using regulatory-compliance knowledge to ensure the company operated in a safe and efficient manner. Trained department personnel in proper handling techniques. Evaluated accidents and identified training or equipment needed to prevent further incidents.
  - Defining standard operating procedures for waste and a site-contingency and emergency-procedures plan, which resulted in consistent and positive local and federal audit results over a five-year period.
  - Developing and implementing a waste-personnel training program and conducting internal waste inspections.
  - Maintaining regulatory compliance records, including waste manifests, standard operating procedures, and training documentation.
  - Creating and launching a waste-tracking database to track all lab waste from creation to final disposal.
  - Transitioning laboratory from a small-quantity to a large-quantity hazardous-waste generator while maintaining regulatory compliance.
  - Applying for annual waste license and calculating annual state-waste tax.
  - Serving as laboratory liaison to evaluate company's nationwide database requirements and work with the information technology department to institute changes.

- As a sample management supervisor, establishing standard operating procedures for the documentation, receipt, and processing of all incoming environmental samples to ensure all USEPA and client data-quality objectives were met. Trained and supervised new and existing personnel to adhere to strict sample-processing guidelines. Coordinated incoming sample loads for efficient processing to ensure all USEPA holding times were met and rush-status samples were processed quickly.

- Receiving samples, evaluating sample information against USEPA-method criteria, and documenting discrepancies. Also scheduled samples for chemical analysis via the laboratory's data-management system per client chain-of-custody requirements. Maintained sample chain of custody and sample storage areas for data integrity. Prepared containers and equipment for sample collection.

- Analyzing transformer oil for a small oil-testing firm that specialized in analyzing and reconditioning transformer oil while the transformers were still in operation. Prepared and analyzed transformer oil for various physical properties using ASTM methods to determine level of insulating value and potential for breakdown. Received samples, assigned analyses, and coordinated sample load to ensure projects were completed on time. Wrote and proofread analytical reports. Maintained sampling equipment and supplies for all field technicians.

Education

In addition to coursework at Normandale Community College, 1993-1995, Andrea has completed specialized training in environmental law; Clean Air Act; Clean Water Act; Comprehensive Environmental Response, Compensation, and Recovery Act; RCRA; Emergency Planning; Community Right-to-know Act; Toxic Substances Control Act; hazardous materials transportation; tank management; technical writing; gas chromatography; liquid chromatography/ mass spectrometry; inductively coupled plasma; inductively coupled plasma/mass spectrometry; applying water chemistry and microbiology for environmental applications; contaminant chemistry and groundwater transport; monitored natural attenuation; and radiation safety and operation of Niton x-ray fluorescence analyzers.

Certification

40-hour OSHA HAZWOPER Emergency-Response-Level training (2000) and eight-hour annual updates (2001-2010)

National Safety Council Fundamentals of Occupational Safety and Health certificate (2006)

Federal Emergency Management Agency (FEMA) IS-700 National Incident Management System (NIMS) Awareness training (2006)

USEPA Region V "How to Survive an SPCC Spill Prevention, Control, and Countermeasure Inspection" training (2006)

Minnesota Safety Council Title 49, Code of Federal Standards and International Air Transportation Association 16-hour Hazardous Materials and Dangerous Goods Transportation course (2001-2004)

MPCA "10 Steps to Hazardous Waste Compliance" training (2004)

Hennepin County Hazardous Waste Generator training program (1999)

Memberships

Minnesota Chromatography Form (2006-2010)

The NELAC Institute (2008-2010)

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Experience

Karen Stoller has 30 years of experience in the areas of industrial hygiene, health and safety, and hazardous materials management. Her work at Barr includes managing the corporate health and safety program for more than 500 staff members, which entails developing and implementing health and safety policies, preparing site-specific health and safety plans, providing health and safety training for employees, managing medical surveillance and controlled-substance-testing programs, performing ergonomic assessments, and conducting exposure air monitoring. Examples of her project experience include:

- **Project Health and Safety Plan Development:** Prepared numerous safety plans for investigation and remediation of coal gasification sites, industrial and municipal landfills, petroleum release sites, scrap yards, petroleum refineries, and lead smelters.
- **Site Safety Officer:** Monitored health and safety on hazardous waste sites. Responsibilities included conducting daily safety meetings, performing air monitoring, observing personal-protective-equipment use, implementing site control procedures and assuring that site operations were consistent with the project health and safety plan.
- **Opus:** Reviewed and updated corporate health and safety training manual. Presented OSHA overview at corporate conference.
- **Minnesota County Environmental Health:** Managed project and conducted county-wide environmental health and safety audits. Work included audit protocol development to determine the level of regulatory compliance and implementation throughout all county divisions.
- **Computing Devices International:** Managed an environmental, health, and safety audit for three Minneapolis facilities. Conducted regulatory evaluation to determine compliance with Minnesota OSHA and Minnesota hazardous-waste-management rules.
- **U.S. Postal Service, Bulk-Mail Facility:** Managed project and conducted safety inspection to identify pinchpoints and rotating equipment that would require guarding or other protective systems in accordance with OSHA regulations. Worked with architects on design of guarding systems.
- **Jostens:** Managed a three-day environmental health and safety corporate conference and presented on several technical topics. Also presented health and safety regulatory information at a subsequent Jostens corporate conference.
- **Electrolux:** Managed several air monitoring projects to determine the extent of employee exposure to hazardous substances.
- **Midwest Center for Occupational Heath and Safety:** Participated in semi-annual 40-hour hazardous waste operations training program as a member of faculty and planning committee. Topics included air monitoring equipment and site safety plans.

- **VeraSun Energy:** Provided exposure monitoring oversight for hexavalent chrome air monitoring.
- **Confidential Former Cement Plant:** Work at this site is being conducted under the oversight of the U.S. EPA and the Michigan Department of Environmental Quality for compliance with an issued CERCLA § 106(a) order. The removal action work included expedited removal actions, extensive site characterization, and interim response activities. Karen provided exposure monitoring oversight for collection of total and respirable silica dust during remediation activities.

Before joining Barr, Karen held positions in environmental consulting, private industry, and county and federal agencies. She also managed corporate industrial hygiene programs and conducted health and safety audits and OSHA compliance inspections involving collection of exposure air monitoring samples that included organic vapors, dust, silica, and asbestos, while with the OSHA federal enforcement program in Wisconsin, and managed in-plant industrial hygiene programs at Control Data Corporation's printed-circuits operation.

Education

M.S., Environmental Health, University of Minnesota, 1990
(emphasis: Industrial Hygiene and Hazardous Waste Management)

B.A., Biology, State University of New York at Oswego, 1978

Certifications

Certified Industrial Hygienist

Certified Safety Professional

Certified Hazardous Materials Manager

Memberships

American Industrial Hygiene Association

American Society of Safety Engineers

American Society for Training and Development

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RESUME

William R. Dahl

EDUCATION:

Bachelor of Science - Chemistry
University of Pittsburgh, Pittsburgh, PA

Bachelor of Arts - History and Philosophy of Science
University of Pittsburgh, Pittsburgh, PA

SEMINARS/SHORT COURSES:

- Measurement of Uncertainty (in Laboratory Testing) - Advanced Systems
- Detecting Improper, Unethical, and Illegal Laboratory Practices - USEPA
- Crosby Quality Enhancement Process/TQM - PCA, Inc.
- Advanced Training in Atomic and Molecular Spectroscopy - Varian Associates
- A Human Relations Approach to Professional Selling - Dale Carnegie
- Problem Solving Salesperson - Knowledge Resources, Inc.
- Competitive Selling Strategy - Knowledge Resources, Inc.
- Counselor Salesperson - Wilson Learning
- Social Styles - Wilson Learning

PROFESSIONAL WORK EXPERIENCE:

LEGEND TECHNICAL SERVICES, INC., St Paul, Minnesota. November, 2009 to Present.

QA/QC Coordinator - Responsibilities include implementing ISO standards; maintaining documents and records of quality control data; coordinating laboratory certifications and accreditations; implementing and maintaining Proficiency Testing program; implementing and maintaining QC limits; updating the QA Manual; coordinating and conducting internal audits; coordinating outside auditing processes and providing required follow-up information; assuring documentation and resolution of corrective actions; overseeing sample log-in.

Inorganic Technical Director - Responsibilities include mentoring inorganic staff, new equipment review, maintaining state-of-the art technology, and development of new methods.

BRAUN INTERTEC CORPORATION, Bloomington, MN, 2002 to October, 2009.

Senior Scientist/Project Manager (2006 to October, 2009) - Responsibilities included focusing on specific client needs and preferences, worked on all facets of operations to meet those needs and preferences including direct client project management, operations management, and quality control; direct management of work flow, support of project managers, partner with clients to ensure that Quality Assurance Project Plans (QAPP) were acceptable to the MPCA, USEPA, and other regulatory agencies. Met with clients and regulators to understand current and future needs, conducted external audits of sub-contractors and vendors; provided chemistry consulting services, and was responsible for coordination of continued LIMS development and maintenance.

Quality Director (2002 to 2005) - Responsibilities included oversaw the creation of a new quality manual and new processes to ensure greater compliance to standards, more expedited resolution of non-conformances, and compliance to ISH 17025 guidelines; designed and conducted training sessions to educate staff on quality concepts; conducted internal audits of staff, procedures, documents, and data; revised format of standard operating procedures (SOPs) to comply with Federal guidelines, state requirements, and AIHA requirements; managed revision of SOP content to comply with methods and regulatory standards as well as provide greater usability for analysts. Maintained Minnesota and Wisconsin certifications including American Industrial Hygiene (AIHA) accreditations, project sponsor for the analytical laboratory information management system (LIMS), duties included specification, purchase, design, configuration, implementation, training, support, and maintenance; and managed installation and method development for new several instruments.

LEGEND TECHNICAL SERVICES, INC., St. Paul, MN, 1999 to 2002.

Technical Director - Responsibilities included managed inorganic and mobile chemistry groups; oversaw revision of SOPs for the Inorganic Department; oversaw purchase, installation and method development for several new instruments.

VARIAN ASSOCIATES, INC., Minneapolis, MN, 1994 to 1999.

Sales Representative - Responsibilities included providing solutions to customer problems through sales of advanced scientific instrument and software for a variety of analytical applications; managed a sales territory for MN, ND, SD, WI, and Inside Adjacent - this included development and execution of an annual business plan to meet company directed sales goals; participated in ISO 9002 accreditation of field sales and service force; developed analytical methods and trained customers on new equipment.

PACE, INC., Pittsburgh, PA, 1992 to 1994.

Team Leader - Responsibilities included supervised a group of scientists in analysis of samples by a variety of techniques; worked directly with the Quality Assurance representative of the USEPA to create and implement CLP certification and a CLP contract; oversaw production of Level 4 data deliverables for the USEPA and Air Force; developed analytical methods and procedures to increase quality and productivity; selected, purchased, and installed new instruments; and served on Total Quality Management team to improve overall laboratory processes.

LANCASTER LABORATORIES, INC., Lancaster, PA, 1990 to 1992.

Chemist - Responsibilities included validated data for final release to client; ensured compliance with appropriate programs including Good Laboratory Practices (GLP) and EPA regulations; coordinated efforts of the atomic absorption spectroscopy group for three shifts including weekends as needed; trained new and existing personnel and evaluated their performance; served on Quality Enhancement teams to make process improvements; developed technical methods to expand services; and analyzed samples by atomic spectroscopy techniques.

UNIVERSITY OF PITTSBURGH, Pittsburgh, PA, 1986 - 1990.

Laboratory Technician - Responsibilities included provided support role in a variety of research labs.

RESUME

Terri A. Olson

EDUCATION

Bachelor of Science, Microbiology with a minor in Chemistry 1980-1984
University of Wisconsin, LaCrosse.

SEMINARS/SHORT COURSES

Crystal Reports Part 1, 2007
LIMS Management Training, 2004
Internal Auditor Training for ISO 9001:2000, 2003
Microsoft Excel - Tips, Techniques & Shortcuts, Rockhurst, 2002
How to Make ISO Certification Happen, MN Quality Conference, 2002
Corrective Action Preventive Action, MN Quality Conference, 2001
Preparing for an FDA Inspection, MN Quality Conference, 2001

PROFESSIONAL WORK EXPERIENCE:

LEGEND TECHNICAL SERVICES, INC., St. Paul, Minnesota. May 2001 to Present.

Client Manager II (2001 to Present) – Responsibilities include monitoring client projects and prepare proposals; generating laboratory reports; determining pricing for clients and generate project invoicing; addressing client requirements, including turnaround times, methods, and reporting formats; communicating with clients on a routine basis as to requirements and/or problems; responding to client problems and concerns in a timely manner; preparing sub-contracts and purchase orders for work required to be sub-contracted.

LIMS Manager (2006 to Present) – Responsibilities include training Legend personnel on using LIMS for sample receiving, project management, invoicing, and reporting purposes; preparing LIMS crystal reports, bids, and worksheets; interfacing with the vendor for daily use.

QA/QC Coordinator (2001 to 2006) - Responsibilities included implementing ISO standards; maintaining documents and records of quality control data; coordinating laboratory certifications and accreditations; implementing and maintaining Proficiency Testing program; implementing and maintaining QC limits; updating the QA Manual; coordinating and conducting internal audits; coordinating outside auditing processes and providing required follow-up information; assuring documentation and resolution of corrective actions.

DAVY LABORATORIES, LaCrosse, Wisconsin. May 1984 to May 2001

Lab Administrator - Responsibilities included implementing and supervising all internal processes necessary from sample receipt to report distribution; developing standard forms for lab reports and invoices; responding to client requests for quotes, results and interpretation; assisting in coordinating and facilitating work of support staff.

Quality Control Coordinator - Responsibilities included reviewing results prior to final submittal to the Laboratory Director; tracking analyst qualifications, MDL studies and corrective action statements; maintaining Proficiency Testing and Blind Standard programs; performing internal audits on data; updating control and warning limits; creating QC spreadsheets for clients; serving as liaison for state and federal certification audits.

Microbiology Supervisor - Responsibilities included training and supervising student interns; analyzing wastewater, drinking water, reagent water, and sludge with various methodologies including Membrane Filtration, Most Probable Number, MMO-MUG, Heterotrophic Plate Count, Water Suitability and immunoassay techniques; analyzing proficiency samples for compliance; performing audits; maintaining documents and records of quality control data.

Inorganic Wet Chemistry Intern - Responsibilities included analyzing wastewater, drinking water and sludge; using methodologies for alkalinity, ammonia, BOD, chloride, chlorophyll, hardness, TKN, nitrite, sulfate, suspended solids, TDS, percent solids, volatile solids, and turbidity.

AFFILIATIONS

MDH Environmental Laboratory Certification Program Advisory Committee (2001-2006)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Personnel Resume

Douglas Weir

Qualifications Summary

Dr. Weir has over 19 years of experience in the environmental laboratory industry that includes experience in high and low resolution GC/MS, GC/ECD, HPLC, UV/visible spectroscopy and magnetic resonance. He has authored method standard operating procedures, Quality Assurance Plans, Project/Cost proposals and thirty scientific papers. He is conversant with a wide variety of US EPA methodologies including SW846 organic and inorganic methods; series 500, 600, and 1600 methods for drinking water and wastewater; and has experience with complex matrices such as air and sediments.

Professional Experience – TestAmerica West Sacramento

Quality Assurance Manager **2009 - present**

As a senior member of management Dr. Weir directs and monitors quality assurance activities at the West Sacramento facility. He is responsible for reports to management, client concerns, project plan review, lab performance review, and review of procedures that will ensure the production of data of a defined quality. He is responsible for performing the systems and method audits of the laboratory. He is also responsible for hosting client and regulatory audits, for writing responses, and assuring all corrective actions are implemented in a timely fashion. Dr. Weir is responsible for maintaining many state certifications, including NELAC, and assuring the laboratory complies with all requirements. He is responsible for conducting routine training for the laboratory staff, including Ethics, Manual Integration, Proper Documentation, and Quality Systems.

Department Manager, Advanced Technology Instrument & Data Analysis Group **2002 – 2009**

Dr. Weir's responsibilities include oversight of personnel responsible for operation of our High Resolution and Liquid Chromatography Mass Spectroscopic instruments, and the write-up, review and release of data obtained for all the mass spectroscopic methods. The group responsibilities include interpretation and review.

Consultant Scientist **2000 – 2002**

Perform data analysis, interpretation and review of high-resolution GC/MS results for dioxins, PCBs, pesticides and PAHs. Operation of VG70 and Autospec Ultima high-resolution GC/MS spectrometers for those analyses according to standard operating procedures. Routine maintenance and troubleshooting of instruments. Advise management on changes and improvements to processes, new technology, and methods.

Professional Experience – Other

QA/QC Chemist – Axys Analytical Services, Sydney, BC, Canada **1999 - 2000**

Perform data analysis, interpretation and review of high resolution and low resolution GC/MS results for dioxins, PCBs, organochlorine pesticides and polycyclic aromatic hydrocarbons. Final authority for the release of analytical results. Prepare final narratives and data packages. Answer client questions and concerns regarding reported results and analytical methods.

Douglas Weir

Consulting Scientist – Weir Environmental Consulting, Naperville, IL 1993 - 1995
Provided captive consulting services for Growth Environmental Services, a national provider of environmental engineering and testing services. Upgraded, updated and computerized the analytical QA/QC programs for Growth's laboratories. Assisted Growth's Chicago laboratory in obtaining the Illinois State Drinking Water Certification as well as its analytical testing license for the state of Wisconsin. Wrote Growth's application for the Florida Department of Environmental Regulation certification.

Laboratory Director — Quality Analytical Labs, Lisle, IL 1990 - 1992
Responsibilities included hiring of all analytical staff, equipment specification and purchase, development of the laboratory budget and creation of the strategic plan for the growth of QAL in the Chicago market. Introduced QAL SOPs for EPA-SW-846 method compliance. Set up and oversaw the QAL quality control, chemical hygiene and waste disposal programs. Brought the facility into full OSHA and USEPA compliance, and ensured compliance with individual state underground storage tank (UST) regulations in EPA Region V.

Group Leader, Organic Photochemistry – University of Notre Dame 1987 - 1990
Headed a research group consisting of 3 staff scientists, 1-3 postdoctoral fellows and 2-3 graduate students/technicians investigating a variety of topics and performing various studies.

Education

- Ph.D, Physical Chemistry – Queen's University (1984)
- BS Chemistry – Queen's University (1980)

Professional Training

- Ethics Training
- Customer Service Training
- ISO 9000 Lead Auditor Certification (SGS, IRCA certified)
- Certified Quality Auditor, American Society for Quality

Personnel Resume

Karen Sellers

Project Manager

Qualifications Summary

Ms. Sellers has worked in the environmental laboratory field since 1997. She has progressed through increasingly responsible positions within the company to her present role as Project Manager. She is well versed in the methods available and their applicability to various environmental matrices and regulatory programs. Her professional experience, attention to detail and dedication to customer satisfaction are the keys to her success.

Professional Experience

Project Manager

TestAmerica Laboratories, Inc., West Sacramento, CA - March 2007 to Present

Her responsibilities include acceptance and initiation of projects into the laboratory, communication of client specific requirements to the lab, coordination and review of analytical laboratory projects for both government and commercial clients including the pulp and paper industry and foreign clients. Project management includes recommendation of appropriate tests and methods, project LIMS set-up, QAPP review, and final report review. In addition, Ms. Sellers is often the initial contact for new client and potential business opportunities. She evaluates bids and proposals, prepares price quotations and gives technical responses. Her goal is to accommodate the customer by immediate response to all inquiries and requests.

Project Manager/Business Development, Client Services

Columbia Analytical Services, Sacramento, CA - May 2005 to January 2007

Ms. Seller's primary responsibilities include client development and increase sales for the northern California laboratory location by initiating client meetings and introducing new laboratory services and developments. She also acted as a project manger and local liaison for on-going projects and key clients. Ms. Seller's responsibilities also included managing the Northern California Service Center to provide support to local projects.

Project Manager, Client Services

Columbia Analytical Services, Redding, CA – April 1999 to February 2005

In an environmental laboratory setting, oversee projects from quote initiation to final report submission. Assist clients in determining analysis needed. Act as a liaison between the client and the laboratory to coordinate needs with capacity. Respond promptly to client questions and requests. Update them on progress of their projects. Major accomplishments include:

- Initiating a format for compiling and tracking project specifications to ensure that laboratory data is compliant with specific client program requirements.

- Successfully managed laboratory workload of approximately 1M annually, with an average of approximately 10 major projects per year.
- Assisted in expansion of lab capabilities to the federal (DoD) market by effectively communicating federal Quality Assurance Project Plan (QAPP) requirements and ensuring that these requirements were consistently met during all phases of work.

Lab Technician, Sample Management
Columbia Analytical Services, Redding, CA - October 1997 to March 1999
Ms. Sellers was responsible for the management of sample custody area. Among the duties was receiving and unpacking client samples, labeling and log-in of the samples and maintaining in-house chain of custody. She was also responsible for communication of short holding time samples to the lab and initiating packing of samples to be subcontracted to other facilities as well as filling bottle orders and assisting clients with technical guidance pertaining to sample collection.

Registered Environmental Health Specialist
Shasta County Environmental Health Division, Redding, CA – January 1990 to May 1997
Enforced state and local environmental health regulations relating to drinking and industrial water wells, public swimming pools, retail food facilities, housing, land-use and hazardous materials. Managed the retail food facilities program for five years during which time I initiated and edited a newsletter, initiated a food handler training program and investigated food-borne illness complaints. Also formulated food program policies and worked closely with local, state and federal officials to maximize enforcement.

Education

BS in Environmental Science

University of Massachusetts, Amherst, MA – 1982

AS in Environmental Science

Holyoke Community College, Holyoke, MA - 1979

Accomplishments/Awards

California Registered Environmental Health Specialist (REHS) # 5800
California Certified Hazardous Materials Technician # 967

Appendix B

CD-ROM Including: Laboratory Quality Assurance Manuals

Legend Technical Services, St. Paul, MN
TestAmerica, West Sacramento, CA

LABORATORY QUALITY ASSURANCE MANUAL

Volume 24

Book #352

Origination Date: April 13, 1992

Revised: May 3, 2011

LEGEND TECHNICAL SERVICES, INC.
88 Empire Drive
St. Paul, MN 55103
651-642-1150

MISSION STATEMENT

Legend Technical Services, Inc. (LEGEND) is dedicated to promoting public health and supporting the environment by providing reliable, legally defensible analytical and consulting services to a wide variety of clientele in an efficient, timely manner.

LEGEND'S primary goal is to maintain extensive experience and flexibility in order to provide a diverse range of services to our clients, giving them the best analytical and consulting services value. LEGEND is committed to continuous improvement in the quality and scope of services provided.

Information contained within this document is confidential and privileged. It is intended for the use of the entity to which it has been given and may not be reproduced.

LEGEND TECHNICAL SERVICES, INC.
QUALITY ASSURANCE MANUAL
May 2011 – Volume 24

Cheryl Sykora, CIH, CSP, CHMM
President
Industrial Hygiene Technical Director
Date: 5/3/11

William Welbes
Vice President of Operations
Date: 5-3-11

William Dahl
QA/QC Coordinator
Inorganic Chemistry Technical Director
Date: 5/3/11

Tom Barrett
Organic Chemistry Technical Director
Date: 5/3/11

Van Pham
Organic Department Manager
Date: 5/3/11

Dan Brezina
Inorganic Department Manager
Date: 5/3/11

Corey Campbell
Marketing Director
Date: 5/3/11

Table of Contents

SECTION 1

SCOPE

1 Scope

The objective of the Legend Technical Services, Inc. (LEGEND) Quality Assurance Program and its systems is to verify that the laboratory data including field observations and analytical data for the client are of good quality generated by using good laboratory practices, and meet all applicable regulatory requirements. The quality assurance manual covers the quality management system consistent with the ISO 9001:2008 and ISO/IEC 17025:2005 (E) standards for the analysis, data collection and reporting of results on samples initiated within LEGEND.

Design is excluded from the ISO scope because LEGEND does not have design responsibilities. Any design decisions are driven by the client and would fall under customer oriented processes.

SECTION 2

REFERENCES

2 References

The quality systems and procedures described in this manual are based on the requirements of ISO 9001:2008, ISO/IEC 17025:2005 (E), EPA QA/G-5, state agencies, federal programs, AIHA, A2LA, NELAC and NVLAP.

SECTION 3

TERMS AND DEFINITIONS

3 Terms and Definitions

This quality assurance manual applies the terms and definitions given in ISO 9001:2008 and ISO/IEC 17025:2005 (E). The term “organization” refers to LEGEND to which the ISO 9001:2008 and ISO/IEC 17025:2005 (E) standards apply.

In this quality manual, the term “product” refers to product and services LEGEND delivers to the market place. Internal services for internal customers are treated as interim steps to satisfying the end-user. Therefore, customer service support and product design information are internal products driving the deployment of the final product to the end-user.

Appendix A lists definitions for common terms and abbreviations.

SECTION 4

QUALITY MANAGEMENT SYSTEM

4 Quality Management System

4.1 General Requirements

The quality assurance manual specifies requirements for a quality management system that helps guide LEGEND to demonstrate its ability to consistently provide service that meet customer needs as well as applicable regulatory requirements and enhance customer satisfaction through continual improvement of services.

4.1.1 Quality Management System Model

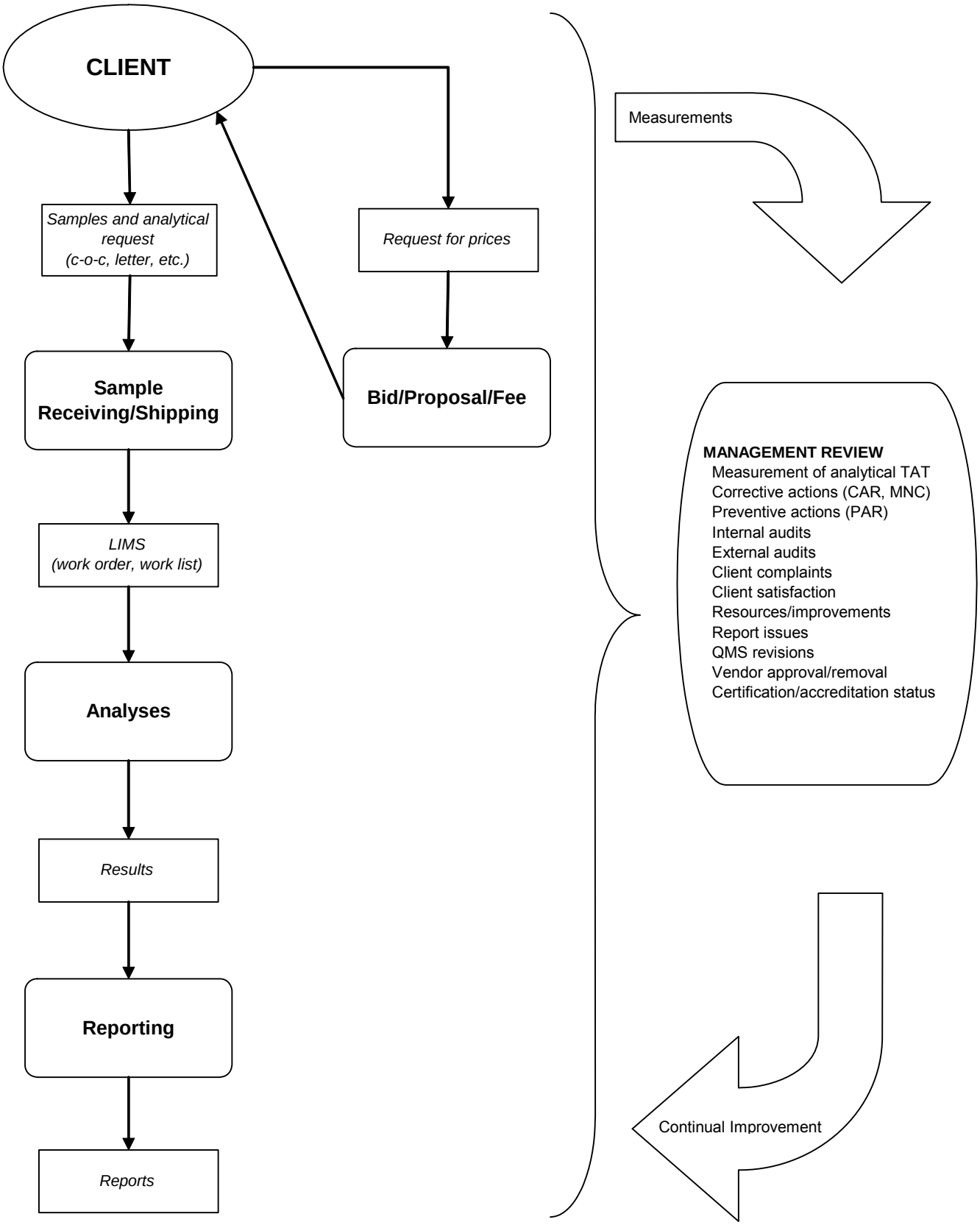
A. Determines criteria and methods needed to ensure that both the operation and control of these processes are effective.

B. Ensures the availability of resources and information necessary to support the operation and monitoring of these processes.

C. Ensures the monitoring, measuring and analysis of these processes.

D. Ensures the implementation of actions necessary to achieve planned results and continual improvement of these processes.

Quality Management System (QMS) Model



4.2 Documentation Requirements

4.2.1 General

The quality management system documentation consists of the following:
Quality Assurance (QA) Manual, Standard Operating Procedures, Work Instructions, and Quality Records.

4.2.2 Quality Manual

The QA Manual provides the overall policy for the laboratory. It follows ISO guidelines. The QA Manual includes:

A. The scope of the quality management system (Section 1).

B. The documented procedures established for the quality management system, or reference to them.

C. A description of the interaction between the processes of the quality management system (Section 4.1).

D. The quality policy (Section 5.3).

The QA Manual shall require the approval of the President before changes are issued. The QA Manual is numbered and a distribution list maintained.

4.2.3 Control of Documents

This QA Manual and the referenced supporting documents are controlled per the requirements of ISO 9001:2008 and ISO/IEC 17025:2005 (E). The documentation of the quality management system for the most part is electronically controlled. Any employee using a printed document is required to verify that the version is current. The main documents are Standard Operating Procedures (SOP), Work Instructions (WI), Forms, and Guidance Documents (GD).

A. Quality Assurance Manual (QAM)

The QA/QC Coordinator shall review and update the QAM when there is a change to a quality policy and/or procedure outlined in the QAM or every two years at a minimum. The QAM shall require the approval of the President before changes are issued. The QAM is numbered and a distribution list maintained.

B. Standard Operating Procedures (SOP)

SOPs shall be developed and implemented for all routine, standardized, and/or special/critical operations. The procedure for preparation of an SOP can be found under SOP 'Preparation of Departmental SOPs'.

SOPs have been divided into four groups, to reflect the different operations as outlined below:

1. General Operations Standard Operating Procedures

This group contains the SOPs required for daily operation of office and business functions, including QA activities.

2. Equipment Standard Operating Procedures

   This group contains the SOPs required for use of field and laboratory equipment.

3. Laboratory Operations Standard Operating Procedures

   This group contains the SOPs required for all facets of lab operations including calibration and specific procedures. It is divided further into environmental (LABENV), industrial hygiene (LABIH), and industrial chemistry (LABIND).

4. Field Operations Standard Operating Procedures

   This group contains the SOPs required for all facets of field operations including calibration and specific procedures.

The current revision of each SOP is stored on-line in the laboratory's network. Paper copies are allowed within the individual departments as long as the revision being followed has been checked against the current revision on-line.

All SOPs are updated and re-issued as needed and are re-evaluated approximately every two years, or more frequently when changes have been made to references or the procedure, to determine suitability for continued use.

SOPs that are revised will retain their original number with an additional number after a decimal point to indicate the revision number, i.e., first revision of an environmental SOP LABENV-001 would be designated SOP LABENV-001.1. Copies of all SOP revisions are kept in the QA department.

New SOPs shall be reviewed by a technical reviewer, approved by the QA/QC Coordinator and authorized by the President.

C. Work Instructions (WI)

Work Instructions (WI) are brief, detailed instructions outlining a specific, routine task. A copy of the Work Instruction is usually located where the task is performed. The procedure for preparation of a WI can be found under SOP 'Preparation of Work Instructions'.

WIs that are revised will retain their original number with an additional number after a decimal point to indicate the revision number, i.e., first revision of WI-001 would be designated WI-001.1. Copies of all WI revisions are kept in the QA department.

D. Forms

Forms are developed to aid in the documentation process and may be accompanied by an SOP. Forms have been divided into the following categories:

1. General (GEN)

   This group contains forms that may be used by everyone in the company.

2. Asbestos (ASB)

   This group contains forms related to asbestos field and laboratory work.

3. Equipment (EQUIP)

   This group contains forms related to equipment use.

4. Field (FLD)

   This group contains forms related to fieldwork.

5. Laboratory (LAB)

   This group contains forms related to laboratory work.

Each form is assigned a unique number under its specific group and an issue date. When a form is reviewed and changes are made, the date changes to reflect the date of change. Forms are revised on an as needed basis. An index of forms maintains a record of each form, its revision date and its title. Copies of all current 'LAB' forms are kept by the QA department.

E. Guidance Documents

A Guidance Document (GD) is a general outline of a protocol. The procedure for preparation of a GD can be found under SOP 'Preparation of a Guidance Document'.

F. Quality Assurance Project Plans (QAPP)

QAPPs are project specific manuals that may be prepared where a project requires unique or different quality assurance requirements or when they are required by regulatory agencies. They require the approval of the Client Manager and/or QA/QC Coordinator and shall be signed and dated before issuance.

All obsolete documents, excluding forms, are retained for either legal or knowledge preservation purposes and become part of the records system. These documents will be marked to indicate they are no longer current. Quality system documents generated by the laboratory will be uniquely identified, including date of issue and/or revision date, and page numbers with the total number of pages in the document (if applicable). The issuing authorities are defined as follows:

QA Plan:	QA/QC Coordinator
Form:	QA/QC Coordinator/IH Administrative Assistant
SOP:	QA/QC Coordinator
WI:	QA/QC Coordinator
GD:	QA/QC Coordinator

4.2.4 Control of Records

All information recorded in support of technical or quality activities are records. All records are maintained in good order by the responsible department. Records are stored in temperature and humidity controlled environments with all employees having access. Records are retained for a minimum of five years with the following exceptions: TO-15 laboratory records – maintained for a period of at least ten years, Material Safety Data Sheets (MSDS) – one version for the life of the chemical and Certificates of Analysis (C of A) – retained for 5 years after last data record that the standard has been used with. Records are disposed of by recycling and/or as general trash. A spreadsheet listing the indexing, ISO reference number, and storage of records is found on the next page.

Control of Records

Record	ISO 9001:2008 Ref. #	Indexing	Storage
QA Manual (archived)	4.2.2	Date	QA Department
SOPs/GDs/WIs (archived)	4.2.3	Date	QA Department
Final Reports	4.2.4	Project/Work Order #	File room and archive area
External Audits	4.2.4	Type, date	QA Department
Material Safety Data Sheets	4.2.4	Alphabetical	Laboratory
Certificates of Analysis	4.2.4	Manufacturer, unique ID	Laboratory
Management Reviews	5.6.1	Date	QA Department
Training	6.2.2	Date	Current – analyst Archive – personnel file
Job Descriptions / Resumes	6.2.2	Alphabetical	Listing – computer Signed – personnel file
Proposals / Quotes / Contracts	7.2.1	Project/Work Order #	Single project – project file Multiple projects – client specific file
Review of requirements	7.2.2	Project/Work Order #	Project file
Subcontractor / Vendor Evaluations	7.4.1	Alphabetical	QA Dept. / Accounting Dept.
Raw Data (runs, chromatographs, etc.)	7.5.2	Date	Current – analytical department Archive – archive area
Instrument Run Logs	7.5.2	Log #	Current – analytical department Archive – archive area
Daily Files	7.5.2	Date	Current – analytical department Archive – archive area
Proficiency Testing	7.5.2	Project/Work Order #	QA Department
Control Charts	7.5.2	Date, department	QA Department
Client Information (samples, project, etc.)	7.5.3, 7.5.4	Project/Work Order #	Project file / LIMS
Instrument Calibration/ Maintenance Logs	7.6	Log #	Specific department, QA Dept. or archive area
Computer Software Validation	7.6	Date	QA Department
Internal Audits	8.2.2	Audit #	QA Department
Release of product	8.2.4	Project/Work Order #	Daily and/or project file
Corrective Action Report (CAR)	8.3 / 8.5.2	CAR #	QA Department
Preventive Action Report (PAR)	8.5.3	PAR #	QA Department

A. Project Files

1. Project files are maintained in accordance with LEGEND's 'Project File Maintenance' SOP. Separate record packages are maintained for each project and are filed according to project number. Project files are maintained in a controlled area. Working files may be established by test during sample analysis and data reduction. The designated client manager is responsible for set-up and organization of his/her project files.

2. Completed files may consist of raw data sheets (i.e. chromatograms and bench sheets), correspondence with the client, chain of custody forms, etc.

3. All records of review, including any significant changes, shall be maintained and noted on the chain-of-custody or in the case narrative. All observations, data, and calculations shall be recorded at the time they are made. The laboratory management does have the authority for permitting departures from documented policies and procedures or from standard specifications. This deviation is documented in the project file, along with management's approval. The client may be consulted and/or informed if the deviation impacts the data.

4. The records will indicate if the laboratory subcontracted any of the work.

   a) For projects requiring specific accreditation, the client shall be informed of subcontracted work in writing. When appropriate, the client will approve the work, preferably in writing.

   b) LEGEND is responsible to the client for the timeliness that the work is coordinated with the subcontractor and the completeness of the resulting data in the final report. LEGEND is not responsible to the client if the client or a regulatory authority indicates which subcontractor is to be used.

5. The client shall be informed if any deviations occurred from the original request.

6. Completed and closed files are transferred to a secured file storage area where laboratory personnel may access them.

B. Laboratory Records

1. All records on original observations, calculations, derived data, and calibrations are recorded. The laboratory maintains a copy of the test report. Each record for each test shall contain sufficient information to permit their reconstruction at a future date. The records will include the person involved in sampling (if available), sample preparation, and sample testing, where appropriate.

2. When mistakes occur in the records, each mistake shall be crossed out with a single line, the correct entry made, and the correction initialed and dated by the individual making the change in ink.

3. Laboratory records fall into two major categories:

   a) Documents that reflect overall laboratory operation such as instrument log books and control charts.

b) Documents specific to a group of samples such as chain-of- custody and raw analytical data.

4. Raw data is defined as the record of observations on a project including laboratory/field notebooks, bench sheets, memoranda, printouts, strip charts, photographs, magnetic media (tape and disc) and any other methods used to capture and record original data.

5. All data recording sheets, calculation sheets, chromatograms, bench sheets etc., shall be dated and signed by the analyst. (Multiple page chromatograms may have the top sheet only, signed & dated.)

6. Any additions to the raw data shall include the original data indicating the reason for change, the date changed, and who was responsible for making the change. If a correction needs to be made to the data, a single-line crossing out the error will be made through the incorrect information. Beside the correct information, record the initials of the person who made the change and the date when the change was made.

7. All laboratory records from time of sample receipt through data reporting and sample disposal shall be available if requested by clients, an authorized regulatory agency, or court. No other outside person or persons shall have access to the laboratory files without written /verbal permission from the client. Records are maintained in the project and daily files.

C. General Laboratory Operations Records

The laboratory departments and/or QA/QC Coordinator shall maintain the following records:

1. Instrument Calibration and Maintenance Logs

A separate log shall be maintained for each instrument listing all maintenance and calibration performed in-house or by outside groups. These logs shall be maintained in the laboratory during use and then archived within the individual department. This maintenance log shall store all calibration reports performed by outside sources.

2. Proficiency Testing Records

The QA/QC Coordinator shall maintain a record.

3. Certification Program Records

Records shall be maintained by the QA/QC Coordinator of all correspondence, analytical data, agency results and certification of performance from all certification programs.

4. Control Charts

Charts shall be filed chronologically. Charts used to define acceptance control limits are printed and archived for a minimum of 5 years. Charts used for

trending are not archived. All control charts can be reproduced as data is stored electronically in the LIMS system.

5. Audit Records

   Formal audit reports of internal and external performance audits shall be filed with the QA/QC Coordinator.

6. Computer Software Verification

   Laboratory data systems are verified by comparing independent calculations to the results generated by the data system. Commercial data systems are verified when the system is upgraded (or downgraded) to a new version of the software. Laboratory generated spreadsheets are verified on an annual basis. Laboratory generated spreadsheets are controlled, protected, and locked from manipulation by the analyst. If an uncontrolled spreadsheet is used to assist an analyst then it is considered the same as a hand calculation and all results for that spreadsheet must be peer reviewed including a review of the calculation.

7. Training Records

   Resumes, external training, and in-house training records shall be maintained in the employee file.

8. Master Corrective Action Log

   The QA/QC Coordinator shall maintain a copy of all Corrective Action Reports (CAR).

9. Master Preventive Action Log

   The QA/QC Coordinator shall maintain a copy of all Preventive Action Reports (PAR).

10. Instrument Run Log / LIMS Sequence

    A list of samples run on each instrument shall be recorded in logbooks or tracked in the LIMS system via use of a LIMS sequence and maintained by the analysts.

11. Standard Operating Procedures / Work Instructions

    A file of historical laboratory SOPs and WIs with issue dates shall be maintained by the QA/QC Coordinator.

12. Subcontractor QA Sample Records

    The QA/QC Coordinator shall maintain results of any QC samples submitted to subcontractors.

13. Vendor Evaluations

The Accounting Department shall maintain completed vendor evaluations.

14. Reports

The results of each test, or series of tests, shall be reported accurately, clearly, unambiguously, and objectively, in accordance with any instructions in the test method. The results should be reported in a test report and should include all the information necessary for the understanding of the test results. If a statement of compliance with a specification is made, the clauses of the specifications that are met or not met shall be made. If requested by the client, the scope of the work may be expanded to include the uncertainty of measurement as part of the report.

15. Proposals/Quotes/Contracts

All requests, tenders, and contracts will be reviewed to ensure:

a) The requirements, including the methods to be used, are adequately defined, documented, and understood.

b) The laboratory has the capability and resources to meet the requirements.

c) The appropriate tests and/or methods are selected and capable of meeting the client's requirements.

d) If a contract needs to be amended after work has commenced, the same contract review process shall be repeated and any amendments shall be communicated to all affected personnel.

The items listed above are further described under SOP 'Preparation of Proposals and Use of General Conditions and Sub-Contract Agreements'

SECTION 5

MANAGEMENT RESPONSIBILITY

5 Management Responsibility

5.1 Management Commitment

A. LEGEND is committed to the development, implementation, and continual improvement of its quality management system. This is accomplished by:

1. Communicating to the organization the importance of meeting customer as well as statutory and regulatory requirements
2. Maintaining the quality policy
3. Ensuring that quality objectives are established
4. Conducting regular management reviews
5. Ensuring the availability of resources

B. Management's mission is to foster, through teamwork, the development of a proactive quality program. It is management's responsibility to ensure that LEGEND's processes, documentation, and service is of the type and quality expected by the customer. Senior management's responsibilities in this endeavor include, but are not limited to:

1. Defining the functional responsibilities of management and staff.
2. Establishing levels of accountability and authority.
3. Creating and supporting lines of communication for planning, implementing, and assessing our programs and services.
4. Providing adequate resources to implement the QA program.
5. Performing an annual assessment of the quality process.
6. Using the internal assessments and those of outside auditors to determine what response/actions are appropriate and implement them.
7. Taking the initiative to remove any barriers that hinder the organization from meeting quality objectives.
8. Ensuring the quality program is reviewed and updated to reflect organizational or policy changes.
9. Ensuring the adequacy of resources and personnel that has the necessary education, training, technical knowledge and experience for their assigned functions to achieve and assure quality in all activities.
10. Formulating the goals with respect to the education training and skills of the laboratory personnel.
11. Ensuring management and personnel are free from any undue internal and external commercial, financial or other pressures and influences that may adversely affect the quality of their work.

12. Ensuring the protection of the customers' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results.

13. Ensuring the data integrity system policy and procedures are followed by all laboratory personnel.

C. Data Integrity System

1. The data integrity system at LEGEND ensures that an ethical approach is being applied to all planning, training, and implementation of laboratory work. To accomplish this goal, LEGEND has implemented a data integrity system that encompasses the following five requirements:

a) A data integrity training program: standardized training is given to each new laboratory employee and a yearly refresher is presented to all laboratory employees. Key topics within this training include:

- Need for honesty in analytical reporting
- Process for reporting data integrity issues
- Specific examples of unethical behavior and improper practices
- Documentation of non-conforming data that is still useful to the data user
- Consequences for identified unethical behavior

b) Signed data integrity documentation for all laboratory employees: this includes a signed training attendance and written agreement to abide by the LEGEND data integrity procedures.

c) Periodic monitoring of data integrity: including technical data review, internal data audits, proficiency testing, etc.

d) Documentation of any review or investigation into possible data integrity infractions. This documentation, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be available for review by laboratory assessors and must be retained for a minimum of 5 years.

e) Confidential reporting of data integrity issues is encouraged via anonymous written narrative submitted to QA.

5.2 Customer focus

Customer satisfaction is critical for the success of LEGEND. Customer interaction is a key to understanding customer needs and is accomplished through multiple means including direct sales/marketing contacts, client managers' interactions, management interactions, and specific performance measurements provided by several customers.

5.3 Quality Policy

LEGEND is committed to meet customer requirements. We are focused on meeting our customer's quality expectations through continual improvement and uncompromising business ethics.

Quality is a team effort and all personnel are responsible for knowing the documentation. Not only is it expected that one implement the policies in one's own work but also support one another to ensure full compliance.

The purpose of the management system is to guide LEGEND to demonstrate its ability to consistently provide service that meets customer needs as well as conforms to applicable international standards, meets regulatory requirements, and enhances customer satisfaction through continual improvement of services.

LEGEND seeks to establish a standard of service where clients can expect data of highest integrity as well as timely deliverables. Improvement in both areas is bench marked with the goal to constantly strive for better performance.

This policy is communicated to the organization and reviewed for continuing suitability.

5.4 Planning

5.4.1 Quality Objectives

Quality objectives are documented in the Management Review Summaries.

5.4.2 Quality Management System Planning

The planning of the quality management system is carried out to ensure the integrity of the model as stated in section 4.1 and to meet the quality objectives, quality plan, and strategic plan.

5.5 Responsibility, Authority, and Communication

5.5.1 Responsibility and Authority

LEGEND's management team manages the quality management system. A copy of the organizational chart is on the following page.

A. Technical Director

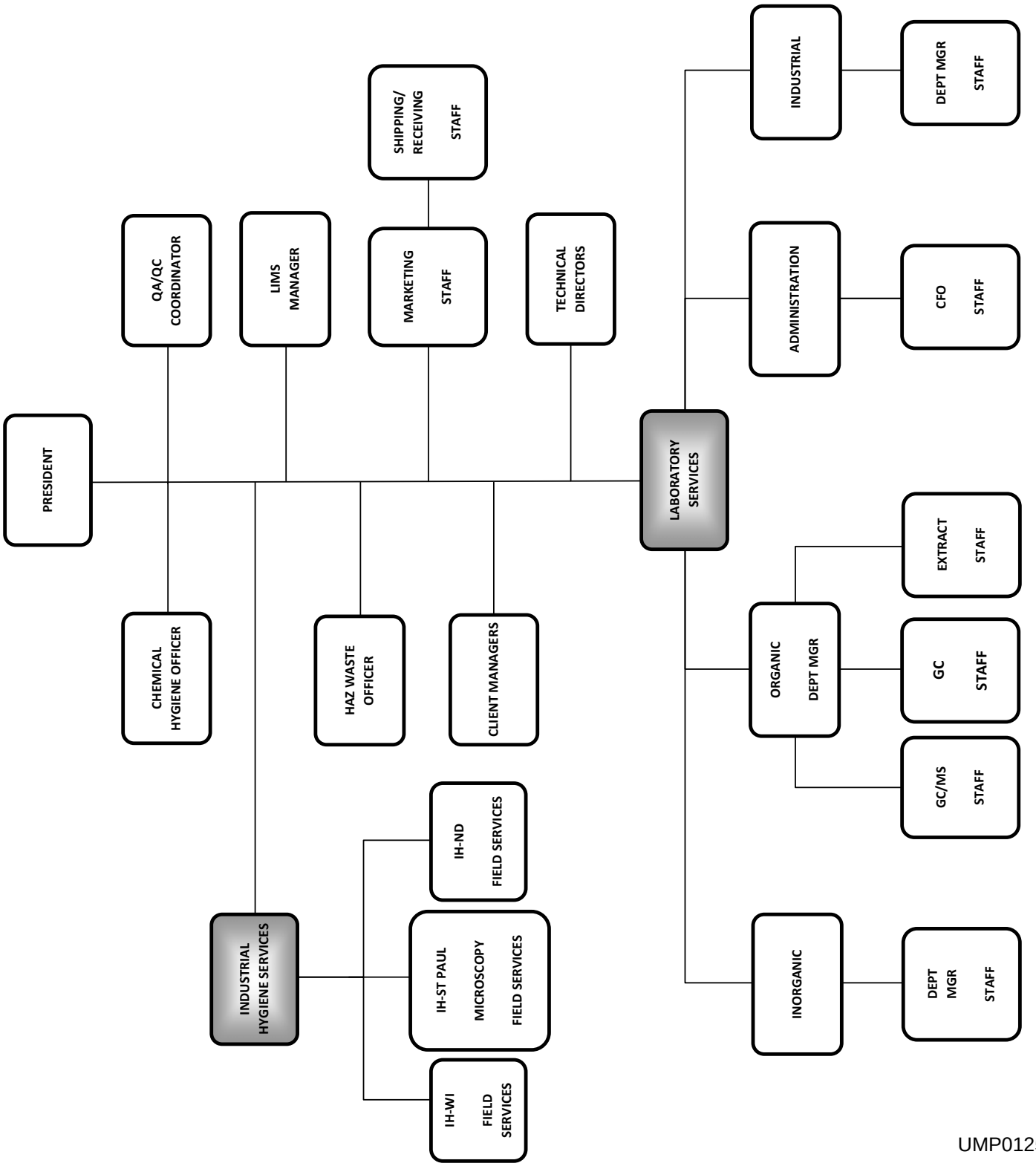
1. Report to President.

2. Responsible for developing procedures, bringing equipment on-line, and maintaining current with regulatory requirements, state-of-the-art technologies, and certification requirements.

3. Assist the QA/QC Coordinator in obtaining laboratory certification/accreditation.

4. Prepares justification statements for capitol expenditures.

5. Assists supervisors in employee reviews including changes in compensation.


PRESIDENT
QA/QC COORDINATOR
LIMS MANAGER
SHIPPING/ RECEIVING STAFF
MARKETING STAFF
TECHNICAL DIRECTORS
CHEMICAL HYGIENE OFFICER
HAZ WASTE OFFICER
CLIENT MANAGERS
INDUSTRIAL HYGIENE SERVICES
IH-ND FIELD SERVICES
IH-ST PAUL MICROSCOPY FIELD SERVICES
IH-WI FIELD SERVICES
LABORATORY SERVICES
INDUSTRIAL
DEPT MGR STAFF
ADMINISTRATION
CFO STAFF
ORGANIC DEPT MGR
EXTRACT STAFF
GC STAFF
GC/MS STAFF
INORGANIC
DEPT MGR STAFF

B. Department Manager

1. Reports to the President.

2. Reviews data generated by their staff.

3. Responsible for instrument performance, calibration, and preventive maintenance.

4. Takes an active role in cross training.

5. Reports out-of-control situations to the QA/QC Coordinator by completing Corrective Action Reports.

6. Maintains adequate and appropriate quantities of laboratory supplies.

7. Is responsible for training and continuing compliance of analysts with methods, SOPs, and quality assurance requirements.

C. Sample Preparation/Analysis Personnel

1. Perform methods, data recording, and data validation using prescribed methods.

2. Report out-of-control situations and nonconformances to the Department Manager.

5.5.2 Quality Assurance/Quality Control Coordinator - ISO Management Representative

The QA/QC Coordinator is the designated quality management representative. The responsibilities include ensuring that processes needed for the quality management system are established, implemented, and maintained, reporting to top management on the performance of the quality management system and any need for improvement, and ensuring the promotion of awareness of customer requirements throughout the organization.

A. Performs statistical analysis on quality control data.

B. Reviews statistical data from laboratory quality control samples.

C. Maintains the quality manual.

D. Maintains records and archives of quality assurance data.

E. Responsible for assuring documentation and resolution of nonconformances.

F. Stops production of laboratory data when quality control data demonstrates significant trend problems.

G. Coordinates laboratory certification/accreditation.

H. Coordinates laboratory quality assurance audits.

I. Reports to the President, Technical Directors, and Department Managers on the

status of quality control program and audit results.

J. Recommends methods, standard operating procedures, and quality control procedures to the appropriate personnel.

K. When absent, the applicable Technical Director assumes the responsibility.

L. Authorizes resumption of the production of laboratory data when corrective actions have been implemented and proven effective.

5.5.3 Internal Communication

The top management conducts annual management review meetings to discuss LEGEND's performance results including quality and customer service indices.

5.6 Management Review

5.6.1 General

The purpose of the review is to evaluate the overall performance of the quality management system and identify improvement opportunities. Reviews are held on an annual basis. Meetings are rescheduled if the President, Industrial Chemistry Technical Director or the QA/QC Coordinator can not be there or if 50% of the following can not attend: department managers, supervisors, or marketing. Records of these reviews are documented and maintained for future reference.

5.6.2 Review Input

Inputs to the quality management system review will include the following:

A. Customer satisfaction

B. Results of audits

C. Product conformity

D. Status of preventive and corrective actions

E. Changes affecting the quality management system (e.g. quality policy, documents, regulatory standards, certifications, vendor approval/removal)

F. Recommendations for improvement/resources identified

G. Report of proficiency testing results

H. Supervisory/managerial reports

Follow-up actions from previous management reviews are addressed within each section. Management will retain and exercise the responsibility for defining and implementing an effective quality assurance program for the organization.

5.6.3 Review Output

The output of the management review summarizes the decisions made and actions assigned for improvement of the quality management system and processes, customer satisfaction improvement, and any resource needs.

SECTION 6

RESOURCE MANAGEMENT

6 Resource Management

6.1 Provision of Resources

Resource needs are identified and allocated to implement and maintain the quality management system and continually improve its effectiveness, and to enhance customer satisfaction by meeting customer expectations.

6.2 Human Resources

6.2.1 General

The selection process and on-going training of individuals performing work affecting product quality is designed to match individuals with appropriate education, training, skills, and experience to the job.

6.2.2 Competence, Awareness, and Training

Job descriptions contain all appropriate requirements for job openings and are used to review and select the right candidates for the job. The job qualifications and requirements help in the selection of the individual with the best set of skills and experience. Appropriate records of education, training, skills and experience are maintained for all employees by the Human Resources Director.

Training at LEGEND is primarily accomplished through on-the-job training. Initial training includes employee orientation, health and safety orientation, and data integrity training (laboratory personnel only). If the task has an SOP or Work Instruction, the trainee must read the document before initiating training. The trainee and supervisor/trainer must sign off acknowledging the reading of the SOP. This record is maintained with the trainee until completely filled and then it is placed in their personnel file. Trainees will observe the job as performed by qualified personnel and may participate in phases of the task at the supervisor/trainer's discretion. A trainee may not perform the task alone until the supervisor/trainer has signed off on the task. Some of the tasks will also require a completion of an Initial Demonstration of Capability (IDC) to show effectiveness of the training. The IDC information is listed in each applicable SOP. Where appropriate or required, training procedures are written. Training procedures should include trainer qualifications, training content, training duration, an IDC procedure and documented authorization to perform the specific tasks.

Laboratory personnel must demonstrate continuing capability of the analytical methods used. This can be accomplished through successful analysis of proficiency testing samples, analysis of laboratory control spikes, or other methods appropriate to the specific analysis. The interval for ongoing demonstration is specific to the test or program governing the test. Personnel involved with the analysis, sample preparation, and data review of a sample must sign off acknowledging the reading of the most current SOP used in the laboratory. Data integrity refresher training is conducted annually.

Public and private personnel files are maintained for each employee (SOP 'Personnel File Maintenance'). Items that may be contained in each are listed below. Public files are open for review by any employee or outside agency. Private files are available only to the Human Resources Director. Any record placed in the public file may not contain the employee's Social Security Number.

A. Public File

1. Resume: current resume for each employee is maintained and updated by the employee.

2. Job Description(s): a signed copy of each applicable job description is maintained.

3. Training: a training log outlining all training activities including on-the-job-training are kept up-to-date and maintained by each employee. Completed logs are archived in the personnel file. Copies of all course completion certificates for external training are also included.

4. Initial Demonstration of Capability (IDC): shall be performed by the analyst initially to show effectiveness of training and when there is a significant change in instrumentation or published test method. Completed IDCs are archived in the personnel file.

5. Continuing Demonstrations of Capability (CDC): shall be performed by the analyst at an interval specific to the test or program governing the test. Completed CDCs are archived in the personnel file.

6. Medical Surveillance: for those employees required to wear respiratory protection during job activities, personnel file documentation includes doctor sign-off that the individual can wear a respirator and respirator fit test records.

B. Private File

1. Formal Reviews/Salary Actions: should be initiated annually by the employee completing an 'Employee Self-Evaluation' form for each of their job descriptions. The completed form(s) are submitted to their supervisor. The written review requires input from both the staff and their supervisor and is designed to positively direct each individual's career and to implement growth and change from within the organization. The review shall also identify training needs and how these needs are to be completed. Salary actions are initiated as needed by the review or as required due to a change in status. These changes are documented on an employee status change form.

2. Tax Forms: each employee's W-9 form, W-4 form, etc. are maintained.

3. Employee Application: each employee's job application form is maintained.

4. Medical/Life/401K: each employee's insurance and 401K information are maintained.

5. New Hire Forms

Where contracted and additional technical and key personnel are used, the laboratory shall ensure that such personnel are supervised and competent, and that they work in accordance with the laboratory's quality system.

6.3 Infrastructure

A. LEGEND has an infrastructure designed to support the company needs to achieve conformity to service and product requirements. The infrastructure includes but is not limited to buildings and associated facilities, instrumentation including hardware and software, and supporting services such as transport and communication.

B. LEGEND's St. Paul, Minnesota operations are housed in a 30,000-ft^2, one-story commercial building located at 88 Empire Drive. The building is air-conditioned and has sufficient three-phase power and water to meet our equipment's requirements. Backup power, backup air-conditioning, and a 24 hour security system are also provided. A floor plan and key for the laboratory layout are on pages 26-29.

C. The laboratory wide deionized water is produced using a system maintained by US Filter/Siemens. The specification is a resistivity of >16.3 megaohm/cm. For certain applications including but not limited to metals, anions, and wet chemistry the water is further passed through a Barnstead Nanopure Diamond™ system. The water purity level is checked and recorded each day of use.

D. Major equipment is classified as calibration (C), reference (R) or hood (H).

1. Calibration equipment is defined as a monitoring or measuring device that yields a value used within an analysis. 'CP' refers to periodic calibration (e.g. balances, thermometers, weights, etc.) and 'CO' refers to operational calibrations (e.g. GC, spectrophotometer, sampling pump). Section 7.6 further defines calibration requirements.

2. Reference equipment is typically used in techniques to prepare the sample (e.g. pellet press, ASE, steam bath).

3. Hoods are located throughout the individual laboratories to limit exposure to hazardous chemical fumes, remove instrument exhaust and/or asbestos.

E. On the following pages are a list of the major equipment indexed by 'C', 'R' or 'H' and a unique number. The record includes the manufacturer, instrument name, location, model #, serial #, and operating manual location. Thermometers are indexed separately. The list is subject to change but the information is updated annually.

EQUIPMENT INFORMATION

New #	Manufacturer and Instrument Name	Location	Model #	Serial #	Software Version	Operating Manual Location	Manual Version
CO-1	HP GC-PID1/FID 1	Organic	5890	3140A38930	Target 4.14	By equip	1989
CO-2	HP GC-PID2/FID 2	Organic	5890	3140A39036	Target 4.14	By equip	1989
CO-3	HP GC-PID3/FID 3	Organic	5890	3336A59628	Target 4.14	By equip	1989
CO-5	HP GC-FID 4	Organic	5890	3203A40004	Target 4.14	By equip	1989
CO-6	HP GC FID 5	Organic	5890E	3336A51432	Target 4.14	By equip	1989
CO-7	HP GC-FID 6	Organic	5890	3336A61999	Target 4.14	By equip	1989
CO-8	HP GC-NPD/FID	Organic	5890	3235A45161	Target 4.14	By equip	1989
CO-9	HP GC-TCD/FPD	Organic	5890	3140A39817	Target 4.14	By equip	1989
CO-10	Agilent GC-Dual ECD #1 (Det. 1&2)	Organic	6890 Plus	US00037991	Chemstation C.00.0.0	By equip	2000
CO-11	HP GC-Dual ECD #2 (Det. 1&2)	Organic	5890	3140A39816	Target 4.14	By equip	1989
CO-12	HP GC/MS 1	Organic	5890E	3336A50197	Chemstation B.01.0.0	By equip	1993
CO-13	HP GC/MS 2	Organic	5890E	3336A52565	Chemstation B.01.0.0	By equip	1993
CO-15	Agilent GC/MS 4	Organic	6890N	CN10429072	Chemstation D.01.0.2	By equip, CD	1999
CO-16	Varian Ion Trap – Out of Service	Organic	3400 CX	22505	N/A	N/A	N/A
CO-17	Orion Ion Analyzer	Inorganic	EA940	TX41A	N/A	By equip	1984
CO-18	O.I. TOC Analyzer	Inorganic	700	I408700175	N/A	By equip	1989
CO-19	Varian Flame AA	Inorganic	220	EL97073198	SpectrAA 3.1	By Equip	1997
CO-20	Cetac Mercury Analyzer	Inorganic	M6000A	069801 MAS	Cetac 4.1	By equip	1.2 1997
CO-21	Varian ICP	Inorganic	VISTA AX	EL99103583	Expert 4.1.0	Software	4.1.0
CO-22	CARY-50 UV-VIS	Inorganic	Cary 50 Bio	EL98113374	WINUV 2.0	By equip	1999
CO-24	Milestone Mercury Analyzer	Inorganic	DMA 80	1050029	DMA 3.23	By equip	2000
CO-25	HACH Turbidimeter	Inorganic	2100N	000500006172	N/A	By equip	2000
CO-26	Site Lab UV Fluorometer	Inorganic	UVF-3100	7-1272-CE	N/A	By equip	1.0 2002
CO-27	Waters® HPLC	Industrial	60F	MX5JM0229M	Empower 2.0	By equip	1994
CO-28	HP 1090 – Out of Service	Industrial	1090	2516A00563	N/A	By equip	N/A
CO-29	Fisher-Johns Melting Point	Industrial	Cat# 12-144	30200010	N/A	IR lab	N/A
CO-30	PE DSC 7	Industrial	DSC-7	519N5022401	Piris 4.02	By equip	1994
CO-31	PE TGA 7	Industrial	TGA-7	519N5030201	Piris 4.02	By equip	1994
CO-32	PE DMA7e	Industrial	DMA7e	539N6082807	Piris 4.02	By equip	1994
CO-33	Thermo-Nicolet FTIR (scope)	Industrial	912A0429	680C	Omnic 6.0a	Software	6.0a
CO-34	Thermo-Nicolet FTIR (bench)	Industrial	470	AEP0200731	Omnic 6.0a	Software	6.0a
CO-35	Dr. Steeg Reuter Polarimeter	Industrial	SRG 5314	-----	N/A	-----	N/A
CO-36	Accumet pH Meter – Out of Service	Industrial	925	160	N/A	By equip	N/A
CO-37	HP Series II 1090 HPLC – Out of Service	Industrial	1090	3338A04348	N/A	By equip	N/A
CO-38	Varian ICP-MS	Inorganic	800 Series	EL04043768	Win Mass 2.1	software	2.1
CO-39	Thermo GC/MS 5	Organic	Focus GC/4660	D650466367P	Target 4.14	By equip	2006
CO-40	Leeman Mercury Analyzer	Inorganic	HydraAA	HA-7008	WinHg 1.7	By equip	Rev C 1999
CO-41	Entech TO-15 Precon/Autosampler	Organic	7100A/7032AQ-L	1420/1083	Xcaliber 2.07	By equip	2008
CO-42	Agilent GC/MS 7	Organic	7890A	US83110706	Target 4.14	By equip	2008
CO-43	Accumet pH/Conductivity Meter	Industrial	XL20	XL94006130	N/A	By equip	1/2008
CO-44	Leco Carbon/Sulfur Analyzer	Organic	CS 230 SH CE	4195	2.13	By equip	2009
CO-45	Dionex	Industrial	ICS-2100	10030406	Chromeleon 6.8	software	6.8

EQUIPMENT INFORMATION

New #	Manufacturer and Instrument Name	Location	Model #	Serial #	Software Version	Operating Manual Location	Manual Version
CP-1	Olympus PCM	IH	BH-2 (BHT)	238874		Equip file	
CP-2	Olympus PLM	PLM Lab	BH-2 (BHSP)	201447		Equip file	
CP-3	Zeiss Stereo Microscope	PLM Lab	SV8	P40355		Equip file	
CP-4	Olympus PCM	IH	CH-2 (CHS)	OE0113		Equip file	
CP-5	Olympus PCM	PCM Lab	BX40	4H06849		By equip	
CP-6	Olympus PCM	IH	CH30	8F12499		Equip file	
CP-7	Olympus PCM	IH	BH2	706007		Equip file	
CP-8	Olympus PCM	IH	CX40	6J13360		Equip file	
CP-9	Ohaus Top Load Balance	Organic	CT-200-S	14916		QA Dept.	
CP-10	Sartorius Analytical Balance	PLM Lab	RC 210 D	10405995		By equip	
CP-11	Ohaus Triple Beam Balance	Industrial	700/800	----------		-----	
CP-12	Ohaus Top Load Balance	Inorganic	CT200	CD05067		QA Dept.	
CP-13	Ohaus Top Load Balance	Log-in	CT200	CD05068		QA Dept.	
CP-14	AND Analytical Balance	Inorganic	HM - 300	13506172		By equip	
CP-15	AND Analytical Balance	Industrial	HM - 200	13506078		By equip	
CP-16	Ohaus Top Load Balance	Extraction	Adventurer	H188 1203090461 P		By equip	
CP-17	Fisher Isotemp Drying Oven	Log-in	625G	108N0165		By equip	
CP-18	Fisher Drying Oven	Inorganic	516-G	106N0214		Dept. Man.	
CP-19	Blue M Drying Oven	Inorganic	SW-17TA-1	SW-1848		Not available	
CP-20	Lab-Line Drying Oven	Industrial	3510	0891-0541		IR lab	
CP-22	VWR Oven	Industrial	1310	0400391		IR lab	
CP-23	Equatherm Vacuum Oven	Industrial	299-751	295-0023		IR lab	
CP-24	Thermolyne Muffle Oven	Inorganic	114300	1206030615824		By equip	
CP-25	Thermolyne Muffle Furnace	Extraction	F30420C	1262030932011		By equip	
CP-29	Block Digester - ICP	Inorganic	SC154	2484VAR1327		By equip	
CP-30	Block Digester - ICP	Inorganic	SC154	4298CEC2068		By equip	
CP-31	Olympus PLM	PLM Lab	BH-2	OH8294		Equip file	
CP-32	Bioscience COD Reactor	Inorganic	163-466T	COD-T348		Dept. Man.	
CP-33	Fisher Scientific Digital Timer	Industrial	14-649-15	72572201		QA/QC office	
CP-34	Fisher Scientific Digital Timer	Industrial	14-649-15	72572185		QA/QC office	
CP-35	Blue M Drying Oven	Industrial	OV-490A-s	OV3		By equip	
CP-36	Scout Pro Balance	Log-in	SP202	7126351126		By equip	
CP-37	Sartorius Analytical Balance	Organic	CPA 1245 S	23550719		By equip	
CP-38	Metrohm Autotitrator	Extraction	18550010	1855001003258		Software	
CP-39	Baxter Drying Oven	Industrial	DX-41	200003		By equip	
R-1	Olympus Microtome	Industrial	CUT4055	550539		By equip	
R-2	Dionex ASE 200 #1	Extraction	200	00120383		By equip	
R-3	Dionex ASE 200 #2	Extraction	200	00120381		By equip	
R-4	Pensky-Martens Flashpoint	Inorganic	K-16200	5021		Dept. Man.	
R-6	Barnstead E-Pure DI System	Inorganic	D4641	10900031044121		Inorg. DM	
R-8	TurboVap II Evaporator	Extraction	TurboVap II	TV9425N4108		By equip	
R-10	Carver Hydraulic Press	Industrial	C	36000-310		By IR	
R-11	Haake Chiller	Industrial	K20	194017764004		IR lab	
R-13	Lab-Line Shaker Bath	Organic	3540	01970088		Not available	
R-14	Branson Sonicator	Extraction	8210	8210R-MT		By equip	
R-15	Labconco Rotary Evaporator	Industrial	421-1655	119109013		By equip	
R-16	Equatherm Orbital Shaker	Extraction	3508	0196-0067		By equip	

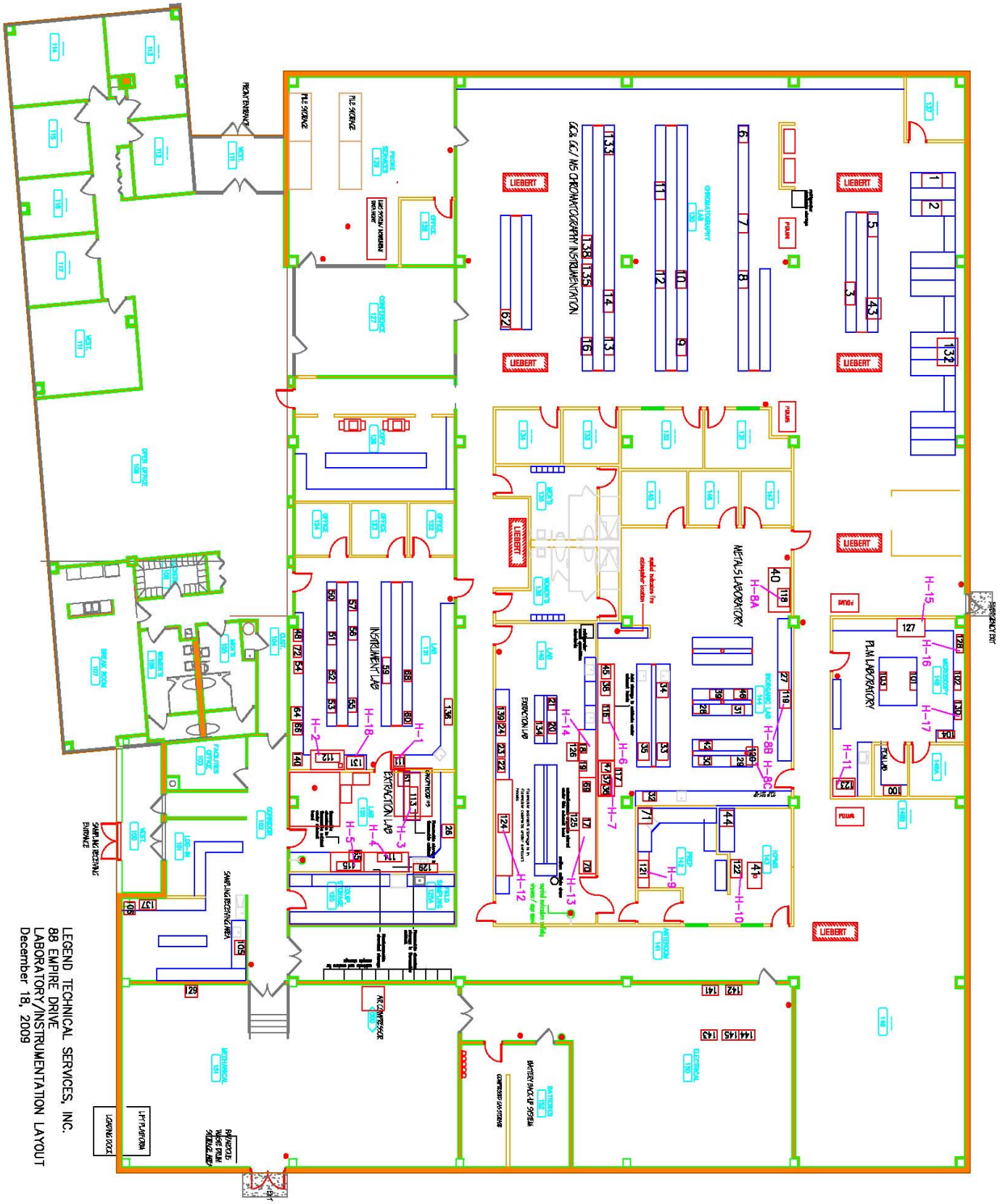
EQUIPMENT INFORMATION

New #	Manufacturer and Instrument Name	Location	Model #	Serial #	Software Version	Operating Manual Location	Manual Version
R-17	Thomas-Wiley Mill	Industrial	3383-L10	-----		By equip	
R-18	Fisher Mini Vortexer	Industrial	128101	2159		By equip	
R-19	Equatherm Ultrasonic Cleaner	Industrial	9313	695-0004		IR lab	
R-20	Leica Stereoscope	Industrial	Wild M3Z	059		-----	
R-21	Rheology International Viscometer	Industrial	RI:1:H_1	6092		Industrial	
R-22	Boekel Steam Bath	Industrial	1494	042403121		Industrial	
R-23	ThermoNeslab Chiller	Industrial	M25	10224902		By equip	
R-24	TurboVap II Evaporator	Extraction	TurboVap II	TV9717N7461		By equip	
R-25	Steambath #1	Extraction	-----	-----		-----	
R-26	Steambath #2	Extraction	-----	-----		-----	
R-27	Barnstead Nano Pure DI System	Inorganic	D11931	1193031256882		Inorganic	
R-28	Organomation N-EVAP	Extraction	12185	15322		-----	
R-29	Fisher Scientific Water Bath	Industrial	228	904N0084		-----	
R-30	Labnet Centrifuge	Industrial	Z 300	47980075		By equip	
R-31	US Filter DI System	Industrial				By equip	
R-32	Bico WD Jaw Crusher	Mechanical	241-36	72047		By equip	
R-33	Bico Pulverizer	Mechanical	242-53X3	72048		By equip	
R-34	Gilson Sample Splitter	Mechanical	SP-1	-----		-----	
R-35	Gilson Mini-Splitter	Mechanical	SP-3	-----		-----	
R-36	Meinzer II Sieve Shaker	Mechanical	II	-----		-----	
H-1	Dedicated Ashing Hood	Industrial	-----	-----		-----	
H-2	Labconco Chemical Fume Hood	Industrial	7280100	050739918G		CHP	
H-3	Dedicated Soxlet Extractor Hood	Industrial	-----	-----		-----	
H-4	Labconco Chemical Fume Hood	Industrial	9683000	050739922K		CHP	
H-5	Labconco Chemical Fume Hood	Industrial	7280100	050739919G		CHP	
H-6	Labconco Chemical Fume Hood	Inorganic	9700400	050739911F		CHP	
H-7	Labconco Open Hood	Inorganic	-----	-----		-----	
H-8A	Vista ICP-AES Exhaust Hood	Inorganic	-----	-----		-----	
H-8B	Varian Spectra-AA Exhaust Hood	Inorganic	-----	-----		-----	
H-8C	Cetac M6000 Exhaust Hood	Inorganic	-----	-----		-----	
H-9	Labconco Chemical Fume Hood	Inorganic	7280100	050739920G		CHP	
H-10	ICP-MS Exhaust Hood	Inorganic	-----	-----		-----	
H-11	Classic Modular Systems, Inc. Fume Hood	PLM Lab	-----	-----		-----	
H-12	Labconco Chemical Fume Hood	Extraction	9700400	050739940F		CHP	
H-13	Labconco Chemical Fume Hood	Extraction	9700400	050739921F		CHP	
H-14	Labconco Chemical Fume Hood	Extraction	9683000	050739923K		CHP	
H-15	NuAire	PLM Lab	NU-425-400	23946WY		-----	
H-16	PLM Macroscope Hood	PLM Lab	-----	-----		-----	
H-17	McCronePLM Sample Prep Hood	PLM Lab	-----	6950593		-----	
H-18	AirClean 600	Industrial	AC632LFUVC	AC632-LFUVC2437		By equip	

CHP=Chemical Hygiene Plan

THERMOMETER INFORMATION

Number	Location	Serial Number	Range	Increments
#3	Investigative Chemistry	No unique ID #	-2.5 to 101.5 C	0.5
#9	QA Dept.	K96-516	20 to 230 F	1.0
#13	QA Dept.	No unique ID #	0 - 40 C	0.2
#17	QA Dept.	No unique ID #	0 to 300 F	2
#22	QA Dept.	No unique ID #	-20 to 50 C	1.0
#26	Flashpoint – Inorganic Lab	758PG	20 to 230 F	1.0
#27	QA Dept.	6967	-5 to 15 C	0.5
#28	QA Dept.	360724	-5 to 5 & 20 to 130 C	1.0
#29	QA Dept.	8882	-5 to 15 C	0.5
#33	Frigidaire Freezer (white)	9052	-30 to 0 C	0.5
#34	Freezer – Extraction Lab Standards	10477	-30 to 0 C	0.5
#35	Freezer – Investigative Chemistry	10521	-30 to 0 C	0.5
#36	QA Dept.	12565	-5 to 15 C	0.5
#38	QA Dept.	5485	-5 to 15 C	0.5
#39	Refrigerator – Investigative Chemistry	10811	-5 to 15 C	0.5
#41	SVOC Cooler	11079	-5 to 15 C	0.5
#45	Refrigerator – Inorganic Lab	11737	-5 to 15 C	0.5
#46	Volatiles Cooler	12127	-5 to 15 C	0.5
#47	Log-in Cooler	13176	-5 to 15 C	0.5
#48	SVOC Cooler	13338	-5 to 15 C	0.5
#53	Investigative Chemistry (thermocouple)	240117969/240173926	-40 to 1200 C	1
#54	QA Dept.	240222209	-50 to 250 C	0.1
#55	Mobile Lab Oven – Inorganic Lab	14-983-17D	-10 to 260 C	1.0
#56	Blue M Oven – Inorganic Lab	13-246	30 to 230 C	1.0
#61	Log-in Cooler	T 7589	-5 to 15 C	0.5
#64	QA Dept.	1065	-5 to 15 C	0.5
#65	QA Dept.	T 63969	15 to 50 C	0.5
#66	PLM Lab	T 42390	15 to 50 C	0.5
#67	pH Meter – Inorganic Lab	F 48863	15 to 50 C	0.5
#68	QA Dept.	6269	20 to 50 C	0.5
#69	Investigative Chemistry	307059	0 to 100 C	1.0
#73	QA Dept.	No unique ID #	-30 to 150 C	1.0
#75	QA Dept.	4473	80 to 130 C	0.5
#77	Steambath – Extraction Lab	HB 177484	20 to 130 C	1.0
#78	QA Dept.	T 8777	15 to 50 C	0.5
#79	QA Dept.	T 8445	15 to 50 C	0.5
#80	TCLP Tumbler – Inorganic Lab	T 42473	15 to 50 C	0.5
#81	QA Dept.	No unique ID #	-10 to 60 C	1.0
#82	Moisture Oven – Log-in	No unique ID #	35 to 230 C	1.0
#86	Freezer – Extraction Lab Extracts	5937	-30 to 0 C	0.5
#87	Inorganic Lab – COD Reactor	No unique ID #	90 to 160 C	1.0
#89	Nitrogen Blowdown – Extraction Lab	No unique ID #	0 to 100 C	1
#90	Inv. Chemistry - Oven	2517	0 to 100 C	1.0
#91	QA Dept.	2511	0 to 100 C	1.0
#92	QA Dept.	2500	0 to 100 C	1.0
#93	QA Dept.	2510	0 to 100 C	1.0
#94	Inv. Chemistry – Freezer	9348	-30 to 0 C	0.5
#95	Extraction Lab – Standards Freezer	9206	-30 to 0 C	0.5
#96	VOC Lab – Small Freezer (Kenmore)	9296	-30 to 0 C	0.5
#97	Log-in	90891429	-50 to 250 C	0.1
#98	Inv. Chemistry – Standards Refrigerator	11690	-5 to 15 C	0.5


LEGEND TECHNICAL SERVICES, INC.
88 EMPIRE DRIVE
LABORATORY/INSTRUMENTATION LAYOUT
December 18, 2009
LIEBERT
INSTRUMENT LAB
EXTRACTION LAB
METALS LABORATORY
PLM LABORATORY
GCs/ GC/ MS CHROMATOGRAPHY INSTRUMENTATION
SAMPLING RECEIVING
SAMPLING RECEIVING AREA
BATTERY BACK-UP SYSTEM
AIR COMPRESSOR

Floor Plan Key

Floor Plan #	LTS #	Manufacturer and Instrument Name
1	CO-3	HP GC-PID 3/FID 3
2	CO-2	HP GC-PID 2/FID 2
3	CO-13	HP GC/MS 2
5	CO-1	HP GC-PID 1/FID 1
6	CO-16	Varian Ion Trap
7	CO-12	HP GC/MS 1
8	CO-15	Agilent GC/MS 4
9	CO-5	HP GC-FID 4
10	CO-7	HP GC-FID 6
11	CO-10	Agilent GC-Dual ECD #1
12	CO-11	HP GC-Dual ECD #2
13	CO-6	HP GC-FID 5
14	CO-8	HP GC-NPD/FID
16	CO-9	HP GC-TCD/FPD
17	R-28	Organomation N-Evap
18	R-8	TurboVap II Evaporator
19	R-24	TurboVap II Evaporator
20	R-3	Dionex ASE 200 #2
21	R-2	Dionex ASE 200 #1
22	R-14	Branson Sonicator
23	R-16	Equatherm Orbital Shaker
24	CP-25	Thermolyne Muffle Furnace
25	R-11	Haake Chiller
26	R-15	Labconco Rotary Evaporator
27	CO-19	Varian Flame AA
28	CO-18	O.I. TOC Analyzer
29	CO-20	Cetac Mercury Analyzer
30	CO-22	CARY-50 UV-VIS
31	CO-24	Miestone Mercury Analyzer
32	R-6	Barnstead E-Pure DI System
33	CP-14	AND Analytical Balance
34	CO-17	Orion Ion Analyzer
35	CP-12	Ohaus Top Load Blanace
36	CP-24	Thermolyne Muffle Furnace
37	R-4	Pensky-Martens Flashpoint
38	CP-29	Varian Block Digester
39	CP-19	Blue M Drying Oven
40	CO-21	Varian ICP
41	CO-38	Varian ICP-MS
42	CO-40	Leeman Mercury Analyzer
43	CO-39	Thermo GC/MS 5
44		Leeman Low-level Mercury Analyzer
45	CP-30	Block Digester - ICP

Floor Plan #	LTS #	Manufacturer and Instrument Name
46	CP-18	Fisher Drying Oven
47	CP-32	Bioscience COD Reactor
48	R-29	Fisher Scientific Water Bath
50	CO-30	PE DSC 7
51	CO-31	PE TGA 7
52	CO-32	PE DMA7e
53	CP-15	AND Analytical Balance
54	R-17	Thomas-Wiley Mill
55	CO-33/CO-34	Thermo-Nicolet FTIR (scope/bench)
56	R-20	Leica Stereoscope
57	R-10	Carver Hydraulic Press
58	CP-7	Olympus PCM
59	CO-27	Waters® HPLC
60	CO-28	HP 1090
61	R-23	ThermoNeslab Chiller
62	R-13	Lab-Line Shaker Bath
64	CP-23	Equatherm Vacuum Oven
65	R-22	Boekel Steam Bath
66	CP-20	Lab-Line Drying Oven
68	CO-37	HP Series II 1090 HPLC
69	R-25	Steambath # 1
70	R-26	Steambath # 2
71	R-27	Barnstead Nano Pure DI System
72	R-30	Labnet Centrifuge
100	CP-5	Olympus PCM
101	CP-2	Olympus PCM
102	CP-3	Zeiss Stereo Microscope
103	CP-31	Olympus PLM
104	CP-10	Sartorius Analytical Balance
105	CP-13	Ohaus Top Load Balance
106	CP-17	Fisher Isotemp Drying Oven
111	H-1	Dedicated Ashing Hood
112	H-2	Labconco Chemical Fume Hood
113	H-3	Dedicated Soxlet Extractor Hood
114	H-4	Labconco Chemical Fume Hood
115	H-5	Labconco Chemical Fume Hood
116	H-6	Labconco Chemical Fume Hood
117	H-7	Labconco Open Hood
118	H-8A	Vista ICP-AES Exhaust Hood
119	H-8B	Varian Spectra-AA Exhaust Hood
120	H-8C	Cetac M6000A Exhaust Hood
121	H-9	Labconco Chemical Fume Hume
122	H-10	ICP-MS Exhaust Hood
123	H-11	Classic Modular Systems, Inc. Fume Hood

Floor Plan #	LTS #	Manufacturer and Instrument Name
124	H-12	Labconco Chemical Fume Hood
125	H-13	Labconco Chemical Fume Hood
126	H-14	Labconco Chemical Fume Hood
127	H-15	NuAire
128	H-16	PLM Macroscope Hood
129	R-31	US Filter DI System
130	H-17	PLM Sample Prep Hood
131	H-18	AirClean 600
132	CO-41	Thermo GC/MS 6/Entech TO-15 Precon/Autosampler
133	CO-42	Agilent GC/MS 7
134	CO-43	pH/Conductivity Meter
135	CO-44	Leco Carbon.Sulfur Analyzer
136	CP-35	Blue M Drying Oven
137	CP-36	Scout Pro Balance
138	CP-37	Sartorius Analytical Balance
139	CP-38	Metrohm Autotitrator
140	CP-39	Baxter Drying Oven
141	R-32	Bico WD Jaw Crusher
142	R-33	Bico Pulverizer
143	R-34	Silson Sample Splitter
144	R-35	Glison Mini-Splitter
145	R-36	Meinzer II Sieve Shaker

6.4 Work Environment

LEGEND has a safe work environment to support the division's needs to achieve conformity to service and product requirements.

A. Laboratory accommodation, test areas, energy sources, lighting, heating, and ventilation shall be appropriate to allow proper performance of tests.

B. The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement.

C. The laboratory shall provide facilities for the effective monitoring, control, and recording of environmental conditions as appropriate.

D. There shall be effective separation between neighboring areas where the activities therein are incompatible.

E. Health and safety programs are listed below. Program documentation, requirements, and designated administrators are included with each program.

1. Medical Surveillance Program: defines medical monitoring requirements
2. Chemical Hygiene Program: defines general laboratory health and safety
3. Respiratory Protection Program: defines when respirators are used and the medical monitoring and fit testing requirements
4. Personnel Exposure Monitoring Program: defines air and wipe sampling to be performed in the lab to assess employee exposures
5. Waste Contingency Plan: defines waste generation, handling, and disposal
6. Bloodborne Pathogens Program: defines special requirements for clean-up and disposal of body fluids during first aid procedures

SECTION 7

PRODUCT REALIZATION

7 Product Realization

7.1 Planning of Product Realization

This process identifies action needed to plan product realization, including:

A. Quality objectives and requirements for the product

B. The need to establish processes, documents, and provide resources specific to the product

C. Required verification, validation, monitoring, inspection, and test activities specific to the product and the criteria for product/service acceptance

D. Records needed to provide evidence that the realization processes and resulting product/service meet requirements

7.2 Customer Related Processes

7.2.1 Determination of Requirements Related to the Product/Service

The testing and analyzing process includes activities that determine the following:

A. Requirements specified by the customer, including the requirements for delivery and post-delivery activities

B. Requirements not stated by the customer but necessary for specified or intended use, where known

C. Statutory and regulatory requirements related to the product

Once received and logged in, the samples are delivered to the appropriate department. The client manager reviews the analytical request. If a method has not been specified by the client, a person with the appropriate technical expertise will select the most current method that has been published in international or national standards, published by reputable technical organizations or in relevant scientific texts or journals.

Employing methods that have not been established, as standard, shall only be done after receiving client approval. The method must be fully documented and available to the client. In the case of a method involving proprietary information a confidentiality agreement must be signed in order to release the method to the client.

7.2.2 Review of Requirements Related to the Product/Service

For new orders that require testing and analysis, a process is used to ensure that product/service requirements are defined, differing order or contract requirements are resolved, and the organization has the ability to meet the defined requirements before committing to supply the product to the customer.

Records of the results of the reviews and actions arising from them are maintained (e.g. work orders and proposals within project files).

Project personnel shall be notified immediately of any discrepancies, and the samples segregated and held until the problem is resolved. The laboratory shall not be responsible for meeting holding times on these samples. A Corrective Action Report may

be used to document actions taken to resolve problems with incoming samples.

7.2.3 Customer Communication

LEGEND's sales and client managers are responsible for direct communication with the customer relating to:

A. Product information

B. Inquiries, contracts or order handling, including amendments

C. Customer feedback

1. Feedback is generally solicited via person to person communication either by the Client Manager, Marketing Director, or by the QA/QC Coordinator. Client Surveys are also sometimes used to get feedback.

2. Unsolicited feedback is filed in the QA Department

3. Customer complaints are processed through LEGEND's complaint process in the SOP 'Client Complaint Resolution'.

7.3 Design and Development – Excluded from LEGEND's ISO 9001:2008 Certification

7.4 Purchasing

7.4.1 Purchasing Process

The specifications and control methods for purchased materials are determined as part of the procurement process for specific products/services. Vendors are selected and managed by the process described in the SOP 'Purchasing and Receiving Procedure'.

7.4.2 Purchasing Information

Appropriate purchasing information is specified in raw material specifications, component drawings, and purchase orders delivered to the vendor. Appropriate technical and purchasing personnel verify the adequacy of the information before it is communicated to the vendor. LEGEND personnel procure externally purchased materials and services.

7.4.3 Verification of Purchased Product

The requesting individual verifies product purchased per SOP 'Purchasing and Receiving Procedure'.

7.5 Production and Service Provision

7.5.1 Control of Production and Service Provisions

The product design and development, testing and analysis, and related activities for LEGEND are carried out under controlled conditions (i.e. internal audits and reviews).

Suitable monitoring and measuring equipment is used and controlled as appropriate.

7.5.2 Validation of Processes for Production and Service Provision

A. LEGEND validates each piece of testing equipment prior to installation. The review of the processes may lead to:

1. Redefining for criteria for review and approval of the process

2. Providing approval of equipment and qualification of personnel

3. Assuring the use of specific new methods and procedures

4. Reassessment requirements for records keeping accessing

5. Revalidation or amendment of certain requirements

B. Data may be manually computed, input into a computer for processing or calculation, or directly acquired from a computer.

1. If the analyst manually processes data, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. These shall be performed on the data sheet or on a LEGEND form that shall be initialed and dated by the analyst and attached to the data sheets.

2. For data entered and processed in a computer, the analyst shall indicate on a copy of the input the sample(s) or project number, sign and date the copy, and attach it to the data sheets.

3. Where computers or automated equipment are used for capture, processing, recording, reporting, storage or retrieval of test data, all applicable requirements of the software will be complied with, the computer software is documented and adequate for use, and all computer and automated equipment is maintained to ensure proper functioning and provided with environmental and operating conditions necessary to maintain the integrity of test data.

4. For data acquired directly from the computer, the analyst shall verify that all parameters (project/sample numbers, response factors, units, detection limits, etc.) are correct. The analyst shall sign and date the output.

C. Review of Data Processing

1. One hundred (100%) percent of all data shall be reviewed by a second analyst, Department Manager, or his/her designee. Reviewed data shall be initialed and dated by the technical reviewer. For certain tests (e.g. FTIR analysis), data review is indicated by signing the final report.

2. If the reviewer disagrees with a number or qualifier, the reviewer shall bring it to the attention of the analyst. Upon agreement between the analyst and the reviewer, the data will be revised by crossing out the incorrect number/qualifier with a single line and recording the revised number/qualifier next to it. All changes need to be initialed and dated. If there is a question on whether a change should be made, the department manager will make the final decision. If the reviewer believes it is a systematic error vs. an isolated error, a Corrective Action Report (CAR) should be initiated.

D. Review of Laboratory Reports

1. The Client Manager shall review all laboratory reports. Then, the laboratory report shall be reviewed and signed by a report reviewer.

2. If the reviewer finds an error, it is noted and given back to the Client Manager for correction. It is at the Client Manager's discretion if the change is made. If the reviewer believes it is a systematic error vs. an isolated error, a Corrective Action Report (CAR) should be initiated.

E. Verification of Software

Commercial Computer software (ChemStation, Target) shall be verified by running sample calculations and comparing the values to hand calculations. Only one example calculation need be verified for an individual data acquisition system. Commercial data systems are verified when the system is upgraded (or downgraded) to a new version of the software. The analyst shall document this procedure by signing and dating both the computer output and the hand calculations. The data and work sheets will be archived in the QA/QC department for future reference.

Where forms developed in-house are used to generate data (using Excel spreadsheets with equations), the calculations are verified by running sample calculations and comparing the values to hand calculations. Equations shall be verified on an annual basis or whenever modified. If an uncontrolled spreadsheet is used to assist an analyst then it is considered the same as a hand calculation and all results for that spreadsheet must be peer reviewed including a review of the calculation.

F. Laboratory Data System Archival

1. The results directory of the central computer system is backed up onto computer tapes every month.

2. The method files are archived every month with the result files. The tapes are archived for a minimum of two years.

3. The backed up result and method files are deleted off the central computer after the back up.

4. The LIMS is backed up and archived every month.

G. Data Reports

1. The format and content of the laboratory data report will vary depending on project or client needs, contract and regulatory requirements, and the need for explanatory text.

2. Each page shall list client, project number (if applicable), and field identification. Data shall be presented in a tabular format whenever possible.

3. Data listed on the report shall include parameters analyzed, reported values,

reporting limit (RL), and units of measurement.

4. Results less than the RL shall be indicated by a "less than" sign (<) or appropriate qualifiers. Qualifiers may also include standard Contract Laboratory Program (CLP) “flags” on actual data values or use of footnotes on data tables to qualify or clarify results. If necessary, case narrative text shall be included in the report or in a separate letter of transmittal.

5. All reports shall be signed by two personnel, usually the client manager and primary chemist, QA/QC Coordinator, or Technical Director. Any analytical results communicated verbally shall be considered preliminary until data are sent in a final report.

6. Results that are transmitted electronically must be accompanied with a confidentiality statement.

H. Proficiency Testing Programs

Specific LEGEND laboratory sections participate in formal inter-laboratory proficiency testing programs (American Industrial Hygiene Association (AIHA) Proficiency Testing Program, state certifications, etc.) depending on their particular area of analysis and the individual program's scope. The QA/QC Coordinator reviews performance as information is received from the specific program agency.

Any deviations from acceptable performance are documented on a Corrective Action Report and a corrective plan of action immediately implemented. Subsequent performance is monitored to assure corrective plan has resulted in acceptable performance.

I. Blind Standard Samples

For AIHA fields of testing not covered by PT programs, a minimum of four independently prepared blind spikes at varying levels are analyzed twice annually by trained personnel. Acceptance criteria are calculated from 20 QC data points as the average ± 3s, and are applied to the blind spike recoveries. The QA/QC Coordinator reviews performance after the results from the blind spikes are technically reviewed. Any deviations from acceptable performance are documented on a Corrective Action Report and a corrective plan of action immediately implemented. Subsequent performance is monitored to assure corrective plan has resulted in acceptable performance.

J. Exchange Of Samples With Other Laboratories

Periodically samples are exchanged with one or more laboratories and the results compiled as a comparison. This is done both formally through the use of inter-laboratory Round Robin programs and informally through the submission of previously analyzed samples to another laboratory for comparison.

K. Certification Programs

LEGEND participates in laboratory accreditation/certification programs such as the American Industrial Hygiene Association (AIHA) accreditation program, the National Voluntary Laboratory Accreditation Program (NVLAP), American Association for

Laboratory Accreditation (A2LA), and various state certification programs.

These programs require a periodic performance of laboratory analytical testing and review (external audit) of laboratory facilities and personnel.

Current certificates are found in the QA Department.

L. External Audits

The QA/QC Coordinator is responsible for coordinating outside agency audits of the laboratory facility. Manufacturers and agencies regularly audit LEGEND. Specific items to be addressed are reviewed by the QA/QC Coordinator including schedule, audit agenda, specific personnel to be involved in the audit. These items are then communicated to the appropriate personnel. Client confidentiality is taken into consideration when producing specific items during the audit. The QC Coordinator prepares a follow-up report after receiving the results of the audit that summarizes the findings and any corrective actions needed. Results of these audits are available for review.

7.5.3 Identification and Traceability

The process for identification and traceability from order entry to the end of the process for products/service is in place.

A. Sample Receipt and Log-In

1. Samples shall be logged in as soon as possible after receipt. If samples arrive during non-business hours, they shall be held in a cooler at 4 ± 2 °C (where applicable) and logged in the next business day.

2. Follow the protocol outlined in LEGEND SOP ‘Sample Receiving, Handling, Log-In, Storage Control, and Holding Times’.

3. Where there is any doubt as to the item’s suitability for testing, where the item does not conform to the description provided, or where the test required is not fully specified, the client shall be contacted for further instructions before proceeding.

B. The Department Managers and/or Technical Director shall be responsible for prioritizing work to assure that holding times and project commitments are met.

7.5.4 Customer Property

Care is exercised with all customer property while it is under LEGEND’s control. Customer property is identified, verified, and safeguarded for use in the output of LEGEND’s operations. Items found to be unsuitable for use or that are lost or damaged are reported to the customer and records are maintained. Typical customer property would be samples for analysis, raw data of the analysis and a final report. All information received and released to clients is strictly confidential. No information may be released to another party without the written approval of the client.

7.5.5 Preservation of Product

Client samples and data collected in the analysis are stored.

A. Client Samples

1. Care is taken in preservation that includes identification, handling, storage, and protection (see SOP 'Sample Receiving, Handling, Log-in, Storage Control, and Holding Times').

2. Analytical samples shall be stored for at least 30 days after submittal of the laboratory report to the client before disposal.

3. It is at the lab's discretion to return samples to the client. (Examples: samples that have been defined as hazardous waste or samples with high levels of oil.) Client managers arrange for the return of these samples.

4. Samples are stored in the laboratory and are accessible by LEGEND employees only. Visitors in the laboratory are required to sign in and wear a 'Visitor Badge'. Access is limited to the area of concern.

5. Samples may be completely consumed during analysis, returned to the client or sampling location, stored under required environmental conditions (if re-analysis is anticipated) or under ambient conditions (if re-analysis is not likely), or disposed by the laboratory. Samples and extracts shall usually be disposed in thirty days unless otherwise specified. Disposition is indicated in the report.

6. Department Managers and/or the Hazardous Waste Coordinator shall determine the method and time for disposal if not specified by the client manager.

7. Some waste may be disposed of in a sanitary sewer as permitted by 40 CFR 261.3(a)(2)(iv). Some samples may be hazardous because of their general characteristics or because they are listed in 40 CFR Part 261. Shipping of these materials is addressed in 40 CFR 172.02, 172.03, 172.04, 172.300, and 172.400.

8. Laboratory waste disposal is addressed in the Waste Contingency and Emergency Response Plan.

B. Data Storage

Data storage is addressed in Section 4.2.4 'Control of Records'.

7.6 Control of Monitoring and Measuring Devices

A. Various monitoring and measuring devices that are utilized in the testing and analysis of samples are calibrated according to calibration procedures.

B. Development and implementation of calibration procedures shall be the responsibility of the Department Managers. At a minimum, each manager must address the following key points for the applicable instruments in their areas of responsibility.

1. Recognized procedures (USEPA, ASTM, NIOSH, manufacturer's instructions etc.) shall be used when available. Written calibration procedures may include the reference materials to be used, calibration technique, acceptable performance limits, frequency, and documentation.

2. Calibration frequency shall be determined by manufacturer's recommendations, agency requirements, equipment type, instrument stability, method requirements and prior experience.

3. Physical standards (weights, certified thermometers) shall be traceable to nationally recognized standards (e.g. NIST). Chemical reference standards shall be those provided by the National Institute of Standards and Technology, Standard Reference Materials (SRMs) and/or vendor certified materials traceable to these standards. Where this is not possible, the laboratory should provide satisfactory evidence of correlation of results. They shall be used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards has not been invalidated.

4. Operationally calibrated equipment that fails calibration shall be removed from service or tagged to indicate that it is out of calibration. The equipment shall be repaired and re-calibrated before re-use. A record of all such occurrences shall be maintained with the instrument maintenance and calibration log and/or instrument run logs.

5. If a periodically calibrated piece of equipment is found to be out of tolerance by either the calibration vendor or LEGEND personnel, the calibration results will be reviewed by the QA/QC Coordinator and the impact on the affected data will be assessed and documented. Corrective action will be taken by the appropriate laboratory personnel as required. The piece of equipment is tagged as “out of service” until such time that it has been repaired and re-calibrated.

C. Calibration is required to demonstrate that instruments are operating properly and to ensure traceability of measurements. Calibration records shall be maintained for each piece of equipment that requires calibration. The analyst will eliminate, or minimize, the source of errors by proper selection of method, equipment, solvents and/or gases. There are two types of calibrations: periodic and operational.

1. Periodic calibrations are performed at prescribed intervals. Balances, weights, microscopes, and thermometers are calibrated annually. Balances are checked, prior to each day of use, with weights bracketing the mass being measured.

2. Operational calibrations are performed or verified prior to instrument usage. Specific calibration requirements are contained in the SOPs applicable to each instrument or analytical method. Examples of equipment using operational calibration are: gas chromatography, mass spectrometry, and atomic spectroscopy. Examples of operational calibrations are calibration curves, calibration verification standards, and flow checks.

3. If calibration curve information is not listed, the following guidelines should be used:

   a) A standard calibration curve should be prepared from the preparation and analysis of at least three standard solutions (including a blank or zero concentration standard as one of the standards if appropriate), unless the

method dictates differently (e.g. ICP-AES), by mixing the species to be analyzed with the appropriate diluent that is used to introduce the species into the instrument. The concentrations of the standard solutions should cover the working range of the instrument (unless the method specifies otherwise) and sample measurements should be made within this range.

b) The calibration curve should be prepared by plotting instrument response versus concentration of the species analyzed so that sample concentrations can be determined.

c) If an average response factor calibration is used, the Relative Standard Deviation (%RSD) must be $\leq$ 20%. If the %RSD is greater than 20%, a linear calibration curve is constructed.

d) The correlation coefficient (r) of a first order, or linear calibration curve, must be 0.99 or greater.

e) Some analytes do not respond linearly and will require a different curve fit such as a second order or quadratic fit. In some cases instrumentation may use a curve fit other than any mentioned above. To evaluate curve algorithms other than linear or average response factor use either r^2, the weighted coefficient of determination (COD), or an alternative method of curve verification. One option is to evaluate the residual values at each point along the curve. In these cases document in the SOP how the curve is to be evaluated and the acceptance criteria.

f) The use of forced zero curves or weighted curve fits is acceptable to best represent the relationship between signal and concentration. The use of these techniques is unacceptable under certain programs or methods. It is important that program or method guidelines are followed at all times.

g) Records of calibrations should be kept in a logbook or LIMS sequence with each instrument. This logbook, maintained by the analyst, should contain the instrument name and number, manufacturer, model #, serial #, location, and a brief record of all calibrations and samples analyzed.

h) It is critical to verify the calibration curve with at least one second source standard. It is also important to check the curve with periodic checks during the analysis as well as a final check at the end. In some cases the method may specify otherwise; follow the method guidance in these cases.

D. When an instrument for calibration has been adjusted or repaired, the calibration results before and after adjustment or repair will be made available to the client upon their request.

E. The laboratory will notify clients promptly, in writing, if any event such as the identification of defective measuring or test equipment, casts doubt in the validity of results given in any test report or amendment to a report.

F. In the event that the laboratory needs to obtain equipment on a temporary basis, the equipment will be calibrated to ensure all relevant requirements are met.

G. When, for whatever reason, equipment goes outside the direct control of the laboratory,

the laboratory should ensure that the functions and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

H. New equipment is validated prior to use within the laboratory. The form 'Initial Qualification of New Equipment' is filled out by the operator, reviewed, and filed with the QA department.

SECTION 8

MEASUREMENT, ANALYSIS, AND IMPROVEMENT

8 Measurement, Analysis, and Improvement

8.1 General

LEGEND monitors, measures, analyzes, and implements improvements to the processes and quality management system.

8.2 Monitoring and Measurement

8.2.1 Customer Satisfaction

A. On-going measures of customer satisfaction are monitored by periodic surveys of clients and direct client feedback. These records are archived with the QA department.

B. Customer complaints are processed through LEGEND's complaint process in the SOP 'Client Complaint Resolution'.

1. A record shall be maintained when a complaint or any other circumstances arise concerning the laboratory's compliance with policies or procedures. Those areas specific to the complaint will be audited and the corrective action taken will be reported to the individual(s) who raised the doubt and recorded in the complaint log maintained by the QA/QC Coordinator.

2. The QA/QC Coordinator will close the complaint by calling the affected party and asking if they were satisfied with the resolution. If further action is required, the action is recorded on the complaint form and the complaint is given back to the complaint initiator to continue the process. If no further action is required, the complaint is signed and dated by the QA/QC Coordinator.

C. Information acquired from these measures is used as inputs for improving products, processes, and service to the customer.

8.2.2 Internal Audits

The internal audit process used to monitor and maintain the quality management system and analyses are documented in the SOP 'Internal Auditing'. LEGEND's Internal Auditors are responsible for planning and conducting the audits, and for reporting results. Internal audits are archived with the QA department.

8.2.3 Monitoring and Measurement of Processes

The quality management system processes are monitored and measured using various means. Corrective action is taken when planned results are not achieved. They include:

A. Internal and external audits

B. Management reviews

C. Customer inputs

D. Preventive and corrective actions

8.2.4 Monitoring and Measurement of Product

LEGEND's processes specify requirements to be met before release of product/service to the customer.

The following criteria may not be 100% applicable to all applications.

A. Calibration Curves – in most cases analytical measurements are achieved via relative measurement techniques. Known standards are used to define a calibration curve and then unknown samples are measured against that curve. The three main types of curve fit algorithms used in the lab are described below: average response, linear, and polynomial. Exact curve fits employed are defined in method SOPs. The SOP may refer to this section when employing one of the curve fits described below. Other curve fits may be employed to better describe the relationship of standards to response. These may be variations on one of the curve fits below that may include weighting of points or forcing the origin through zero; or, it may include other types of curve fits like rationale or point-to-point. If these alternative approaches are employed the technique is described in the individual method SOP. Factors affecting algorithm selection include technology, instrument software, method criteria, program rules, and analyte behavior.

1. Average Response Factor Calibration

a) Calculate response factors (RFs) for each compound at each level.

$$RF = \frac{A_x}{C_x}$$

Is rearranged to solve for $Area_{Compound}$: $$A_x = (RF)(C_x)$$

Is rearranged to solve for $Conc_{Compound}$: $$C_x = \frac{A_x}{RF}$$

A_x = Area of the characteristic ion for the compound being measured
C_x = Concentration of the compound being measured

b) If internal standards are used, calculate response factors (RFs) for each compound at each level relative to the preceding internal standard.

$$RF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

Is rearranged to solve for $Area_{internal\ Standard}$: $$A_{is} = \frac{(A_x)(C_{is})}{(RF)(C_x)}$$

Is rearranged to solve for $Area_{Compound}$: $$A_x = \frac{(A_{is})(C_x)(RF)}{C_{is}}$$

Is rearranged to solve for $Conc_{nternal\ Standard}$: $$C_{is} = \frac{(RF)(A_{is})(C_x)}{A_x}$$

Is rearranged to solve for $Conc_{Compound}$: $$C_x = \frac{(A_x)(C_{is})}{(A_{is})(RF)}$$

A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard

c) The average response factor, standard deviation, and percent relative standard deviation (%RSD) should be calculated for each compound. The %RSD should be less than the method criteria.

$$\%RSD = \frac{(SD)(100)}{RF_m}$$

Is rearranged to solve for SD: $$SD = \frac{(\%RSD)(RF_m)}{100}$$

Is rearranged to solve for RF_{mean}: $$R_m = \frac{(SD)(100)}{\%RSD}$$

RSD = relative standard deviation
SD = standard deviation of RFs for a compound
RF_m = mean of RFs for a compound

NOTE: 'By response' must be used for Target applications so that the calculation is performed correctly. The graph will depict response on the 'x-axis'.

2. Linear Regression Calibration

a) A regression equation that does not pass through the origin, where the instrument response is treated as the dependent variable (y) and the concentration is treated as the independent variable (x). Results are calculated as follows:

$$y = ax + b_y$$

Is rearranged to solve for concentration: $$x = \frac{(y - b_y)}{a}$$

Is rearranged to solve for 'y' intercept: $$b_y = y - ax$$

Is rearranged to solve for slope of the line: $$a = \frac{(y - b_y)}{x}$$

y = Instrument response (peak area)
a = Slope of the line
b_y = The 'y' intercept
x = Concentration of the calibration standard

b) If internal standards are used, the y-axis is the instrument response divided by the internal standard response and the x-axis is the concentration divided by the internal standard concentration.

$$C_s = \frac{\left[\left(\frac{A_S}{A_{is}}\right) - b_y\right]}{a}(C_{is})$$

Is rearranged to solve for Area$_{\text{target analyte}}$: $$A_s = \left[\left[\frac{(C_s)(a)}{(C_{is})}\right] + b_y\right](A_{is})$$

Is rearranged to solve for Area$_{\text{internal standard}}$: $$A_{is} = \frac{(A_s)}{\left[\left[\frac{(C_s)(a)}{(C_{is})}\right] + b_y\right]}$$

Is rearranged to solve for Conc$_{\text{internal standard}}$: $$C_{is} = \frac{(C_s)(a)}{\left[\left(\frac{A_s}{A_{is}}\right) - b_y\right]}$$

Is rearranged to solve for slope of the line: $$a = \frac{(C_{is})\left[\left(\frac{A_s}{A_{is}}\right) - b_y\right]}{C_s}$$

Is rearranged to solve for 'y' intercept: $$b_y = \left(\frac{A_s}{A_{is}}\right) - \left[\frac{(C_s)(a)}{C_{is}}\right]$$

A_s = Area of the peak for the target analyte
A_{is} = Area of the peak for the specific internal standard
C_s = Concentration of the target analyte
C_{is} = Concentration of the specific internal standard

c) To display the concentration intercept on the x-axis, software may perform the following reciprocal equation:

$$x = \left(\frac{1}{a}\right)(y) + b_x$$

Is rearranged to solve for instrument response: $$y = \frac{(x - b_x)}{\left(\frac{1}{a}\right)}$$

Is rearranged to solve for inverse$_{\text{slope of the line}}$: $$\frac{1}{a} = \frac{(x - b_x)}{y}$$

Is rearranged to solve for 'x' intercept: $$b_x = x - \left(\frac{1}{a}\right)(y)$$

b_x = The 'x' intercept

NOTE: '1/a' is the inverse of the slope as depicted in the Target signal calibration report.

If internal standards are used, the equation becomes:

$$x = \left[\left(\frac{1}{a}\right)\left(\frac{A_s}{A_{is}}\right) + b_x\right](C_{is})$$

Is rearranged to solve for $Conc_{internal\ standard}$: $$C_{is} = \frac{(x)}{\left[\left[\left(\frac{1}{a}\right)\left(\frac{A_s}{A_{is}}\right)\right] + b_x\right]}$$

Is rearranged to solve for $Area_{target\ analyte}$: $$A_s = \frac{\left[\left(\frac{x}{C_{is}}\right) - b_x\right](A_{is})}{\left(\frac{1}{a}\right)}$$

Is rearranged to solve for $Area_{internal\ standard}$: $$A_{is} = \frac{(A_s)\left(\frac{1}{a}\right)}{\left[\left(\frac{x}{C_{is}}\right) - b_x\right]}$$

Is rearranged to solve for $inverse_{slope\ of\ the\ line}$: $$\frac{1}{a} = \frac{\left[\left(\frac{x}{C_{is}}\right) - b_x\right]}{\left(\frac{A_s}{A_{is}}\right)}$$

Is rearranged to solve for 'x' intercept: $$b_x = \left(\frac{x}{C_{is}}\right) - \left[\left(\frac{1}{a}\right)\left(\frac{A_s}{A_{is}}\right)\right]$$

NOTE: 'By Amount' must be used for Target applications so that concentration is on the 'x-axis'. Target will perform the reciprocal equation above to display the 'x-axis' intercept.

3. Non-linear (Polynomial) Calibration

   a) Quadratic (second order) calibrations are only used when historical information or analyst experience shows that the instrument response does not follow a linear model. It is not used for those compounds that have previously shown to exhibit linear calibration or to compensate for poor instrument performance. Quadratic calibrations require a minimum of six points for environmental applications and three points for other applications. Results are calculated as followed:

$$y = ax^2 + bx + c$$

Is rearranged to solve for concentration: $$x = \frac{\sqrt{b^2 - (4)(a)(c - y)} - b}{(2)(a)}$$

Is rearranged to solve for quadratic coefficient: $$a = \frac{y - bx - c}{x^2}$$

Is rearranged to solve for linear coefficient: $$b = \frac{y - ax^2 - c}{x}$$

Is rearranged to solve for constant coefficient: $$c = y - ax^2 - bx$$

NOTE: 'By Amount' must be used for Target applications so that concentration is on the 'x-axis'.

b) Third order calibrations are used in rare cases and require a minimum of seven points for environmental applications and four points for other applications. If used, the calibration curve is verified by a second method and the information is stored with the data.

$$y = ax^3 + bx^2 + cx + d$$

B. Precision, Accuracy, Comparability, and Completeness

1. Precision: *A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions expressed generally in terms of the standard deviation (EPA QA/G-5).*

Precision represents the ability of the laboratory to reproduce the same result over and over using the same technique. Standardizing the way a measurement is taken minimizes random error. The laboratory uses established methods where possible and writes and follows standard operating procedures in order to increase overall precision.

Precision on an individual sample is measured by preparing and analyzing the sample in duplicate or by preparing and analyzing a matrix spike duplicate. Precision is reported as RPD and calculated as:

$$\textit{Relative Percent Difference (\%RPD)} = \left| \frac{\textit{Result1} - \textit{Result2}}{\textit{Average of Result1 \& Result2}} \right| (100)$$

2. Accuracy: *A measure of the closeness of an individual measurement or the average of a number of measurements to the true value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; the EPA recommends using the terms 'precision" and "bias", rather than "accuracy" to convey the information usually associated with accuracy (EPA QA/G-5).*

Accuracy is simply about getting close to the "right answer". The problem lies in how to define the "right answer". Why does the EPA recommend the term "bias" over "accuracy" in its definition of accuracy? The reason is systematic

error based on inherent bias in the test method employed. Analysis of a given analyte by a variety of methods will yield different results based on the limitations or differences in each method. One way to limit systematic error of this kind is to compare and contrast results of different methods. This is often impractical. In most cases a test method is decided upon in order to best facilitate comparability. This is discussed further below. In some cases an acceptable alternate method may be selected if a known limitation exist for a particular method with a particular matrix. Within a method accuracy is often measured by processing a laboratory control (LCS) sample, standardized reference material (SRM) or a matrix spike (MS). The LCS and SRM are used to evaluate the accuracy and consistency of the test method in a controlled environment. The result of the analysis of the LCS or SRM is compared to a known value and established control limits. The matrix spike is used to evaluate the effect of individual sample characteristics that may interfere with the test method and create a bias. The sample is spiked with a known amount of the analyte of interest and evaluated against the true value. The calculation is described below:

$$\%\ Recovery = \left(\frac{Matrix\ Spike - Sample}{Spike\ Amount}\right)(100)$$

3. Comparability: *A measure of the confidence with which one data set or method can be compared to another (EPA QA/G-5).*

   It is critical that results from one data set be comparable to another. This is true when comparing data over time, across laboratories, or in some cases method to method. In order to satisfy the need for regulatory standards in industrial hygiene work, environmental compliance, or consumer product testing it is critical to create established base methods. The sources of these methods are plenty: ASTM, EPA, NIOSH, etc. Independent performance testing and round robin style testing is also used to generate consistency.

   When establishing a methodology for the first time it is important to sometimes try a variety of test methods in order to establish consistency. If you are able to employ three different approaches and two of them agree then you have established some degree of comparability. This will help to minimize a limitation inherent to any one of the approaches.

4. Completeness: *A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal, conditions (EPA QA/G-5).*

   Completeness refers to the thoroughness of the supporting data, the compliance to control limits, and the usefulness of results . Did you run all of the appropriate QC samples? Were the results in control? Is the data readily available and traceable? Are the reporting limits useful when compared to action levels? Does the raw data contain all of the appropriate information to reproduce the testing? Does the analytical report provide enough information for the client to make a reasonable assessment as to completeness as it refers to the data quality objectives? In most cases the data reported in the analytical report suffices in terms of evaluating completeness. Otherwise data and supporting SOPs are stored in the laboratory if further clarification is needed.

C. Acceptance Criteria for Quality Control Checks

1. Acceptance criteria for "run QC" or those checks that are specific to the analytical run or sequence are specified in individual SOPs. The criteria is determined by the method, like methods, instrument manufacturer recommendations, program rules or guidance, client requirements, or by the experience of the analyst setting up the method and writing the SOP.

2. Acceptance criteria for "batch QC" including laboratory control samples, matrix spikes, standardized reference materials (SRM), and surrogates are determined by one of the following methods.

   a) Laboratory determined control limits.
   b) Reference method requirements.
   c) Interim limits (dependent on analysis) if no method limit exists and laboratory determined control limits have not been set.
   d) Manufacturers limits, in the case of the use of an SRM.

3. Acceptance criteria for %RPD are determined by one of the following methods:

   a) Laboratory determined control limits.
   b) Reference method requirements
   c) Interim limits of $\leq$ 20% if no method limit exists and laboratory determined control limits have not been set.

4. Each SOP states specific requirements for accuracy and precision, where applicable.

D. Processing of Quality Control Data

The most common types of quality control checks are listed below with a description of their function. Frequency and control limits are specified in the individual SOP. Individual Standard Operating Procedures provide criteria for each analysis, and may supersede the information contained below.

1. There are a variety of blanks taking many names and having many uses. Instrument blanks (reagent blanks, ICBs, CCBs) are used to ensure that the analytical system is in control. Method blanks and/or matrix blanks are processed through the entire preparation and analysis procedure to evaluate a batch of samples. The specification on all of the blanks is that the absolute value is less than the reporting limit.

2. Field blanks, trip blanks, or equipment blanks are used to evaluate potential contamination from sampling events. These are reported like any other sample and are used by the client to evaluate the sampling process.

3. Initial calibration evaluation using a second source standard. This is a standard made up from a source different than that used to create the calibration. The purpose of this is to evaluate the accuaracy of the calibration standards.

4. Continuing calibration verification. This is an ongoing calibration check used to evaluate the analytical system stability over time. This may be made from either the calibration source or the second source material.

5. Interference check samples are used to evaluate the effects of high levels of potentially interferences on other analytes.

6. Laboratory control samples (LCS) are processed through the entire preparation and analysis procedures and are used to evaluate that the method is in control.

7. Matrix spikes are samples spiked with a known amount of target analyte processed through the preparation and analysis procedure. This is used to evaluate the effect of the sample matrix on the process.

8. Duplicate sample evaluation: The duplicate results shall be used to calculate the precision for the sample matrix or control sample as defined by the RPD. This may be on a sample duplicate, a matrix spike duplicate, or laboratory control sample depending on the method

9. Surrogate standards. Surrogates are compounds added to each sample with the purpose of behaving like target compounds but that are not expected to be found in the samples themselves. They are used to evaluate the extraction procedure.

10. Breakthrough: Breakthrough may result when the quantity of analyte sampled exceeds the capacity of the sampling device or because of atmospheric conditions (i.e. high relative humidity). Breakthrough is determined by analyzing the front and back sections of sorbent media separately. In general, if the quantity of analyte in the backup section exceeds 25% of the amount in the front section, breakthrough has occurred and resampling may be necessary.

E. Control Charts

Control charts for precision and accuracy shall be established for all major parameters. Control charts are used to trend performance over time.

Control charts may also be used to establish chart limits. A minimum of 20 data points should be used to establish chart limits. Warning limits of two standard deviations and control limits of three standard deviations shall be used in most cases.

True control limits are set based on a combination of the input from charted control limits, method defined limits, and the experience of the chemists responsible for the test. Using control charted limits without reference to method limits or experience is not responsible as it represents a small sample size that is biased completely to what is happening at Legend. We do want to control and improve processes at Legend so the input is valuable when determining limits and improving processes.

In some cases program rules require the use of specific limits, method limits, or control charted limits without reference to the other inputs. In such cases compliance to the program rules is maintained.

F. Measurement of Uncertainty – Statistical Analysis

1. The measurement of uncertainty is the estimate of the difference between a measured value and the true value. Many factors arising from systematic effects can attribute to uncertainty. The following factors were evaluated to determine the appropriate QC data to be used for statistical calculations.

   a) Sampling or sub-sampling
   b) Transportation, storage, and handling of samples
   c) Preparation of samples
   d) Environmental and measurement conditions
   e) Analysts
   f) Test method variations
   g) Instrumentation
   h) Calibration standards or reference material
   i) Results generation

   The sampling and transportation of samples is typically outside the control of the laboratory and is not included as part of LEGEND's measurement of uncertainty.

   Laboratory Control Samples (LCS) do address the other factors listed and are used to estimate the analytical measurement of uncertainty (U_a) for each significantly different matrix (e.g. air vs. wipe).

2. Identify what is being measured (measurand), matrix, and test method. For example: µg/m^3 of Lead in air by HNO_3/HCl digestion followed by analysis on the ICP-AES.

3. Calculate the standard deviation, average, and relative standard deviation (RSD) for a set of LCS data (at least 20 points). An RSD is used to compare the uncertainty between different measurements of varying results. An outlier test is not performed on the data. All data is used to err on the side of caution.

$$Relative\ Standard\ Deviation\ (RSD) = \frac{Standard\ Deviation}{Average}$$

4. Calculate the expanded measurement of uncertainty (U) by multiplying the RSD by a coverage factor (k). The coverage factor used is based on the level of confidence desired. LEGEND uses a coverage factor of 2 which represents a 95% confidence level. Multiply this number with the analyte concentration for the analytical measurement of uncertainty (U_a). For example: standard deviation = 2.7004, average = 106.15, lead in air result = 22 µg/ m^3.

$$RSD = \frac{2.7004}{106.15} = 0.025439$$

$$U = (0.025439)(2) = 0.050878$$

$$U_a = (22)(0.050878) = 1.1\ \mu g / m^3$$

5. Calculate the bias by subtracting the average recovery from 100 (retain the '+' or '-' sign), convert to a decimal form, and multiply by the test result.

$$Bias = \frac{(106.15 - 100)}{100}(22) = 1.4\ \mu g / m^3$$

6. Report the analytical measurement of uncertainty and/or bias when requested by the client. Report the analytical measurement of uncertainty at the 95% confidence level, along with the reported analyte result, in the same unit as the result. If bias is needed, report in the same unit as the result. From the example above, the following information would be reported:

   Result = 22 µg/m^3
   U_a = ± 1.1 µg/m^3
   Bias = + 1.4 µg/m^3

7. Recalculate the measurement of uncertainty and/or bias if significant changes are made to any of the factors above.

G. Detection and Reporting Limits

1. Instrument Detection Limit (IDL)

   Specific to metals analysis, this is the lowest concentration that an analyte can be detected on the instrument using a particular methodology. The value is obtained by calculating three times the standard deviation of a reagent blank analyzed 10 times at the same wavelength. The IDL is calculated before any samples are analyzed and when there has been a significant change with the instrument.

2. Method Detection Limit (MDL)

   Defined as the minimum concentration that can be reported for a specific substance with 99% confidence, it is determined by analyzing a minimum of seven samples at a concentration between one and five times the estimated detection limit. The policy at LEGEND is to run a minimum of eight samples so an outlier test may be performed. The MDL is calculated as MDL = T(s) where s is the standard deviation of the analysis, and T is the student's T-value associated with a 99% confidence level and a standard deviation estimate with N-1 degrees of freedom. The MDL study is determined when a significant change in either the method or instrumentation has occurred.

3. Practical Quantitation Limit (PQL)

   The PQL is determined by multiplying the standard deviation calculated in the MDL by ten.

4. Reporting Limit (RL)

   Defined as a value that is set by the laboratory for reporting purposes. The RL should be above the calculated PQL but must be above the MDL.

   Results below the reporting limit are reported as less than the value of the RL. Sample results below the RL but above the MDL are flagged at the client's request.

H. Trend Analysis

Major parameters shall be monitored in applicable analyses against established control charts for data trends. If a trend is identified, corrective action must be taken. Below is a list of run rules that should be used to identify trends. The source for this run rule is the Handbook of Statistical Methods for Engineers and Scientists, by Harrison M. Wadsworth, 1990

1. Six consecutive points in a downward direction
2. Six consecutive points in an upward direction

8.3 Control of Nonconforming Product

There are three areas defined as internal nonconforming product at LEGEND: method error, analytical data error, and report error.

A. Method errors are documented through method nonconformances (MNC) as described in Work Instruction 'Method Nonconformance (MNC) Documentation'.

B. Analytical data errors (systematic) are documented through Corrective Action Reports (CAR) as described in 7.5.2, C.

C. Report errors (systematic) are documented through Corrective Action Reports (CAR) as described in 7.5.2, D.

8.4 Analysis of Data

Data is collected and analyzed to determine the effectiveness of the quality management system and identify areas for improvement. This data analysis includes information relating to:

A. Customer satisfaction as described in 8.2.1

B. Conformity to product requirements collected during the testing and analysis process

C. Customer complaints as described in 8.2.1

D. Processes as described in 8.2.3

E. Vendors from the vendor management process described in 7.4.1.

8.5 Improvement

8.5.1 Continual Improvement

Continual improvement is an on-going expectation. Continual improvement is achieved through the use of quality objectives, audit results, analysis of data, corrective and preventive actions, and management review processes.

8.5.2 Corrective Action

A. Corrective action is taken to prevent recurrence of a nonconformance. All employees shall be responsible for reporting any nonconformance that they observe or identify to their supervisor, department manager, technical director, and/or QA/QC department. The appropriate supervisor is responsible for assuring that the corrective actions are taken. It is the responsibility of the QA/QC department to monitor all corrective actions taken.

B. Nonconformances may include (but are not limited to) results from the following:

1. External and internal audits

2. Client complaints

3. Management reviews

4. Chronic QC data outside of limits (i.e. blanks, duplicates, spikes, etc.)

5. Proficiency testing (PT) samples outside limits

6. Nonconforming equipment/materials

7. Nonconforming product (laboratory error examples)

    a) Chronic exceedances of sample holding time
    b) Incorrect sample preparation
    c) Wrong analysis procedure
    d) Improper sample storage
    e) Mixing up samples during analysis
    f) Reporting wrong data

C. Corrective action is divided into two sections:

1. Remedial action – dealing with the immediate problem (i.e. recalibrating, updating training records, responding to an audit finding, etc.)

2. Cause analysis – searching for the underlying problem or root cause (the most basic reason, which if eliminated, would prevent recurrence). Eight areas are defined and used to categorize the root cause. They are:

    a) Process – no procedure exists or the existing procedure did not meet the required objectives
    b) Training – analyst was not trained or inadequately trained for the issue found
    c) Competency – analyst was trained but did not have the skills required to perform the task
    d) Accountability – analyst was trained but did not do what was required of them
    e) Resources – equipment or supplies was not sufficient for the task
    f) Documentation – inadequate documentation or records maintained
    g) Supplier/Vendor – subcontractor or supplier did not meet requirements
    h) Client Requirements – client specific items were not known or defined adequately

D. Corrective actions are required in writing and must be completed within an agreeable timeframe. A 'Corrective Action Report (CAR)' is filled out as follows:

1. Assign responsibility for taking the corrective action

2. Identify and describe the problem

3. Determine if the nonconformance impacts the client

4. Document the remedial action taken

5. Investigate the possible cause(s) looking for the root cause

6. Determine the action to be taken

7. Implement any new preventive action (fill out 'Preventive Action Report')

E. There may be times when the remedial action is sufficient to eliminate the problem. In this case, it is acceptable to record 'Not applicable' in the root cause section of the form. The corrective action should be appropriate to the magnitude and risk of the nonconformance.

F. The area where the nonconformance occurred should be revisited (internal audit) at a later stage to determine the efficiency of the corrective action. All records generated should be kept in the QA department.

8.5.3 Preventive Action

A. Preventive action is taken to prevent occurrence of a nonconformance. It is a pro-active process to identify opportunities for improvement rather than a reaction to problems or complaints. Preventive actions may result from the following areas:

1. Corrective actions

2. Suggestions from staff members (employees are encouraged to identify possible improvements to the quality system or test procedures)

3. Suggestions from clients

4. External and internal audits

5. Management reviews

6. Meetings of professional organizations

7. Literature

8. Routine maintenance

B. Preventive action is divided into two sections:

1. Risk assessment – assessing areas where failures may occur and have procedures in place to prevent them (i.e. staff training, servicing equipment, monitoring equipment, validating methods, etc.)

2. Continuous improvement – identifying and addressing improvements in the system and potential sources of nonconformances

C. One of the largest areas of preventive action is preventive maintenance of equipment. The initial determination of a specific preventive maintenance item is considered a preventive action. Preventive maintenance maintains proper instrument and equipment performance and prevents instruments and equipment from failing during use. It considers instruments, equipment and parts that are subject to wear, deterioration or other changes in operational characteristics; spare parts that should be available to minimize downtime; and the frequency which maintenance is required. See equipment SOPs for specific information.

D. When a preventive action is initiated, a review of the current procedure is performed to develop an action plan to determine what action will be taken. A 'Preventive Action Report (PAR)' should be filled out as follows:

1. Identify the potential issue

2. Evaluate the need for the action

3. Specify the action to be taken

4. Assign personnel to perform the preventive action

5. Determine if the action was effective and if it should be implemented throughout the company (if applicable)

SECTION 9

SPECIAL QUALITY ASSURANCE

9 SPECIAL QUALITY ASSURANCE

Specific analyses may require participation in an accreditation program that does not apply to general lab analyses. This section applies to these specific analyses.

9.1 Bulk Asbestos Quality Assurance

LEGEND will comply with the requirements in the NIST Handbooks 150 and 150-3.

A. Bulk Analysis Procedure

LEGEND uses a quality assurance procedure entitled, "BULK SAMPLE IDENTIFICATION USING POLARIZED LIGHT MICROSCOPY," for its in-house analysis program. The procedure is based on the Environmental Protection Agency (EPA), "Interim Method for the Determination of Asbestos in Bulk Building Materials," 1993 (EPA-600/R-93/116). The procedure describes the process used in identifying the presence of asbestos, prepping samples, and estimating the percent composition of asbestos in bulk samples.

B. Data Reporting

1. Use of NVLAP Logo

Reports that contain results covered by NVLAP accreditation must indicate in the report and in the records any and all results that are not covered by the NVLAP accreditation. A statement must be made in the report referencing the data not covered by NVLAP accreditation and the NVLAP logo may only be used on the page(s) that NVLAP accreditation applies. For marketing materials, websites, or other company literature, the NVLAP logo must indicate the facility where the accreditation is held. For all applications where the NVLAP logo is used, it must be accompanied by the NVLAP Lab Code.

2. Layered Samples

When layers are present in the sample, each layer is analyzed and reported separately unless a composite is requested by the client, and must indicate so on the report.

3. Approved Signatures

Approved signatures for reports referencing data covered by NVLAP accreditation are limited to currently approved personnel.

C. Quality Assurance/Quality Control (QA/QC)

LEGEND's QA Manual is used as the guiding document for all company operations. Procedures, training records, and equipment calibration are all dictated through this document. All documents and analysis sheets shall be completed, signed, and dated before a second analyst reviews them.

The Microscopy Supervisor has overall responsibility for the technical operations of the PLM laboratory.

D. Inter-Laboratory Quality Control Program

LEGEND participates in a tri-annual Interlaboratory Round Robin Quality Control Program for bulk sample analysis that includes analysis of three to four bulk samples. The program is sponsored by the Twin City Asbestos Round Robin Program and includes approximately six laboratories. A designated laboratory collects the data and provides the analytical statistics for the program.

LEGEND participates in the NIST National Voluntary Laboratory Accreditation Program (NVLAP) for bulk asbestos proficiency analysis. Proficiency samples are analyzed twice a year. Analyses are not contracted out to another laboratory. All analysts will participate and analyze the samples separately. One single result will be submitted to NVLAP by the laboratory.

The samples will be kept for in-house use in interanalyst comparisons. Problems observed from the test results will be discussed and resolved by the analysts and the Microscopy Supervisor.

E. Intra-Laboratory Quality Control Program

1. 10% of the samples will be set up in duplicate by the same analyst. The results of these samples will be recorded on the 'Bulk Asbestos Chart Table' form. The acceptable ranges are calculated on each appropriate form (1-10% and >10%).

2. If two different analysts read the same slide, the acceptable ranges are:

   a) $\leq$ 10% asbestos – within 3% units
   b) 10% to 75% - within 20% units
   c) $\geq$ 75% asbestos – within 10% units

3. NIST standard bulk asbestos reference materials are set up as needed and used to crosscheck samples analyzed for clients.

F. Instrument Calibration/Maintenance/Contamination Control

Appropriately trained in-house personnel will service all polarizing microscopes used in bulk analysis annually.

All polarizing microscopes used in bulk analysis will be calibrated by checking the alignment of the central stop. Ensure that the polarizer and analyzer are orientated at 90 degrees to one another, center the objectives, and check that the substage condenser and iris diaphragm are centered. Standard reference materials from NIST (SRM 1866 and 1867) are used as analysis reference materials.

Refractive index liquids will be calibrated with an accuracy of $\pm$ 0.004 once a month, or as needed to verify accuracy, with Cargill R.I. glass beads and recorded in a notebook. Approximately once a day, when used, slides, cover slips, and regularly used refractive index oils will be checked for contamination and a blank sample (non-asbestos containing) will be analyzed and recorded in a notebook.

To ensure a contamination free workspace, the hood, sample boards, tools, and counters will be wiped down with wet wipes after sample analysis. Phase contrast microscopy (PCM) samples will be run in the asbestos laboratory area, analyzed, and recorded on a bi-annual basis.

G. Measure of Uncertainty

In estimating the measure of uncertainty, some test procedures preclude an actual calculation. The components of uncertainty would be defined as follows:

1. Homogeneity of the sample
2. Analyst accuracy and precision
3. Preparation procedures
4. Calibration of the microscope
5. Calibration of the refractive index liquids
6. Cleanliness of the facility
7. Quality of the standard reference materials

9.2 Collection and Analysis of Asbestos Fiber Air Samples Quality Assurance Program

A. Objective

The objective of the program is to ensure consistent and accurate fiber count data is generated both in the field and in the laboratory and comply with OSHA 29 CFR 1926.58, Appendix A requirements.

B. Sampling and Analytical Procedure

1. Sampling

The sampling medium for air samples shall be mixed cellulose ester filter membranes manufactured so as to be suitable for asbestos fiber determination. The standard medium for phase contrast microscopy (PCM) shall be a 25 mm, 0.8-micron pore size MCE filter in an open-faced 50-mm electrically conductive extension cowled cassette. The standard medium for transmission electron microscopy (TEM) shall be a 25-mm 0.45 micron pore size MCE filter followed by a 0.5 micron pore size MCE filter in an open faced 50-mm electrically conductive extension cowled cassette shipped loaded from the manufacturer.

Samples will be collected using both high and low flow vacuum pumps. The sampling pumps shall be calibrated each day prior to use with a primary standard calibrator or a rotometer which is calibrated every 3 months against a primary standard. Personal and area enclosure samples shall be collected using low flow pumps at a flow rate of 0.5 to 2.5 liters per minute (LPM). Exterior area enclosure and final clearance air samples shall be collected using high flow pumps at a flow rate of from 5 to 16 LPM.

Personal samples shall be collected in the breathing zone of the employee being monitored.

2. Air Volume

   Where possible, a sufficient volume for each air sample shall be collected to yield between 100 and 1,200 fibers per square millimeter on the membrane filter. A quantifiable limit will be calculated for each sample based on sample air volume.

3. Field Blanks

   Each set of samples taken will include 10 percent (10%) blanks or a minimum of 2 blanks. The blank results shall be analyzed with the samples and an average of the results subtracted from the sample counts.

   Any filter media having blanks that when analyzed yield 5 or more fibers per 100 fields, shall be discarded. Any samples collected with that media shall be voided and re-sampling conducted. For situations where more than 40 samples are collected using the same media on the same day, the air sampling professional will analyze 4 blanks initially. If the results of the 4 blanks are less than 5 fibers per 100 fields and the counts are within 2 fibers of each other, no further blanks will be collected for that set of samples.

4. Phase Contrast Microscopy (PCM)

   Air sample analysis shall be in accordance with NIOSH 7400A Method. Sample data shall be reported on the appropriate LEGEND TECHNICAL SERVICES, INC. (LEGEND) data sheets. Field analysis shall be conducted under the most dust free conditions obtainable at the site.

   The analyst shall observe the NIOSH 7400A counting rules and count enough graticule fields to yield 100 fibers or count a minimum of 20 fields.

   Blind recounts by the analyst shall be conducted at the rate of 10 percent. Upon completion of a project, the analyst returns his/her samples and count sheets to the designated quality assurance (QA) person. The QA person randomly selects 10 percent of the samples, relabels the samples, and resubmits them to the original analyst for blind recount. The blind recount results are then submitted back to the QA person for compilation and determination of acceptability. Results of the blind recheck are included in the client's reports.

   At completion of a project, samples are stored and disposed of in accordance with LEGEND policies.

5. Microscope Maintenance

   The microscopes used by LEGEND personnel shall be aligned at the beginning of each day that the microscope is used and at any time the analyst observes deterioration in the image. It shall be done according to written protocols and shall include but is not limited to alignment of the microscopic optics.

   The analyst shall run the HSE/HPL test slide on the microscope, according to the written procedure, to ascertain whether its performance is within the established guidelines.

The analyst shall then measure the projected diameter of the Walton-Beckett graticule to assure that it is within established guidelines.

Any microscope not meeting the established criteria shall not be used for asbestos fiber counting.

C. Personnel Requirements

All analysts performing asbestos fiber counting analysis must have successfully completed an in-house training course for the evaluation of fibrous materials that is equivalent to the NIOSH 582 course or have completed the NIOSH 582 course.

1. Precision

Ten percent (10%) of the samples analyzed by each analyst shall be randomly selected, re-numbered, and submitted to the analyst for blind re-check. The results of the re-check will be compared to the results of the original analysis. Results will be considered acceptable if the difference between the two results is less than 2.77 $(F)S_r$, where F is the average of the two fiber counts and S_r is the relative standard deviation which is derived from historical in-house data. If deficiencies are noted, the analyst will be required to perform further re-checks and a review of the microscope and analyst performance will be made by supervisory personnel until deficiencies are corrected.

2. Accuracy

a) Intra-laboratory

Reference slides are analyzed per SOP 'Asbestos Fiber Counting NIOSH 7400 Method'.

b) Inter-laboratory

LEGEND will participate in the American Industrial Hygiene Association (AIHA) Proficiency Analytical Testing (PAT) Program for asbestos fiber counting. PAT samples will also be analyzed by a number of analysts to assess variation in technique.

3. Identification of Analytical Bias Process

Analytical bias will be identified through the following sources:

a) Blind re-count analyses
b) Repetitive counts on the same filter
c) Intra-laboratory quarterly analyses
d) Inter-laboratory Round Robin and AIHA PAT analyses

If the analyses from the above sample analyses are not within acceptable limits for their respective quality control (QC) parameters, then analysis bias will be suspected. The designated QC supervisor will evaluate each outlier and determine what corrective action shall be taken.

9.3 Collection and Analysis of Industrial Hygiene Samples

A. Objective

The objective of the program is to ensure consistent and accurate industrial hygiene sample data is generated both in the field and in the laboratory. Industrial Hygiene samples are performed within LEGEND's laboratory that also performs environmental and industrial sample analysis.

B. Applicability

This section is applicable to airborne analysis of:

1. Solvent
2. Free silica
3. Metals
4. Gravimetric dust
5. Other specialty IH parameters

C. Sampling Procedure

1. Sampling Media

Sample in accordance with established OSHA/NIOSH procedures and LEGEND SOP's utilizing the media recommended in the procedures.

Media typically used includes the following:

a) Solvent vapor - charcoal, silica gel, or passive organic vapor monitor
b) Free silica - 37mm 0.8 micron mixed cellulose ester filters or PVC filters
c) Metals - 37mm 0.8 micron mixed cellulose ester filters
d) Gravimetric dust - 37mm 0.8 micron PVC filters
e) Other specialty IH parameter - per method requirements

2. Sample Collection

Samples are collected using both high and low flow vacuum pumps. The sampling pumps shall be calibrated each day prior to use with a primary standard calibrator or a rotometer which is calibrated every 3 months against a primary standard. Air sampling duration and flow rate depend on the method and expected level of concentration.

A sufficient volume of air shall be collected on the appropriate sampling media to provide a lower limit of detection within the applicable OSHA PEL or ACGIH TLV at the laboratory lower detectable limit for the compound of interest.

3. Field Blanks

   Each set of samples taken will include a minimum of one blank. Additional blanks may be required where the sampling media is known to be contaminated with a low baseline level of the compound of interest. Specific compounds requiring multiple field blanks to assess level of media contamination include potassium, sodium, and gravimetric dust.

4. Replicate Samples

   Where sampling conditions allow, collect and submit blind replicates to the laboratory for verification of sampling and analysis precision. Report the results of blind replicate sampling with sampling results.

5. Spikes Samples

   Submit sampling media spiked with the target compound(s) at expected levels to the laboratory with each batch of laboratory samples collected for solvents or metals analysis. Report the results of the spiked samples with sampling results.

6. Sample Submission

   Samples shall be submitted to the laboratory with a completed chain-of-custody.

D. Personnel Requirements

1. Field Sampling

   Personnel performing the field sampling are under the direction of the field supervisor and have received training specific to sample collection and analysis.

2. Laboratory Analysts

   Laboratory analysts performing the industrial hygiene analysis have received either in-house or outside training in their specific area of expertise including chromatography, atomic absorption, infrared spectroscopy, etc.

   Training is documented in the employee individual training manuals.

E. Laboratory Analysis

1. Standard Operating Procedures

   Standard operating procedures shall be written for each routine industrial hygiene analysis performed in the laboratory. Standard operating procedures are written and updated in accordance with SOP 'Preparation of Department Standard Operating Procedures'. Where methods exist, SOPs are based on established EPA, OSHA, and NIOSH methods.

2. Analytes Not Routinely Performed by the Laboratory

   Where an analysis is requested and a written LEGEND SOP does not exist, the analyst must first consult the current OSHA and NIOSH Air Sampling Method Books

to identify existing published methods for the analyte of interest. Consult with the IH Technical Director before committing resources to method development. Verify that LEGEND has the proper equipment, materials, etc. prior to setting up to perform the method. Analyze standards and spiked samples to verify analytical recoveries prior to accepting any field samples for analysis. Provide field personnel with spiked samples to be transported and analyzed with the field samples.

F. Data Reporting

Data are reported in accordance with LEGEND SOP 'Report Format for Factual Reports' in the case of reports sent directly to an outside client. Internal data is submitted back to the member of the field staff who is acting as the client manager for data interpretation and reporting in accordance with LEGEND SOP 'Report Format for Formal Report'.

G. Quality Assurance/Quality Control (QA/QC)

LEGEND's QA Manual is used as the guiding document for all company operations. Procedures, training records, and equipment calibration are all dictated through this document.

9.4 Consumer Products Quality Assurance

LEGEND will comply with the requirements of A2LA, including P101.

A. Use of the A2LA symbol and accreditation status

Where the A2LA name is used in a narrative reference to accredited status, it shall always be accompanied by at least the word "accredited". In proposals, quotations, and reports, analyses that are covered under A2LA accreditation shall be distinguished from those that are not. For marketing materials, websites or other company literature, the "A2LA Accredited" symbol must always be accompanied by the LEGEND A2LA testing laboratory certificate number and a clear indication which location the accreditation is for.

SECTION 10

LABORATORY QUALITY ASSURANCE MANUAL REVIEW AND UPDATES

10 LABORATORY QUALITY ASSURANCE MANUAL REVIEW AND UPDATES

10.1 Review

A. The Quality Assurance Manual (QAM) will be reviewed every two years at minimum by the QA/QC Coordinator or designated representative.

B. The review will be documented on the 'Document Review' Form.

10.2 Updates

A. Changes in the plan will be documented on the 'Document Review' Form and maintained within the QA Manual.

B. The changes will be incorporated in the plan and the issue date on the cover be changed to reflect the date of changes.

C. Changes in the Organizational chart may be made without documentation or revision to the QA Manual.

DOCUMENT REVIEW

DOCUMENT:	QUALITY ASSURANCE MANUAL
REVIEWER:	William Dahl
DATE:	5/3/11

SECTION	CHANGE	RATIONALE
2	Added reference EPA QA/G-5	EPA QA Document
4.2.2	Removed "reviewed annually."	Annual review is not a requirement
4.2.3 A	Changed to "The QA/QC Coordinator shall review and update the QAM when there is a change to a quality policy and/or procedure outlined in the QAM or every two years at a minimum" from "The QA/QC Coordinator shall review and update the QAM on an annual basis."	Annual review is not a requirement
4.2.3 B	Removed "(except LABENV)"	All SOPs are treated the same
4.2.4	Removed ELLAP as exception to 5 year record retention rules	ELLAP Updated policy 4/2010 to go from 10 years to 5 years
4.2.4	Changed C of A to coincide with data rather than indefinite	C of A is part of data record rather than a QC record
4.2.4	Changed storage of C of A from QA Department to laboratory	Current Process
4.2.4 C4	Control chart storage re-defined to those charts used to establish control limits to be stored for 5 years. Charts used for trending need not be stored. All charts can be reproduced from the data in LIMS.	Clarification
4.2.4 C6	Software verification distinction made between commercial software and laboratory created spreadsheet. Verification of commercial software changed to versioning rather than annual.	Neither laboratory personnel nor QA has access to vendor source code; therefore, potential for manipulation or corruption is very low with no change in version.
4.2.4 C10	Added LIMS sequence as an option for tracking samples run on each instrument.	The LIMS sequence mimics the function of a paper logbook tracking the order of samples analyzed and associating information.
4.2.4 C14	Uncertainty of measurement changed to be available on all types of reporting per client request.	Current Process
5.5.3	Changed from quarterly to annual	Current Process
5.6.1	Changed from quarterly to annual	Current Process
6.2.2	Changed ongoing demonstration of capability from annual to test or program specific requirements.	Current Process
6.3 C	Added references to US Filter/Siemens and to Nanopure systems and deleted E-pure system	E-Pure system removed
Equipment Information	Added software version and operating manual version to the table	AIHA Requirement

DOCUMENT REVIEW

DOCUMENT:	QUALITY ASSURANCE MANUAL
REVIEWER:	William Dahl
DATE:	5/3/11

SECTION	CHANGE	RATIONALE
7.2.1	Added confidentiality clause for release of methods with proprietary information to client.	This is needed to protect any trade secrets.
7.2.1	Removed "validated" under employing non-standard methods.	Not all methods undergo full validation prior to use. This is negotiated client by client.
7.2.3	Removed annual survey	Clients get annoyed if surveys are sent every year and this mechanism is not effective
7.2.3	Added that feedback is received via contact with the Client Manager, Marketing Director, and QA/QC Coordinator.	This has proven the most effective mechanism for feedback
7.5.2 E	Verification of commercial software changed to versioning rather than annual.	Neither laboratory personnel nor QA has access to vendor source code; therefore, potential for manipulation or corruption is very low with no change in version.
7.5.2 E	Added clarification where non-controlled spreadsheets are used.	Clarification
7.6 B 5	Added 'The piece of equipment is tagged as "out of service" until such time that it has been repaired and re-calibrated.'	This step is necessary to prevent further deviations due to a piece of equipment that is out of calibration.
7.6 C 3	Section on calibration curves revised	Current Process and Clarification
8.2.4 A	Added language to introduce curve fit equations	Clarification
8.2.4 B	Added sourced definitions for terms discussed	Clarification
8.2.4 B 3	Removed "representativeness" and added "comparability"	Information is more useful
8.2.4 C	Removed frequency	Addressed elsewhere in QAM
8.2.4 C	Reworded some information for clarity	Clarification
8.2.4 D	Reworded to define control samples as to their function	Clarification
8.2.4 E	Control charting reworded	Current Practice
8.2.4 F	Uncertainty completely re-done	AIHA requirement
8.2.4 G	MDL annual requirement removed	Current Process

DOCUMENT REVIEW

DOCUMENT:	QUALITY ASSURANCE MANUAL
REVIEWER:	William Dahl
DATE:	5/3/11

SECTION	CHANGE	RATIONALE
8.2.4 H	Trend Analysis Changed	AIHA requirement
10.1	Changed QAM review to every 2 years	Annual Review is not a requirement

Term	Definition
AA	Atomic Absorption
Accreditation	Procedure by which an authoritative body gives formal recognition that a body or person is competent to carry out specific tasks.
ASE	Accelerated Solvent Extractor, used to extract solid samples
Audit	Systematic, independent and documented process for obtaining audit evidence and evaluating it objectively to determine the extent to which audit criteria are fulfilled. First-party (internal) audits – conducted by, or on behalf of, the organization itself for internal purposes and can form the basis for an organizations self-declaration of conformity. Second-party audits – conducted by parties having an interest in the organization, such as customers, or by other persons on their behalf. Third-party audits – conducted by external independent organizations. Such organizations provide certification or registration of conformity with requirements such as those of ISO 9001 and ISO 14001:1996. Combined audit – when quality and environmental management systems are audited together Joint audit – when two or more auditing organizations cooperate to audit a single auditee jointly.
Batch	One to twenty samples of the same matrix prepared for single or multiple analyses that will be analyzed during one operation at a given specific time frame.
Blind Standard	Sample with a known amount of analyte where the concentration of the analytes is unknown to the analyst but known to the supervisor or QA/QC Coordinator.
°C	Degrees Celsius (Temperature)
Calibration	Adjusting a measuring instrument to make it accurate. The set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system and the corresponding values or a quantity realized by a reference standard.
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
Certification	Procedure by which a third party gives written assurance that product, process or service conforms to specific requirements.

Term	**Definition**
Client Protocol	A document created specifically for a client that summarizes the method to be used.
COC	Chain-of-Custody, record that documents the possession and handling of samples from collection through submittal to the laboratory.
Competence	Demonstrated ability to apply knowledge and skills.
Compliance	An affirmative indication or judgment that the supplier of a product or service has met the requirements of the relevant specifications, contract or regulation; also the state of meeting the requirements.
Concession	Permission to use or release a product that does not conform to specified requirements. NOTE: A concession is generally limited to the delivery of a product that has nonconforming characteristics.
Conformity	Fulfillment of a requirement.
Continual Improvement	Set of activities routinely carried out to increase the ability to fulfill requirements. NOTE: The process of establishing objectives and finding opportunities for improvement is a continual process through the use of audit findings and conclusions, analysis of data, management reviews and corrective or preventive action.
Contract	Agreed requirements between a supplier and customer transmitted by any means.
Controlled	Orderly, repeatable, manageable, and predictable.
Correction	Action to eliminate a detected nonconformity. NOTE: A correction can be made in conjunction with a corrective action.
Corrective Action	Action to eliminate the cause of an existing nonconformity or other undesirable situation. Corrective action addresses the actual problem. NOTE 1: There can be more than one cause for nonconformity. NOTE 2: Corrective action is taken to prevent recurrence whereas preventive action is taken to prevent occurrence.
Customer	Organization or person that receives a product or service from a supplier (e.g. consumer, client, end-user, retailer, beneficiary and purchaser). NOTE: A customer can be internal or external to the organization.

Term	**Definition**
Customer Satisfaction	Customer's perception or the degree to which the customer's requirements have been fulfilled. NOTE: Customer complaints are a common indicator of low customer satisfaction but their absence does not necessarily imply high customer satisfaction.
CVAA	Cold Vapor Atomic Absorption
CVS	Calibration Verification Standard
Design and Development	Set of processes that transform requirements into specified characteristics or into the specification of a product, process or system. NOTE 1: The terms "design" and "development" are sometimes used synonymously and can be used to define different stages or the overall design and development process. NOTE 2: A qualifier can be applied to indicate the nature of what is being designed and developed (e.g. product design and development or process design and development).
Document	Information and its supporting medium (e.g. record, specification, procedure document, drawing, report, or standard). NOTE 1: The medium can be paper, magnetic, electronic or optical computer disc, photograph or master sample, or a combination thereof. NOTE 2: A set of documents, for example specifications and records, is frequently called "documentation".
DQO	Data Quality Objective (Precision, Accuracy, Representativeness, Comparability, Completeness)
Duplicate	Sample from which two equal representative portions have been taken and analyzed separately.
ECD	Electron Capture Detector
Effectiveness	Extent to which planned activities are realized and planned results achieved.
Efficiency	Relationship between the result achieved and the resources used.
ELCD	Electrolytic Conductivity Detector
Equipment Blank	Water sample that has been processed through the sampling equipment in the same manner as an actual sample to determine if field cleaning procedures were adequate.

Term	Definition
FID	Flame Ionization Detector
Finding	An important conclusion based on observations.
FLAA	Flame Atomic Absorption
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GLP	Good Laboratory Practices
HPLC	High Performance Liquid Chromatography
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
ICV	Initial Calibration Verification
ID	Identification
IDC	Initial Demonstration of Capability
IEC	Interelement Correction
Infrastructure	System of facilities, equipment and services needed for the operation of an organization.
Inspection	Conformity evaluation by observation and judgment accompanied as appropriate by measurement, testing or gauging.
Interference	Any physical property or chemical constituent of a sample that causes either a positive or negative error in the analytical result.
International Organization for Standardization (ISO)	The specialized international agency for standardization, at present comprising the national standards bodies of 140 countries. The American National Standards Institute (ANSI) is the member body representing the United States. The address of ISO is: ISO, Case Postale 56 CH-1211 Geneva 20, Switzerland.
IStd.	Internal Standard, pure analyte or analytes, not typically found in environmental samples, added to a test sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes and surrogates that are components of the sample or solution.
LCS	Laboratory Control Standard (or Sample), a laboratory blank that has known amounts of the analytes of interest added to it. Percent recoveries are calculated for each analyte to assess the analytical accuracy for the method.

Term	Definition
LCSD	Laboratory Control Standard (or Sample) Duplicate, a duplicate sample of the LCS. The RPD between the samples is used to assess the analytical precision for the method.
LIMS	Laboratory Information Management System
LUST	Leaking Underground Storage Tank
Management	Coordinated activities to direct and control an organization.
Management Representative	The person with the defined authority and responsibility to carry out the requirements of ISO 9001.
Management System	System to establish policy and objectives and to achieve those objectives. NOTE: A management system of an organization can include different management systems, such as quality management system, a financial management system or an environmental management system.
MB	Method Blank, clean matrix where all reagents are added in the typical amount used in the samples and then processed through the entire sample preparation and analytical process.
MDL	Method Detection Limit, lowest concentration level that can be determined to be statistically different from a blank for an analytical test method. The calculation is found in 40CFR, Part 136, Appendix B.
Measuring Equipment	Measuring instrument, software, measurement standard, reference material or auxiliary apparatus or combination thereof necessary to realize a measurement process.
Monitor	Observe, supervise, keep under review. Measure or test at intervals, especially for the purpose of regulation of control.
MS	Matrix Spike, sample to which a predetermined quantity of analytes of interest is added prior to sample preparation (extraction, digestion, etc.) and analysis. Percent recoveries are calculated for each analyte to assess the analytical precision including all potential sample interferences.
MSD	Matrix Spike Duplicate, a duplicate sample of the MS. The RPD between the samples is used to assess the analytical precision including all potential sample interferences.
MSDS	Material Safety Data Sheets
NIST	National Institute of Standards and Technology. An agency of the United States Department of Commerce the institute develops measurement standards and techniques for American science and industry and for

Term	Definition
	other government agencies. NIST also helps U.S. companies adopt new technologies to increase their international competitiveness.
Noncompliance	A deviation from the requirements of the standard.
Nonconformity	Non-fulfillment of a requirement.
Objective Evidence	Data supporting the existence or verity of something. NOTE: Objective evidence may be obtained through observation, measurement, test, or other means.
Organization	Group of people and facilities with an arrangement of responsibilities, authorities and relationships (e.g. company, corporation, firm, enterprise, institution, charity, sole trader, association, or parts or combination thereof). An organization can be public or private.
Outlier Test	$Grubb's\ T\ Test = \frac{\lvert suspected\ outlier - mean \rvert}{s}$ s = Standard Deviation
Parameter	Any chemical, biological, physical, microscopic test, examination, or analysis conducted on a specific matrix.
Preventive Action	Action to eliminate the cause of a potential nonconformity or other undesirable potential situation. NOTE 1: There can be more than one cause for a potential nonconformity. NOTE 2: Preventive action is taken to prevent occurrence whereas corrective action is taken to prevent recurrence.
Procedure	Specified way to carry out an activity or a process. NOTE 1: Procedures can be documented or not.
Process	Set of interrelated or interacting activities that transform inputs into outputs. NOTE 1: Inputs to a process are generally outputs of other processes. NOTE 2: Processes in an organization are generally planned and carried out under controlled conditions to add value. NOTE 3: A process where the conformity of the resulting product cannot be readily or economically verified is frequently referred to as a "Special process."

Term	**Definition**
Process Control	A system of measurements, decisions and adjustments within a process intended to ensure the output of the process conforms with pertinent specifications.
Product Realization	All processes that are used to bring products into being.
Proficiency Testing Sample	Sample obtained from an approved provider to evaluate the ability of a laboratory to produce an analytical test result meeting the definition of acceptable performance. The concentration of the analyte in the sample is unknown to the laboratory at the time of analysis.
Qualified	Verified as capable of providing the required performance.
Quality	Degree to which a set of inherent characteristics fulfills requirements. NOTE 1: The term "quality" can be used with adjectives such as poor, good or excellent. NOTE 2: "Inherent", as opposed to "assigned", means existing in something, especially as permanent characteristic.
Quality Assurance	Part of quality management focused on providing confidence that quality requirements will be fulfilled.
Quality Audit	An audit of the quality management system.
Quality Control	Part of quality management focused on fulfilling quality requirements.
Quality Management	Coordinated activities to direct and control an organization with regard to quality (e.g. quality policy, quality objectives, quality planning, quality control, quality assurance and quality improvement).
Quality Management System	Management system to direct and control an organization with regard to quality.
Quality Manual	Document specifying the quality management system of an organization.
Quality Objective	Something sought, or aimed for, related to quality. NOTE 1: Quality objectives are generally based on the organization's quality policy. NOTE 2: Quality objectives are generally specified for relevant functions and levels in the organization.

Term	**Definition**
Quality Policy	Overall intentions and direction of an organization related to quality as formally expressed by top management. NOTE 1: Generally the quality policy is consistent with the overall policy of the organization and provides a framework for the setting of quality objectives. NOTE 2: Quality management principles presented in this International Standard can form a basis for the establishment of a quality policy.
Record	Document stating results achieved or providing evidence of activities performed. NOTE 1: Records can be used, for example, to document traceability and to provide evidence of verification, preventive action and corrective action. NOTE 2: Generally records need not be under revision control.
Release	Permission to proceed to the next stage of a process.
Repair	Action on a nonconforming product to make it acceptable for the intended use. NOTE 1: Repair includes remedial action taken on a previously conforming product to restore it for use, for example as part of maintenance. NOTE 2: Unlike rework, repair can affect or change parts of the nonconforming product.
Requirement	Need, expectation, or obligation that is stated or generally implied. NOTE 1: "Generally implied" means that it is custom or common practice for the organization, its customers and other interested parties, that the need or expectation under consideration is implied. NOTE 2: A qualifier can be used to denote a specific type of requirement (e.g. product requirement, quality management requirement, customer requirement). NOTE 3: A specified requirement is one which is stated, for example, in a document. NOTE 4: Requirements can be generated by different interested parties.
Responsibility	Being obliged to answer, as for one's actions, to an authority that may impose a penalty for failure. The ability to respond to meet an obligation.

Term	Definition
Review	Activity undertaken to determine the suitability, adequacy and effectiveness of the subject matter to achieve established objectives.
RL	Reporting Limit, minimum value set by the laboratory that should be greater than the calculated PQL but must be greater than the calculated MDL.
Root Cause	A fundamental deficiency that results in nonconformity and must be corrected to prevent recurrence of the same or similar nonconformity.
RSD (%)	$\frac{Standard\ Deviation}{Average}(100)$
SOP	Standard Operating Procedures
SPCC	System Performance Check Compounds
Specification	A document stating requirements.
Standard	An acknowledged measure of comparison for quantitative or qualitative value.
Standard Deviation	Measure of the dispersion of a series of results around their average (measured as n-1) $s = \frac{\sum (x - x)^2}{n - 1}$
Subcontractor	An organization that provides a product or service to the supplier.
Summary Protocol	An internal document that summarizes the method used.
Supplier	Organization or person that provides a product (e.g. producer, distributor, retailer or vendor of a product, or provider of a service or information). NOTE 1: A supplier can be internal or external to the organization. NOTE 2: In a contractual situation a supplier is sometimes called "contractor".
Surrogate	Organic compound that is similar to analytes of interest in chemical composition, extraction, and chromatography but is not normally found in environmental samples. It is spiked into all blanks, standards, samples and spike samples prior to preparation and analysis. Percent recoveries are calculated for each surrogate.
SW-846	EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

Term	**Definition**
System	Set of interrelated or interacting elements.
Technical Expert	Person who provides specific knowledge of or expertise on the subject to be audited. NOTE 1: Specific knowledge or expertise includes knowledge of or expertise on the organization, process or activity to be audited, as well as language or cultural guidance. NOTE 2: A technical expert does not act as an auditor in the audit team.
Technology Audit	An audit of any testing method or technique.
Test	Determination of one or more characteristics according to a procedure.
Testing	A means of determining an item's capability to meet specified requirements by subjecting them to a set of physical, chemical, environmental, or operating actions and conditions.
Top Management	Person or group of people who direct and control an organization at the highest level.
Traceability	Ability to trace the history, application or location of that which is under consideration. NOTE 1: When considering product, traceability can relate to - the origin of materials and parts - the processing history - the distribution and location of the product after delivery
Trip Blank	Reagent grade water (liquids) or methanol (solids) in a sample bottle that accompanies sample bottle(s) from the lab, to the field, and back to the lab.
Validation	Confirmation, through the provision of objective evidence, that the requirements for a specific intended use or application have been fulfilled. The term "validated" is used to designate the corresponding status. NOTE 1: The use condition for validation can be real or simulated.

Term	**Definition**
Verification	Confirmation, through the provision of objective evidence, that specified requirements have been fulfilled. The term "verified" is used to designate the corresponding status. NOTE 1: Confirmation can comprise activities such as - performing alternative calculations - comparing a new design specification with a similar proven design specification - undertaking tests and demonstrations - reviewing documents prior to issue
Work Environment	Set of conditions under which work is performed. NOTE: Conditions include physical, social, psychological and environmental factors (such as temperature, recognition schemes, ergonomics and atmospheric composition).
ZHE	Zero Headspace Extractor

END OF QA MANUAL

Quality Assurance Manual

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SECTION 2

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REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

REFERENCED LABORATORY SOPs

SOP Reference	Title
WS-PEHS-001	Respiratory Protection Plan
WS-PM-0003	Program Setup and Distillation
WS-PQA-0011	Manual Integration Documentation Procedures
WS-PQA-003	Quality Control Program
WS-PQA-012	Technical Data Review Requirements
WS-PQA-013	Procedures to Address Customer Complaints
WS-QA-0003	Sample Receipt and Procedures
WS-QA-0004	Maintenance and Calibration Check of Fixed and Adjustable Volume Autopipettors, Autodispensers and Volumetric Containers
WS-QA-0005	Temperature Monitoring and Corrective Actions for Refrigerators and Freezers
WS-QA-0006	Method Detection Limits (MDL) and Instrument Detection Limits (IDL)
WS-QA-0016	Thermometer Calibration
WS-QA-0017	Standards and Reagents Preparation and Quality Control Check Procedure [Quality Assurance Procedure]
WS-QA-0018	Subsampling and Compositing of Samples
WS-QA-0021	Preparation and Management of Standard Operating Procedures
WS-QA-0022	Employee Orientation and Training
WS-QA-0023	Nonconformance and Corrective Action System
WS-QA-0028	Multi-Incremental Subsampling of Soils and Sediments
WS-QA-0050	Management of Change
WS-QA-0009	Data Scanning
WS-QA-0028	Multi-Incremental Subsampling of Soils and Sediments

SOP Reference	Title
WS-QA-0041	Calibration and Calibration Check of Balances

SECTION 3 INTRODUCTION *(NELAC 5.1 - 5.3)*

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica West Sacramento's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (2005). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Statement of Work for Inorganics & Organics Analysis, SOM, DLM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th and 21st Edition.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 4.1, April 2009.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 4.0.02, May 2006.
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management

policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

This manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our SOP "Preparation and Management of Standard Operating Procedures" (refer to SOP No. WS-QA-0021).

SECTION 4 ORGANIZATION AND MANAGEMENT *(NELAC 5.4.1)*

4.1 OVERVIEW

TestAmerica West Sacramento is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica West Sacramento is presented in Figure 4-1.

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's West Sacramento laboratory.

4.2.2 Laboratory Director / Technical Director

TestAmerica West Sacramento's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

4.2.3 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025. (where applicable)

- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.
- Evaluation of the thoroughness and effectiveness of training.

- Compliance with ISO 17025. (where applicable)

4.2.4 Operations Manager/Technical Director

The Operations Manager has the responsibility for the day to day operations of the analytical staff within the laboratory. **The Operations Manager is responsible for compliance with the ISO 17025 Standard.** The Operations Manager reports directly to the Laboratory Director. The Operations Manager schedules analytical operations, ensures that the laboratory meets quality requirements, investigates technical issues as they arise, and performs other tasks as required by the NELAC standards.

4.2.5 Manager of Customer Services

The Manager of Customer Services has the responsibility for the day to day operations of the client services staff, which includes the Project Management and other administrative groups within the laboratory. The Manager of Customer Services reports directly to the Laboratory Director. The Manager of Customer Services has signature authority for contracts for laboratory services (as detailed in TestAmerica policy), and for laboratory reports.

4.2.6 Project Manager

Project Managers are a liaison between the laboratory's clients and the analytical staff. They report directly to the Manager of Customer Service. The Project Managers have signature authority for final reports, and review project data packages for completeness and compliance with client needs and quality requirements.

4.2.7 Project Administrator

Project Administrators are a liaison between the laboratory's clients and the analytical staff. They report directly to the Manager of Customer Service. The Project Administrators review project data packages for completeness and compliance with client needs and quality requirements.

4.2.8 Department Manager, Team Leader, or Supervisor

Department Managers report directly to the Operations Manager. They supervise the daily activities of analysis with a given laboratory area, and either oversee the review and approval, or perform the review and approval of all analytical data within that area.

4.2.9 Analyst

Analysts report to their respective Department Managers. They perform sample analyses and generate analytical data in accordance with documented procedures.

4.2.10 Sample Custodian

The Sample Custodian ensures the implementation of proper sample receipt procedures, including maintaining chain-of-custody. The Sample Custodian logs samples into the LIMS and ensures that all samples are stored appropriately.

4.2.11 Report Production Staff

The Report Production Staff accurately generates and compiles analytical reports and the associated deliverables as required by the client.

4.2.12 Quality Assurance Staff

The QA Staff has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

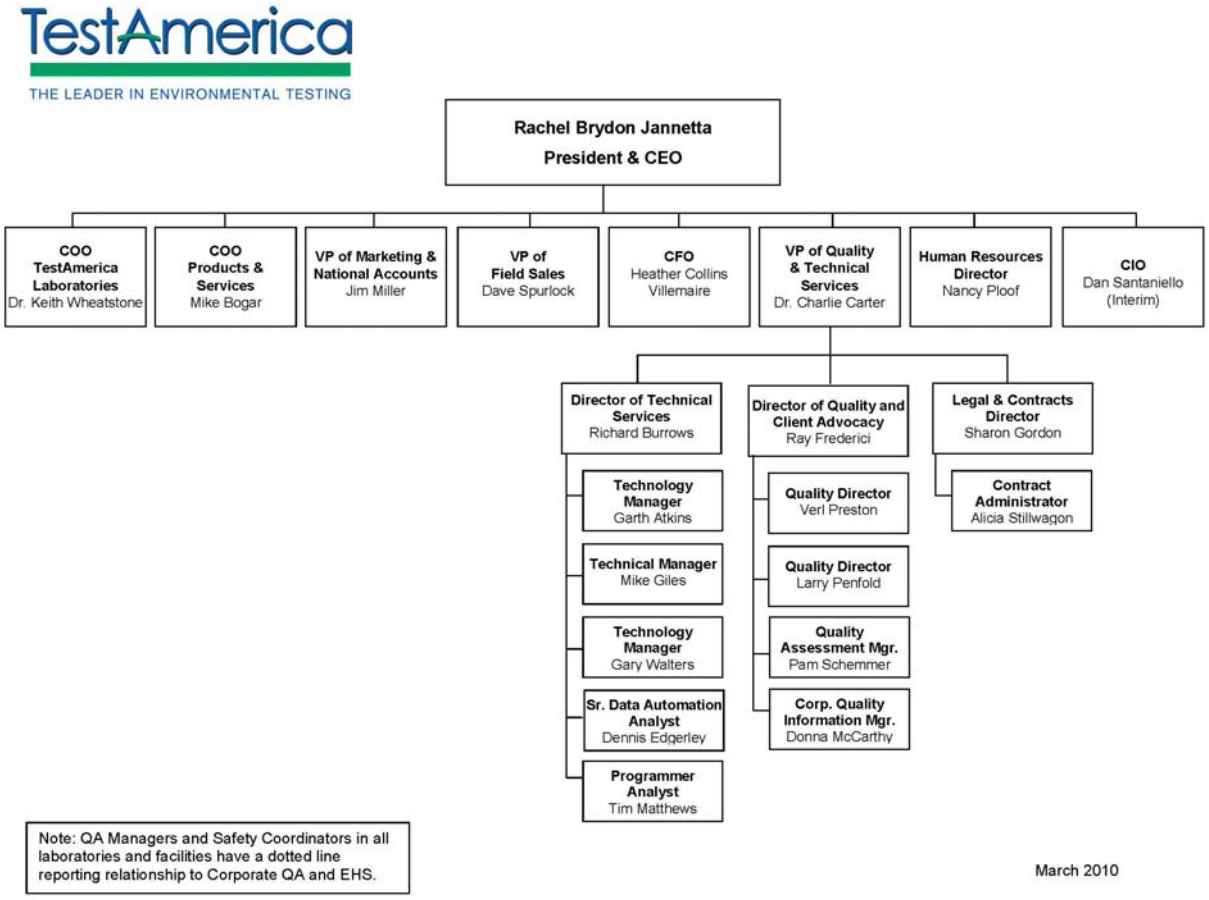
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying the QA manager of deficiencies in the quality system and ensuring corrective action is taken.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025. (where applicable)

4.3 DEPUTIES

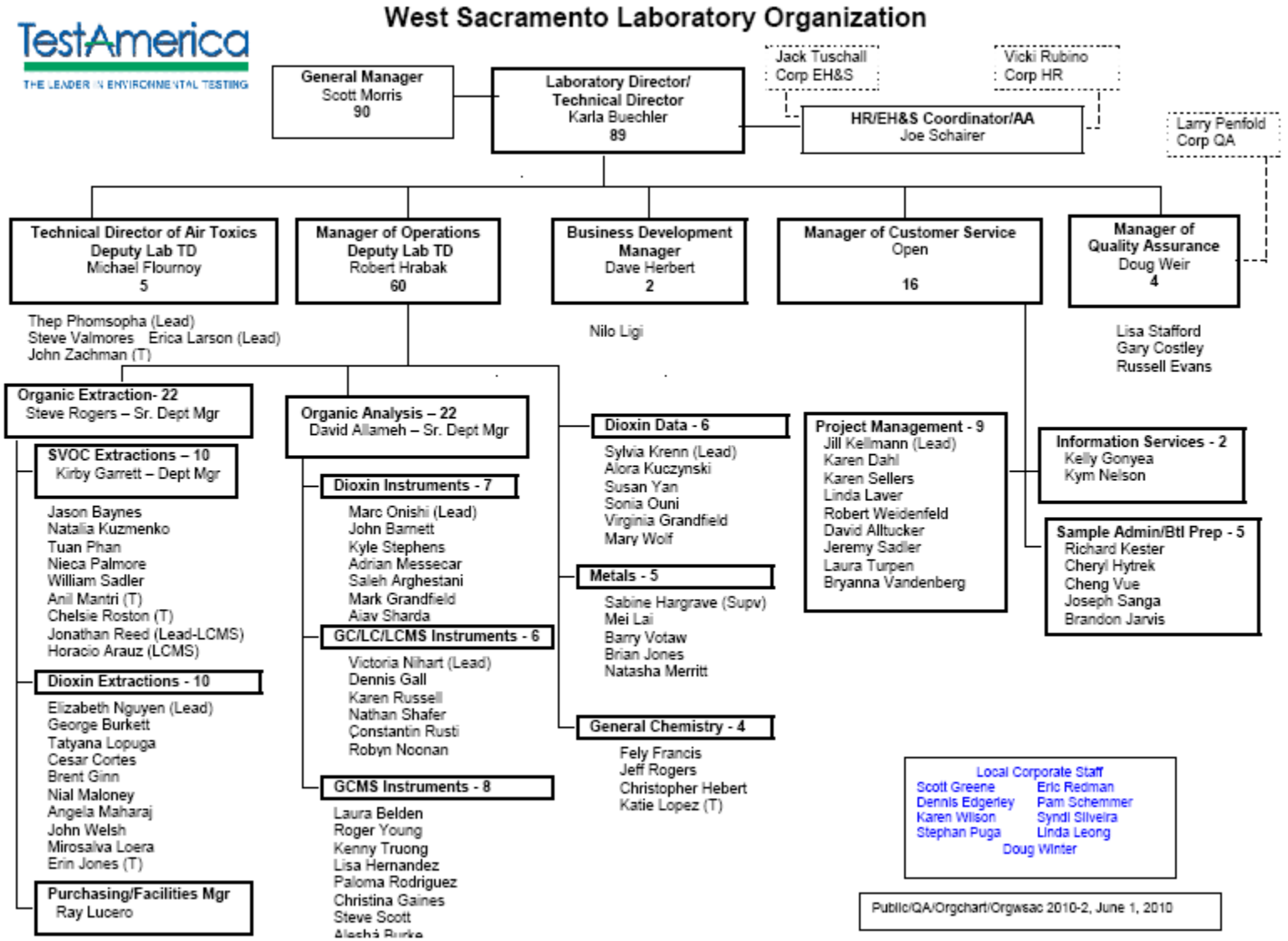
The following table defines who assumes the responsibilities of key personnel in their absence:

Title	Key Personnel	Deputy
Laboratory Director	Karla Buechler	David Herbert
QA Manager	Douglas Weir	Lisa Stafford
Technical Director	Karla Buechler	Michael Flournoy Robert Hrabak
Operations Manager	Robert Hrabak	David Allameh
Customer Services Manager	David Herbert	Jilll Kellmann
Business Development Manager	David Herbert	Michael Flournoy
EHS Coordinator	Joseph Schairer	Richard Kester

Figure 4-1. Corporate and Laboratory Organization Chart



West Sacramento Laboratory Organization



SECTION 5 QUALITY SYSTEM *(NELAC 5.4.2)*

5.1 QUALITY POLICY STATEMENT

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- To comply with the ISO/IEC 17025:2005 International Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CA-L-P-001) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CA-L-S-001.)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CA-L-S-001).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.

- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Corporate Quality Policy Memorandums
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory's has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be

documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains a Reference Data Summary from the LIMS that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an

effective date, is updated each time new limits are generated and is managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in "Quality Control Program" Policy WS-PQA-003 and Section 24.

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Director and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in "Quality Control Program" Policy WS-PQA-003 and Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. Control charts are generated according to laboratory SOP No. WS-PQA-003, "Quality Control Program".

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6 DOCUMENT CONTROL *(NELAC 5.4.3)*

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain the official document on file. The official document is provided to all applicable operational units (may include electronic access). Controlled documents are

identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder for the applicable revision, and are accessible using the laboratory's Intranet.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP and SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures". The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized in the QA office. Electronic versions are kept on a hard drive in the QA department. The procedure for the care of these documents is in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

SECTION 7

SERVICE TO THE CLIENT *(NELAC 5.4.7)*

7.1 OVERVIEW

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has all required certifications, and can meet the clients' data quality and reporting requirements. The PM will also get approval by the Laboratory Director to commit to delivery schedules that are shorter than the published standard TATs. The Laboratory Director updates these TATs on a routine basis, and it is the responsibility of CSMs and PMs to review them prior to making commitments for the laboratory.

It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

If the project is an air, drinking water, or high resolution opportunity, a message describing the opportunity will be immediately sent to the appropriate specialty market distribution list.

New opportunities with an estimated value greater than $100K are passed to the laboratory CSM or BDM, and a message regarding the project details is immediately forwarded to the Large Opportunity Tracking (LOT) distribution list. Specialty market distribution will be included in this notification as appropriate, as well as the associated sales person.

The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below.)

- Legal & Contracts Director
- General Manager
- The Customer Service Manager
- The Business Development Manager
- Laboratory and/or Corporate Technical Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors

- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts**,** as does the local Business Development Manager.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Contract negotiations and finalization is the responsibility of the Business Development Manager. These records are archived by client and project in a restricted network folder accessible to laboratory department managers, project managers, and senior managers.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. Each Laboratory Project Manager keeps a phone log of conversations with the client. In addition, all conversations involving notification of important information, or actions directed by the client are documented with a follow up e-mail and archived in the contracts folder or the SDG documentation and case narrative. Instances include change in scope, alterations to the requests listed on a chain of custody, directions to proceed in the event of a non-conformance, and any other conversation that changes the direction of a COC or contract.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the

supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are updated to the QAS and introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 SPECIAL SERVICES

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Any member of senior staff or technical experts is available to discuss any technical questions or concerns that the client may have.

7.6 REPORTING

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8 SUBCONTRACTING OF TESTS *(NELAC 5.4.5)*

8.1 OVERVIEW

For the purpose of this quality manual, the phrase “subcontract laboratory” refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients due to project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOPs on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). 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. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work. Documentation of approval is stored electronically in the quote folder within SACSALES share on a local laboratory server.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories and supporting documentation is available on the

TestAmerica intranet site. Verify necessary accreditation, where applicable, (e.g., on the subcontractors NELAC, A2LA accreditation or State Certification).

- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, PMs or CSMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director/Manager. The Laboratory Director/Manager requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.

- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers and Sales Personnel.

8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or CSM) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it is current and scope-inclusive. The information is stored electronically in the quote folder within SACSALES share on a local laboratory server.
For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. Results submitted by a network work-sharing laboratory on the same LIMS will be designated in the case narrative.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, the QA Manager will be required to verify certifications. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1. Example: Work Sharing Agreement

TestAmerica	Work Sharing Agreement (Include with every sample shipment)		Exporting Lab	TAL Chicago
			Project Name	
Import Lab Information			**Export Lab Information**	
Lab Name	TAL Chicago		PM Contact Name	
PM Contact Name			Backup Contact Name	
Backup Contact Name			Agreement Date	
Pricing Information			**Project Information**	
QA/QC (i.e. MS/MSD) Billable?	No		Client Company Name	
Raw Data Surcharge		%	Date First Samples to Arrive	
EDD Surcharge		%	Est. Duration of Sampling Event	
TAT Surcharges		%		
Penalty Terms	None			
Other Charges Not in Unit Price? (i.e. canisters, Level IV/III , shipping, bottles)	None		Quote or Contract Reference ID	
Project Details				
Non-Standard Work Product	No			
Quality Assurance Plan	No			
List Certifications Required				
Analyte/Cmpd. List with RLs Attached	No			
Results Dry-Weight Corrected	No			
Special Method Holding Times	None			
Internal Chain of Custody Required	No			
Known Hazards/High Analyte Level	No			
Saturday/Special Delivery Options	None			
Special Instructions	None			
Reporting Limit Value Convention	Report to MDL with "J" Values up to		"Non-Detect" Presentation Convention	U

Deliverable Requirements		Transmittal medium	Format Column	Date Due to Export Lab	**Import and Export Lab Agreement**
Preliminary Report:	No				*Import lab must acknowledge*
Final Report:	No				receipt of Agreement and samples
EDD:	No		Type Format Name		*via E-Mail*
Total Access Reporting	No	NA	NA		
Custom Forms:	No	NA	Type Format Name		
"Other" Deliverable Definitions or Notes					

Analysis	Method	Matrix	# of Samples	Import Lab's Unit Price	Unit Price w/Surcharges	Extended Price
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					Approximate Total Project Value	$ -

Work Instruction No. CA-WI-010/B-10/08

SECTION 9 PURCHASING SERVICES AND SUPPLIES *(NELAC 5.4.6)*

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Many of the items used routinely are pre-qualified and placed into the on-site consignment system.

For items not available from the consignment system, or items that are not used routinely, an order in placed in the JDE ordering system. Only personnel trained in the ordering program JDE may place orders using the program. All relevant information, including quantity, must be entered. Only approved vendors may be used. A vendor must be approved by corporate to be

on the approved vendor list in JDE. The Laboratory Director or designee approves all orders placed in JDE.

9.3.2 Receiving

It is the responsibility of the facilities manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOP's expiration date unless 'verified'. See laboratory SOP No. WS-QA-0017, "Standards and Reagent Preparation and Quality Control Check Procedures", for standard verification procedures.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning every other day. The minimum total pressure must be 250 psig for the automatic bank of gas tanks before the system switches to the next bank of tanks. No individual compressed gas tanks are used at the instrument benches at the laboratory. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- mmho/cm (or specific resistivity of greater than 1.0 megaohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Department Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard. See laboratory SOP No. WS-QA-0017, "Standards and Reagent Preparation and Quality Control Check Procedures", for standard QC procedures.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. Certificates of analysis are also scanned and stored electronically. These records include date of receipt, lot number (when applicable), and expiration date (when applicable).

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Director and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench and inventoried in the master document list.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers/Technical Director.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technology Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10 COMPLAINTS *(NELAC 5.4.8)*

10.1 OVERVIEW

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following laboratory policy WS-PQA-013 "Procedure to Address Customer Complaints".

10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to laboratory policy WS-PQA-013 "Procedure to Address Customer Complaints".

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11 CONTROL OF NON-CONFORMING WORK *(NELAC 5.4.9)*

11.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the Department Manager for resolution. The Department Manager may elect to discuss it with the Operations Manager or QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non-NELAC state would need to note the change made to how the method is normally run.

11.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CA-L-S-001) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director/Manager, a Department Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This

information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, the Operations Manager, the Manager of Project Management, and the Business Development Manager. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director/Manager, QA Manager, ECOs, Corporate Quality, the COO, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CA-L-S-001) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CA-L-S-001.

11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc…). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Supervisor) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12 CORRECTIVE ACTION *(NELAC 5.4.10)*

12.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) (refer to Figure 12-1).

12.2 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Isolated Reporting / Calculation Errors
- Failed or Unacceptable PT results.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors

12.2.3 Non-Conformance Report (NCR) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.4 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCRs.
- Issues found while reviewing NCRs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Health and Safety violations.

12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Laboratory SOP No. WS-QA-0023, “Nonconformance and Corrective Action System”, provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Department Manager or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.

- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Department Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and reviewed to ensure that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or CAR.

Laboratory SOP No. WS-QA-0023, "Nonconformance and Corrective Action System" includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Laboratory SOP No. WS-QA-0023, "Nonconformance and Corrective Action System" provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The SOP also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

12.5 BASIC CORRECTIONS

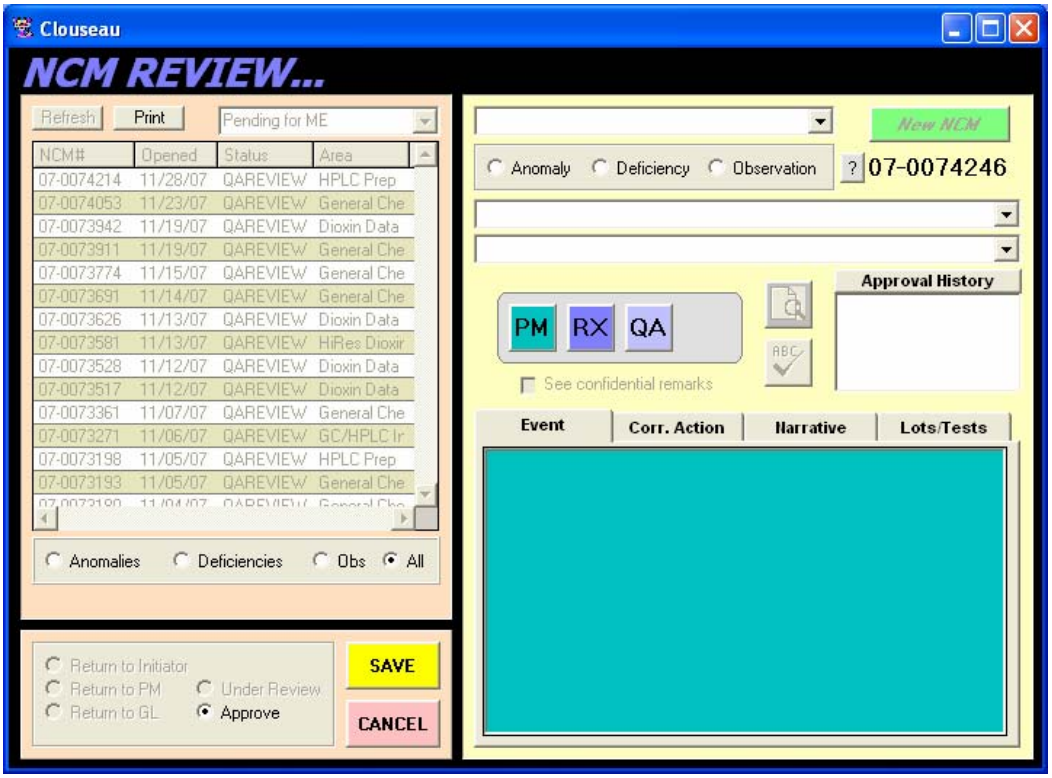
When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

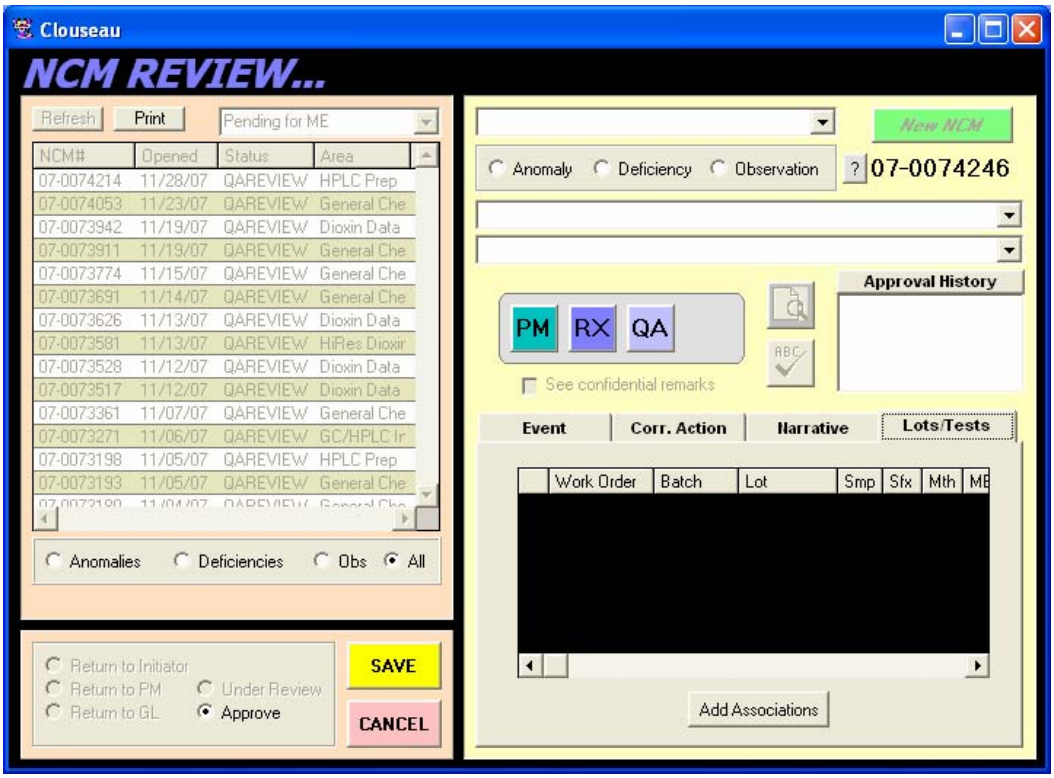
This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Example: Non Conformance Memo

Example Screens:




Clouseau
NCM REVIEW...
Refresh
Print
Pending for ME
NCM# Opened Status Area
07-0074214 11/28/07 QAREVIEW HPLC Prep
07-0074053 11/23/07 QAREVIEW General Che
07-0073942 11/19/07 QAREVIEW Dioxin Data
07-0073911 11/19/07 QAREVIEW General Che
07-0073774 11/15/07 QAREVIEW General Che
07-0073691 11/14/07 QAREVIEW General Che
07-0073626 11/13/07 QAREVIEW Dioxin Data
07-0073581 11/13/07 QAREVIEW HiRes Dioxir
07-0073528 11/12/07 QAREVIEW Dioxin Data
07-0073517 11/12/07 QAREVIEW Dioxin Data
07-0073361 11/07/07 QAREVIEW General Che
07-0073271 11/06/07 QAREVIEW GC/HPLC Ir
07-0073198 11/05/07 QAREVIEW HPLC Prep
07-0073193 11/05/07 QAREVIEW General Che
Anomalies Deficiencies Obs All
Return to Initiator
Return to PM
Return to GL
Under Review
Approve
SAVE
CANCEL
New NCM
Anomaly Deficiency Observation
07-0074246
PM RX QA
See confidential remarks
Approval History
Event Corr. Action Narrative Lots/Tests
Work Order Batch Lot Smp Sfx Mth
Add Associations

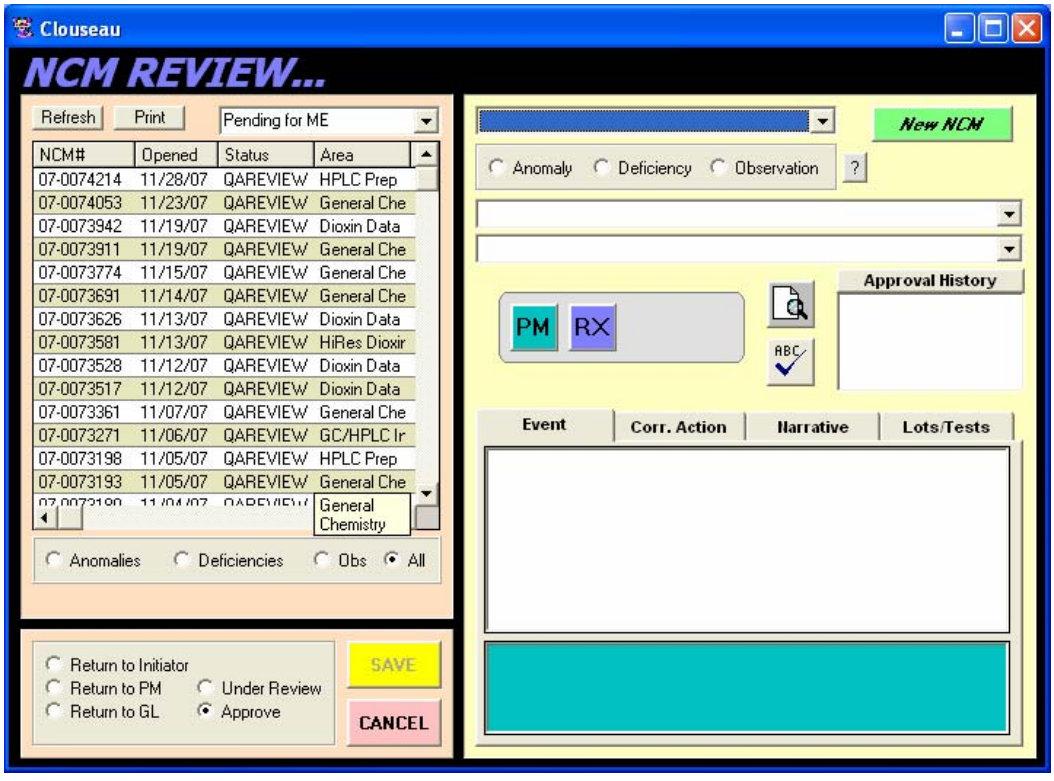

Clouseau
NCM REVIEW...
Refresh
Print
Pending for ME
NCM# Opened Status Area
07-0074214 11/28/07 QAREVIEW HPLC Prep
07-0074053 11/23/07 QAREVIEW General Che
07-0073942 11/19/07 QAREVIEW Dioxin Data
07-0073911 11/19/07 QAREVIEW General Che
07-0073774 11/15/07 QAREVIEW General Che
07-0073691 11/14/07 QAREVIEW General Che
07-0073626 11/13/07 QAREVIEW Dioxin Data
07-0073581 11/13/07 QAREVIEW HiRes Dioxir
07-0073528 11/12/07 QAREVIEW Dioxin Data
07-0073517 11/12/07 QAREVIEW Dioxin Data
07-0073361 11/07/07 QAREVIEW General Che
07-0073271 11/06/07 QAREVIEW GC/HPLC Ir
07-0073198 11/05/07 QAREVIEW HPLC Prep
07-0073193 11/05/07 QAREVIEW General Che
General Chemistry
Anomalies Deficiencies Obs All
Return to Initiator
Return to PM
Return to GL
Under Review
Approve
SAVE
CANCEL
New NCM
Anomaly Deficiency Observation
PM RX
Approval History
Event Corr. Action Narrative Lots/Tests

Table 12-1.

Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Supervisor)*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Supervisor)*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in ***(state where limits are maintained)***.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in ***(state where limits are maintained).***	- Batch must be re-prepared and re-analyzed. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Proficiency Testing (PT) Samples *(QA Manager, Department Manager/Supervisor)*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Department Manager/Supervisor, Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc...	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors *(Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager/ Supervisor, QA Manager, Corporate QA, Corporate Management)*	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) *(QA Manager, Lab Director/Manager, Department Supervisors/Managers)*	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation *(Safety Officer, Lab Director/Manager, Department Supervisor/Manager)*	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:

1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants as defined in policy WS-QA-0003. These

include: methylene chloride, toluene, acetone, 2-butanone, phthalates and OCDD. This is contingent on whether these contaminants appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

SECTION 13 PREVENTIVE ACTION *(NELAC 5.4.11)*

13.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc.

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 16). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these various tracking indicators, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of indicators monitored under this collective system include:

- SOP Tracking
    - Current Revisions w/ Effective Dates
    - Required Annual/Biennial Revisions w/ Due Date
- Proficiency Testing (PT) Sample Tracking
    - Pass / Fail – most current 2 out of 3 studies.
- Instrument / Equipment List
    - Current / Location
- Accreditations
    - New / Expiring
- Method Capabilities
    - Current Listing by program (e.g., Potable Water, Soils, etc.)
- Key Personnel
    - Technical Managers, Department Supervisors, etc...

These items are maintained on TestAmerica's Intranet (Proposal Library) or on our internal database (TotalAccess) which uploads to our company internet site.

SECTION 14 CONTROL OF RECORDS *(NELAC 5.4.12)*

The laboratory maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department in a database, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Department Managers.

Table 14-1. Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals	5 Years from document retirement date*
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years

	Record Types [1]:	Retention Time:
	EH&S Manual, Permits, Disposal Records	7 years
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or the Iron Mountain data storage facility that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records before 09/09/2009 were maintained on-site at the laboratory for at least 1 month after their generation and moved offsite for the remainder of the required storage time. Records generated after 09/09/2009 are maintained on-site at the laboratory for at least 1 month after their generation and then scanned into PDF files. The electronic files are stored on-site and backed up daily offsite. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 and WS-PQA-017 for more information.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. Refer to SOP WS-QA-0009, "Document Archiving". Instrument data is stored by project, except for inorganics and calibration data. Inorganics and calibration data is stored sequentially by instrument as appropriate. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP WS-QA-0009.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

14.2 TECHNICAL AND ANALYTICAL RECORDS

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;

- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet or in the LIMs.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;

- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in a logbook or using the Veritas Electronic Standards Logbook**.** Records are considered archived when noted as such in the records management system.

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in

cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a “Certificate of Destruction” is required.

SECTION 15 AUDITS *(NELAC 5.4.13)*

15.1 INTERNAL AUDITS

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Audits, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are shown in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits - Evaluate raw data versus final reports - Analyst integrity - Data authenticity	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Technical Director	- All SOPs within a 2-year period - All new analysts or new analyst/methods within 3 months of IDOC
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given area. The area audits may be done on a rotating schedule throughout the year to ensure

adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, MintMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Director at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Soil, Water Supply, Water Pollution, Air, and round-robin studies for sediments and biological materials.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. A copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined 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." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

15.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16 MANAGEMENT REVIEWS *(NELAC 5.4.14)*

16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Directors, Operation Manager, laboratory senior management, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Directors, Operations Manager, Customer Service Manager, Business Development Manager, and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining quality goals & objectives. Corporate Operations and Corporate QA personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:

- Adequacy of staff, equipment and facility resources.
- Adequacy of policies and procedures.
- Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

SECTION 17 PERSONNEL *(NELAC 5.5.2)*

17.1 OVERVIEW

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 TRAINING

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics is maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP WS-QA-0022, “Employee Orientation and Training”.

17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CA-L-P-001) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS *(NELAC 5.5.3)*

18.1 OVERVIEW

The laboratory is a 66,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, and temperature in the laboratory. In the event of a power outage, the laboratory can be equipped with a back up power supply for sample storage, as detailed in SOP No. WS-QA-0005, “Temperature Monitoring and Corrective Action for Refrigerators and Freezers”.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

18.5 BUILDING SECURITY

Building keys and alarm codes are distributed to employees as necessary.

Employees wear photographic identification name cards while on the premises.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19 TEST METHODS AND METHOD VALIDATION *(NELAC 5.5.4)*

19.1 OVERVIEW

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 STANDARD OPERATING PROCEDURES (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP WS-QA-0021 (Preparation and Management of Standard Operating Procedures).
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics & Organics Analysis, SOM, DLM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*

- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Underground Storage Tanks Procedures Manual, State of Alaska Department of Environmental Conservation, Division of Spill Prevention and Response Contaminated Sites Program, November 7, 2002*
- *Tri-Regional Board Staff Recommendations for Preliminary Investigation and Evaluation of Underground Tank Sites, North Coast Regional Water Quality Control Board, San Francisco Bay Regional Water Quality Control Board and Central Valley Regional Water Quality Control Board, August 10, 1990*
- *Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997*
- *Compendium of Methods for the Determination of Air Pollutants in Indoor Air, (EPA 600/4-90-10, April 1990)*
- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, (EPA 625/R-96/010a, June 1999*
- *Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources, Stationary Source Test Methods, Volume 3, California Air Resources Board*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

19.4.2.1 A demonstration of capability (DOC, Lab SOP WS-QA-0022) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel.

19.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

19.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to SOP WS-QA-0022 for an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 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 from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For DoD QSM 4.1 projects the QL is referred to as the LOQ. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 19.7.10). Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. Alternatively, the MDL may be determined using a series (ideally 50-100) of method blanks for “uncensored” methods which always return a signal (i.e., ICP).

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory’s SOP No. WS-QA-0006, for details on the laboratory’s MDL process.

19.8 INSTRUMENT DETECTION LIMITS (IDL)

19.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

19.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

19.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL. The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. The analytes must be qualitatively identified or see SOP No. WS-QA-0006 for other options. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

For DoD ELAP certified methods: Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP WS-QA-0006 Method Detection Limits (MDLs/DLs) for further details.

19.9.2 When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

For DoD ELAP certified methods: The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for

specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent

recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 SAMPLE REANALYSIS GUIDELINES

Because there is a certain level of uncertainty with any analytical measurement, a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Manager *or* Laboratory Director if unsure.

19.14 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP Nos. S-ITQ-005, "QuantIMS/JDE user Profile Setup and Maintenance", and S-ITQ-007, "Software Testing, Validation and Verification. The laboratory is currently running the QuantIMS which is a custom in-house developed LIMS system that has

been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes DB2 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 **Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

19.14.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 **Maintain Confidentiality:** Ensure data confidentiality through physical access controls when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The data review checklists are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and WS-PQA-0011.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.

19.14.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are outlined in several SOPs (WS-PQA-003, "Quality Control Program", WS-PQA-012, "Technical Data Review Requirements", WS-PM-0004, "Final Report Assembly and Third Level Data Review") to ensure that reported data are free from calculation and transcription errors, and that QC parameters have been reviewed and evaluated before data is

reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (WS-PQA-0011, "Manual Integration Documentation and Practices"). The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

19.14.4.2 The next level of data review occurs with the analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analysts transfer the data into the LIMS and add data qualifiers if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. One hundred percent of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.14.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Operations Manager, or Department Manager for further investigation. Corrective action is initiated whenever necessary.

19.14.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP No. WS-PQA-0011, "Manual Integration Documentation and Practices".

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration

performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1
Example: Demonstration of Capability Documentation

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Demonstration of Capability Certification Statement

TestAmerica West Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
(916) 373-5600

Date:
Method:
Matrix: Aqueous
SOP:

Analyst(s):

We, the undersigned, CERTIFY that:

1: The analyst(s) identified above, using the cited test method, with the specifications in the cited SOP, which is in use at the facility for the analysis of samples under the TestAmerica West Sacramento Quality Assurance Manual, has met the Demonstration of Capability.

2: The test method was performed by the analyst(s) identified on this certification following the TestAmerica West Sacramento SOP.

3: A copy of the laboratory-specific SOP is available for all personnel on-site.

4: The data associated with the demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.

5: All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/ Observations:

Karla Buechler
Technical Director _____ Technical Director Signature _____ Date

Douglas Weir
QA Manager _____ QA Manager Signature _____ Date

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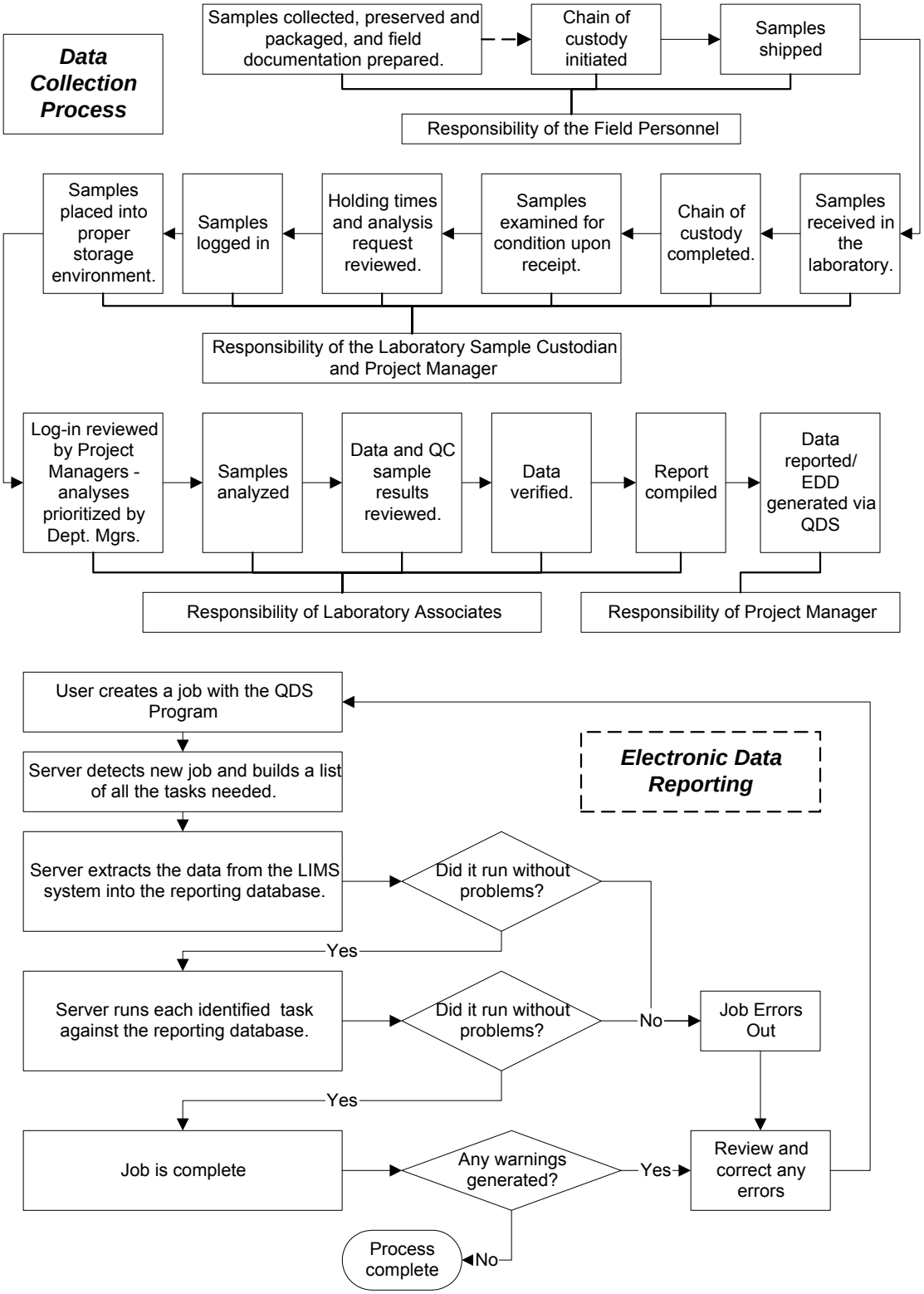
Figure 19-2
Example: Work Flow



SECTION 20 EQUIPMENT (AND CALIBRATIONS) *(NELAC 5.5.5)*

20.1 OVERVIEW

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 PREVENTIVE MAINTENANCE

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. See SOP No. WS-QA-0041, "Calibration and Calibration Check of Balances" for more details.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of at least 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP No. WS-QA-0016, "Thermometer Calibration."

20.3.4 Refrigerators/Freezer Units, Waterbaths, and Ovens

The temperatures of all refrigerator units and freezers used for sample storage are monitored 7 days a week; and each working day for units used for standard storage.

Ovens and waterbaths are monitored on days of use. Drying oven temperature must be recorded before and at the end of use. For example, an oven used for moisture determination must have its temperature recorded at the start and end of the drying process. Temperature must be ± 5% of set temperature for DoD work.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements. See SOP WS-QA-0004, “Maintenance and Calibration Check of Fixed and Adjustable Volume Autopipettors, Autodispensers and Volumetric Containers”.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy. Glass micro-syringes are marked with a unique identification number and are calibrated.

20.3.6 Autoclaves

Autoclaves used for sample digestion are capable of maintaining conditions of 15 psi at 120ºC for 15 minutes. The temperature of the autoclave is verified quarterly.

20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day. Further details regarding the calculations involved are present in SOP No. CA-Q-S-005, “Calibration Curves (General).”

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Average RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually, however, the annual requirement does not apply to Isotope Dilution methods.

20.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration

factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i. e., RPD, per NELAC (2003) Standard, Section 5.5.5.10.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used. Detailed calculations for each fitting method can be found in CA-T-P-002.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and

analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are 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 calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

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 purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Laboratory Equipment and Instrumentation

This table is updated by QA annually at a minimum.

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put Into Service	Condition When Received
GC	Agilent	6890N	CN10543080	2005	New
	Hewlett-Packard	6890	US00001087	1997	New
	Hewlett-Packard	6890	US00006442	1997	New
	Hewlett-Packard	6890	US00006441	1997	New
	Hewlett-Packard	6890	US00006438	1997	New
	Agilent	6890N	CN10521082	2005	New
	Hewlett-Packard	6890	US00000311	1997	Used
	Hewlett-Packard	6890	US00006455	1997	New
	Agilent	6890N	CN10521015	2005	New
HPLC	Varian	ProstarV 9065-01384	000498	1998	New
	Waters	2695	B02SM4 915M B02487 788M C02475 452N	2002	New
	Agilent	1100	DE43631861 DE43607107 DE33229050	2005	New
	Agilent	1100	DE43633762 DE43603468 DE43630549	2005	New
	Agilent	1200	DE62961719 JP62360107 DE64762303 DE63055783 DE63064176	2007	New
LCMS	Micromass	Quattro	9319	2000	New
	Micromass	Quattro Premier XE	VAB 452	2006	New
	Agilent	6410A Triple Quad	US64810220	2006	New
	Waters	Quattro Premier XE	VAB 1006	2009	New

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put Into Service	Condition When Received
HIRES	Fisons	VG70	7054	1988	New
	Fisons	VG70	7083	1989	New
	Fisons	Ultima	S176U	1992	New
	Micromass	Ultima	M421	1998	New
	VG Analytical	VG70	US82321724	2001	New
	Micromass	Ultima	M637	2004	New
	Waters	Ultima	M318	2008	Used
	Waters	Ultima Premier	P741	2008	New
METALS	Leeman	PS200 II	HG-8008	1998	New
	Leeman	PS200 II	HA-3027	2004	New
	Perkin-Elmer	Optima 4300DV	077N3022401	2003	New
	Perkin-Elmer	ELAN 6000	51950460	1994	New
	Perkin-Elmer	ELAN 6000	4719801	1998	New
	Perkin-Elmer	ELAN 9000 DRC-e	W0170304	2005	Used
GCMS Semivolatiles	Hewlett-Packard	HP 5973	US80221476	1998	New
	Hewlett-Packard	HP 5973	US80321345	1998	New
	Hewlett-Packard	HP 5973	US80221400	1998	New
	Hewlett-Packard	HP 5975	US61633479	2007	New
	Hewlett-Packard	HP 5973	US00023149	1999	New
	Hewlett-Packard	HP 5973	US00023182	1999	New
	Varian	Saturn 2200	06370 13651 CP8400-6358	2007	New
Volatiles	Hewlett-Packard	HP 5973	US800020780	1998	New
	Hewlett-Packard	HP 5973	US10227041	2002	New
	Hewlett-Packard	HP 5973	US10214090	2002	New
	Hewlett-Packard	HP 5973	US53931405	2005	New

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put Into Service	Condition When Received
General Chemistry	Man-Tech Associates	PC-Titrate	190H0238 2330 MS-0L0-477 MS-0C1-471 MS-0B1-276 MS-0E1-579	2001	New
	OI Corp	Flow System	20850488	2000	New
	Systea	EasyChem Plus	2006E1001205	2006	New
	Mettler-Toledo S/N	MC126 / 225646	225646	2004	New
	Dionex	DX500	99120668	1999	New
	Dionex	ICS-2000	3040054	2003	New
	Dionex	ICS-1000	4010013	2004	New
	Accumet	AB15	AB92321437	2005	New
	Thermo	Genesis 20	3SGH080004	2005	New
	OI Corp	Model 1010 Solids Module	J245710347 C247776181	2003	New
	HF Scientific	Micro 100	402223	2004	New

Table 20-2. Schedule of Routine Maintenance

INSTRUMENT	MAINTENANCE	FREQUENCY
APCI/ESI LC/MS/MS	Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.	As Needed
	Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.	Daily(2)
	Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable.	Semi-Annually
	Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.	Annually
HIGH PRESSURE LIQUID CHROMATOGRAPH(1)	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements. Rinse flow cell with 1N nitric acid if dirty flow cell. Change pump seals when flow becomes inconsistent. Backflush column if applicable. Change in-line filters for solvents.	As Needed
	Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse delivery lines to prevent contamination of the new solvent. Check gas supply if applicable. Flush with an appropriate solvent to remove all bubbles. Pre-filter all samples.	Daily(2)
	Change pump seals.	Every 6-9 Months

INSTRUMENT	MAINTENANCE	FREQUENCY
GAS CHROMATOGRAPH(1)	Replace septum. Clean injector port Cut 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. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturers suggested maintenance schedule Replace fuse. Reactivate external carrier gas dryers. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents. Check inlets, septa.	As Needed
	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily(2)
	ECD: perform wipe test.	Semi-Annually
PURGE AND TRAP SYSTEMS	Change trap. Check purge flow. Flush lines (after foaming sample). Periodic leak checks (when replace traps/spargers) Replace/condition traps and/or spargers (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), and clean or replace glassware/spargers. Bake trap as needed to correct for high background. Change trap whenever loss of sensitivity, or erratic response or failing resolution is observed. Purge & trap autosamplers: leak check system, clean sample lines, valves.	As Needed
	Bake out trap & analyze primers (as needed) prior to commencing analysis.	Daily(2)
GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETER(1)	Replace septum. Clean injector port. Cut 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. Replace injection port liner when front portion of capillary column is removed. Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed. Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	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. Replace filaments when both filaments burn out or performance indicates need for replacement. Check mass calibration (PFTBA or FC-43). Check ion source and analyzer (clean, replace parts as needed). Check vacuum, relays, gas pressures and flows. Change oil in the mechanical rough pump. Relubricate the turbomolecular pump-bearing wick. HP 7673 Autosampler: Replace syringe.	
	Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. Autosampler: fill wash bottle, dispose of waste bottle contents.	Daily(2)
	Replace the exhaust filters on the mechanical rough pump every 1-2 years.	Annually
GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETER(1)	Full Bake-Out. Change oil in rotary pump. Change oil in diffusion pump. Replace o-rings. Solvent rinse the flight tube. Clean the first field free region. Check detector voltages. Clean and dust connectors, etc on the outside of the instrument. Check the vacuum: ~5 x. 10^{-7} MBAR on both analyzer ion gauges, and ~5 x 10^{-6} MBAR on the source, with no helium flowing. Check isolation valve for leaks, correct if needed. Check for thermal trip by taking the magnet to maximum current, and verify that the coolant flow is acceptable. Replace septum. Clean injector port. Cut 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. Replace injection port liner when front portion of capillary column is removed. 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. Replace filaments when performance indicates need for replacement.	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	Check resolution sensitivity. Check stability. Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily(2)
COLD VAPOR ATOMIC ABSORPTION (LEEMAN PS 200) (1)	Change pump tubing. Check/change Hg lamp. Clean optical cell. Change drying tube. Grease pump.	As Needed
	Check sample tip for clogs. Check drying tube. Check pump tubing/drain tubing. Check gas pressure. Check liquid/gas separator. Check tubing.	Daily(2)
INDUCTIVELY COUPLED ARGON PLASMA/MASS SPECTROMETRY (ICAP/MS)(1)	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics. Measure quartz torch for proper alignment when removed and cleaned. Clean spray chamber and nebulizer. Clean all filters and fans. Check chiller coolant level. Check and drain oil mist eliminator on roughing pumps.	As Needed
	Check sample waste container level. Check quartz torch condition. Check RF coil. Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing. Check condition of sampler and skimmer cones. Check oil level of roughing pumps.	Daily(2)
	Replace oil in roughing pumps.	Every 2-3 Months
ICP(1)	Check that argon feed pressure is 80-120 psi. Check that chiller coolant pressure is 45-80 psig, no leaks. Check purge and shear gasses. Nitrogen purge gas pressure 40-120 psig, compressed air shear gas pressure 80-120 psig. Check radial purge and axial windows for deposits. Check that nebulizer is not clogged. Check that capillary tubing is clean and in good condition. Check that peristaltic pump windings are secure. Check that exhaust vent is operational Check that torch, glassware, aerosol injector tube are clean.	Daily(2)
	Clean plasma torch assembly to remove accumulated deposits. Check RF coil. Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance.	Monthly or As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	Clean filters on back of power unit to remove dust. Replace when needed: peristaltic pump tubing. sample capillary tubing. autosampler sipper probe. Check performance with manganese. Check O-rings. Clean/lubricate pump rollers	
	Check chiller coolant filter. (may require more or less frequently)	Semi-Annually
	Notify manufacturer service engineer for scheduled preventive maintenance service.	Annually
ION CHROMATOGRAPH(1)	Clean micromembrane suppressor when decreases in sensitivity are observed. Check fuses when power problems occur. Change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated. De-gas pump head when flow is erratic. Check all air and liquid lines for discoloration and crimping, if indicated. Check/change bed supports guard and analytical columns, if indicated.	As Needed
	Check plumbing/leaks. Check eluent level. Check gases. Check pump pressure. Check conductivity meter.	Daily(2)
	Check pump heads for leaks. Check filter (inlet).	Weekly
	Change pump seals. Change injection valve. Clean conductivity cell. Check conductivity cell for calibration.	Annually
ALPKEM COLORIMETRIC AUTO ANALYZER(1)	Prepare fresh reagents. Replace tubing. (About every 100 hours of use)	As Needed
	Check detector. Make sure there are no trapped bubbles in detector cell. Check Valves Check peristaltic tubing. Check sampler.	Daily(2)
	Clean pump, and XYZ Sampler.	Weekly
	Lubricate pump roller.	Monthly
	Clean pump rollers with steel wool and lubricate.	Semi-Annually
SYSTEA COLORIMETRIC AUTO ANALYZER(1)	Prepare fresh reagents. Replace waste tubing. Replace probes. Replace lamp	As Needed
	Perform washes. Perform filters autozero. Check temperatures.	Daily(2)
CHEMICAL OXYGEN DEMAND (COD) REACTOR(1)	Electronics serviced.	As Needed
	Check temperature with NIST reference thermometer.	Annually

INSTRUMENT	MAINTENANCE	FREQUENCY
AUTO TITRATOR[1]	Electronics serviced.	As Needed
	Calibrate with check standards. Inspect electrodes daily, clean as needed. Inspect electrode proper levels of filling solutions daily, fill as needed. Clean probe, each use. Prime buret Check rinse water reservoir.	Daily[2] (When Used)
CONDUCTANCE METER[1]	Electronics serviced. Replace batteries	As Needed
SPECTROPHOTOMETER[1]	Replace lamp. Replace fuse.	As Needed
	Check instrument manual. Perform wavelength calibration. Replace lamp annually or when erratic response is observed.	Annually
PH METER[1]	Clean electrode. Refill reference electrode.	As Needed
	Inspect electrode. Verify electrodes are properly connected and filled. Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer.	Daily[2]
TOTAL ORGANIC CARBON ANALYZER (OI 1010 AND SOLIDS)	Check injection port septum after 50-200 runs. Perform leak test. Calibrate reagent pumps. Change sample loops. Adjust flow. Indicating drying tube. NDIR zero, after 100 hours of use. Sample pump, after 2000 hours for use. Digestion vessel/condensation chamber. Permeation tube, after 2000 hours of use. NDIR cell, after 2000 hours of use.	As Needed
	Check: Nitrogen supply, (oxygen supply for solids). Persulfate supply (1010 unit). Acid supply (1010 unit). Rinse water reservoir supply (1010 unit). IR millivolts for stability (after 30 min. warm-up).	Daily[2]
TURBIDIMETER[1]	Electronics serviced.	As Needed
	Clean instrument housing.	Monthly
DIGESTION BLOCK	Check temperature with NIST thermometer.	Annually
SONICATOR [1]	Replace probe tip. Disassemble and clean sonicator probe tips. Tune sonicator assembly.	As Needed
	Inspect probe tips for inconsistencies (etching/pitting).	Daily[2] (When Used)
ANALYTICAL/TOP LOADING BALANCES[1]	Check using ASTM Class 3 weights once daily or before use. Clean pan and weighing compartment.	Daily[2]
REFRIGERATORS/WALK-IN COOLERS[1]	Manufacturer cleaning and calibration.	Annually
	Refrigerant system and electronics serviced.	As Needed
	Temperatures checked and logged.	Daily[2]

INSTRUMENT	MAINTENANCE	FREQUENCY
OVENS[1]	Electronics serviced.	As Needed
	Temperatures checked and logged.	Daily[2]
ZYMARK PE WORKSTATION	Change O-rings whenever there are visible leaks or poor sealing on the SPE columns. Sample lines are clean after samples have been extracted by SPE with a program "Clean Sample Lines" with methanol followed by water. Occasionally for a more rigorous cleaning, or after a highly contaminated sample, a mixture of methanol/DCM at 50:50 may be used in place of methanol, follow by methanol, then water (never use acetone). Syringe pump may be primed using a program "Prime Solvent Lines" whenever air bubbles are suspected in the lines from running out of solvents and whenever solvents are changed. Syringe pump in good condition – replace if showing signs of wear or suspected of poor performance. Sample pumps may be re-calibrated whenever major repairs are performed, or whenever the pumps are suspected to be out of calibration. Follow manufacturer's procedure for re-calibrating the sample pumps. For method 8330, the pump loads 1050 mL of sample on the SPE. It should used up the whole sample bottle (quart bottles and 1-L bottles).	As Needed
SONICATION WATER BATH[1]	If the water bath is dirty, empty and refill with tap water. A couple drops of anti-bacterial solution may be added to inhibit the growth of bacteria in the water. The water level in the sonication batch should be about 1.2 to 1 inch from the top while in operation. Do not allow sonication batch to operate with water bath at lower levels. If the level is low, add more water, if the levels is too high, remove water to the proper level.	As Needed

Footnotes to Preventive Maintenance Tables

(1) Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.

(2) Daily checks and verifications are performed prior to instrument startup and are not documented in maintenance logs unless problems are noted.

SECTION 21

MEASUREMENT TRACEABILITY *(NELAC 5.5.6)*

21.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

The laboratory's calibration policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica West Sacramento contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the

calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica West Sacramento does not use a firm who subcontracts the work.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA or NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the ‘true’ value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer’s requirements in order to prevent contamination or deterioration. Refer to the Corporate

Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

21.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. (Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the departments, and online. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP No. WS-QA-0017, "Standards and Reagent Preparation and Quality Control Check Procedures".

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material (for 1613B dioxin/furan analyses the purity must be 98% or corrections must be made).

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database or standards logbook.

- Standard ID
- Description of Standard
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)

- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from the preparation logbook)
- Special Health/Safety warnings if applicable

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22

SAMPLING *(NELAC 5.5.7)*

22.1 OVERVIEW

The laboratory does not provide sampling or in-field testing services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs, such as AFCEE and Alaska Department of Environmental Conservation, which determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located SOP Nos. WS-QA-0018, "Subsampling and Compositing of Samples (Method ASTM D 6323-98**)**" and WS-QA-0028, "Multi-Incremental Subsampling of Soils and Sediments".

Table 22-1.
Holding Times, Preservation and Container Requirements: Drinking Water (SDWA)

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[23]	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Asbestos	Plastic/Glass	4ºC	None	48 hours[5]	1 L
Coliforms (Total and Fecal)	Plastic/Glass[20]	10ºC	$Na_2S_2O_3$	30 hours[21]	120 mL
Cyanide	Plastic/Glass	4ºC	NaOH to pH >12 Ascorbic acid [9] or Sodium arsenite [9]	14 days	500 mL
Fluoride	Plastic/Glass	None	None	None	250 mL
Perchlorate (EPA 331.0)	Plastic/Glass[20]	4ºC	None Filtered, 1/3 Headspace to minimize anaerobic conditions	28 days	250 mL
Heterotrophic Plate Count	Plastic/Glass[20]	10ºC	$Na_2S_2O_3$	8 hours (24 hours[22])	120 mL
Mercury	Plastic/Glass	None	HNO_3 to pH<2	28 days	250 mL
Metals [4]	Plastic/Glass	None	HNO_3 to pH<2 [24]	6 months	250 mL
Nitrate	Plastic/Glass	4ºC	None	48 hours[6]	250 mL
Nitrate-Nitrite	Plastic/Glass	None	H_2SO_4 to pH<2	28 days	250 mL
Nitrite	Plastic/Glass	4ºC	None	48 hours	250 mL
THMs Only	Glass[8]	4ºC	$Na_2S_2O_3$ [9] HCl to pH <2 may also be used	14 days	3 X 40 mL
Volatile Organic Compounds	Glass[8]	4ºC	HCl to pH <2 $Na_2S_2O_3$ [9] or Ascorbic acid [9]	14 days / 24 hrs[25]	3 X 40 mL
EDB, DBCP, 1,2,3-TCP (EPA 504.1)	Glass[8]	4ºC	$Na_2S_2O_3$	14 days[11]	3 X 40 mL
Organochlorine Pesticides/PCBs (EPA 505)[10]	Glass[8]	4ºC	$Na_2S_2O_3$	14 days[11]	3 X 40 mL

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[23]	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Nitrogen and Phos. Pesticides (EPA 507)	Glass-Amber[8]	4ºC	$Na_2S_2O_3$	14 days[12]	1 L
Total PCBs (EPA 508A)	Glass-Amber[8]	4ºC	None	14 days[13]	1 L
Pesticides and PCBs (EPA 508.1)[14]	Glass-Amber[8]	4ºC	HCl to pH <2 $Na_2S_2O_3$[9]	14 days[13]	1 L
Chlorinated Acids (EPA 515.1)	Glass-Amber[8]	4ºC	$Na_2S_2O_3$	14 days[12]	1 L
Nitrosamines (EPA 521)	Glass-Amber[8]	4ºC	$Na_2S_2O_3$	14 days[12]	1 L
Semivolatiles (EPA 525.2)	Glass-Amber[8]	4ºC	HCl to pH <2 $Na_2S_2O_3$[9]	14 days[13]	1 L
N-Methylcarbamoyloxamines and N-Methylcarbamates (EPA 531.1)	Glass[8]	4ºC	$Na_2S_2O_3$, Monochloroacetic Acid buffer to pH<3	28 days	3 X 60 mL
Acetamide Herbicide Degradates (EPA 535)	Glass-Amber[8]	4ºC	Ammonium Chloride	14 days[12]	250 mL
Glyphosate (EPA 547)	Glass[8]	4ºC	$Na_2S_2O_3$	14 days	3 X 60 mL
Endothall (EPA 548)	$Na_2S_2O_3$	4ºC	None	7 days[15]	1 L
Diquat/Parquat (EPA 549.1)	Glass-Amber[8] (Silanized or PVC amber)	4ºC	H_2SO_4 to PH <2 $Na_2S_2O_3$[9]	7 days[16]	1 L
Chlorinated Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides/Herbicides (EPA 551)	Glass[8]	4ºC	Phosphate Buffer and Ammonium Chloride[19]	14 days[17]	3 X 60 mL
Haloacetic Acids (EPA 552.1)	Glass-Amber[8]	4ºC	Ammonium Chloride	28 days[18]	250 mL
2,3, 7, 8 TCDD	Glass-Amber[8]	4ºC	$Na_2S_2O_3$	1 year	1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the

Key to Table

preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. All metals except Hg.
5. Instructions for containers, preservation procedures and holding times as specified in Method 100.2 must be adhered to for all compliance analysis including those conducted with Method 100.1.
6. If the sample is chlorinated, the holding time for an un-acidified sample kept at 4°C is extended to 14 days.
7. Nitrate-Nitrite refers to a measurement of total nitrite.
8. With Teflon lined septum.
9. If chlorinated, add reagent prior to acidification (for Cyanide, add before NaOH).
10. Heptachlor has a 7 day hold time.
11. 14 days until extraction. 24 hours after extraction.
12. 14 days until extraction. 28 days after extraction.
13. 14 days until extraction. 30 days after extraction.
14. For cyanazine, cool to 4°C only.
15. 7 days until derivitization. 1 day after derivitization.
16. 7 days until extraction. 21 days after extraction.
17. 14 days until extraction. 14 days after extraction.
18. 28 days until extraction. 48 hours after extraction.
19. Sodium Sulfite may be used as a dechlorinating agent in some instances. Verify with laboratory prior to sampling.
20. Sterilized. Plastic must be Polypropylene.
21. 40 CFR part 141.74 regulations to avoid filtration or disinfection state 8 hours (DW compliance testing). Most facilities are using either disinfection or filtration so the 8 would not apply in most cases.
22. 40 CFR part 141.74 regulations for Disinfection By-Product rule state 8 hours (DW compliance testing) where SM 9215 allows up to 24 hours if sample is stored between > 0 and $\leq$ 4° C.
23. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to $\leq$ 6°C is acceptable.
24. Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.
25. Holding Time is 24 hours if pH adjustment is not performed.

Table 22-2
Holding Times, Preservation and Container Requirements: NPDES – Bacteria, Protozoa, Toxicity Tests

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.	PRESERVATION[2,3] Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Total, Fecal, and E.coli Coliforms	Plastic/Glass	10ºC	0.0008 % $Na_2S_2O_3$[6]	6 hours	100 mL
Fecal Streptococci	Plastic/Glass	10ºC	0.0008 % $Na_2S_2O_3$[6]	6 hours	100 mL
Enterococci	Plastic/Glass	10ºC	0.0008 % $Na_2S_2O_3$[6]	6 hours	100 mL
Cryptosporidium	LPDE Plastic	0-8ºC	None	96 Hours	500 mL
Giardia	LPDE Plastic	0-8ºC	None	96 Hours	500 mL
Toxicity – Acute/Chronic	Plastic/Glass	$\leq$ 6ºC[5]	None	36 Hours	2 L

Key to Table

1. Plastic should be Polypropylene or other sterilizable plastic.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4^{o}C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. Samples must not be frozen. Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present, when samples arrive, it is necessary to measure the temperature of the samples and confirm that the $\leq$ 6^{o}C temperature has not been exceeded.
6. Should only be used in the presence of residual chlorine.

Table 22-3
Holding Times, Preservation and Container Requirements: NPDES - Inorganic

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp[14].	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Acidity	Plastic/Glass	≤ 6ºC	None	14 days	100 mL
Alkalinity	Plastic/Glass	≤ 6ºC	None	14 days	100 mL
Ammonia	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH<2	28 days	400 mL
BOD 5 Day	Plastic/Glass	≤ 6ºC	None	48 hours	1000 mL
Boron	Plastic[5]	None	HNO_3 to pH<2	6 months	200 mL
Bromide	Plastic/Glass	None	None	28 days	100 mL
CBOD 5 Day	Plastic/Glass	≤ 6ºC	None	48 hours	1000 mL
COD	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH<2	28 days	100 mL
Chloride	Plastic/Glass	None	None	28 days	50 mL
Chlorine, Residual	Plastic/Glass	None	None	15 min.[6]	200 mL
Color	Plastic/Glass	≤ 6ºC	None	48 hours	50 mL
Cyanide –Total [16, 17]	Plastic/Glass	≤ 6ºC	NaOH to pH >12, 0.6 g Ascorbic Acid[7]	14 days	100 mL
Cyanide – Amenable [16, 17]	Plastic/Glass	≤ 6ºC	NaOH to pH >12, 0.6 g Ascorbic Acid[7]	14 days	100 mL
Fluoride	Plastic	None	None	28 days	300 mL
Hardness	Plastic/Glass	None	HNO_3 to pH<2[8]	6 months	100 mL
Hexavalent Chromium	Plastic/Glass	≤ 6ºC	Ammonium sulfate buffer pH = 9.3 - 9.7	28 days / 24 hrs[15]	200 mL
Hydrogen Ion (pH)	Plastic/Glass	None	None	15 min.[6]	200 mL
Kjeldahl and organic Nitrogen	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	500 mL
Mercury[11]	Plastic/Glass	None	HNO_3 to pH<2	28 days	200 mL
Metals[9,10]	Plastic/Glass	None	HNO_3 to pH<2 [18]	6 months	200 mL
Nitrate	Plastic/Glass	≤ 6ºC	None	48 hours	100 mL
Nitrate-Nitrite	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	100 mL
Nitrite	Plastic/Glass	≤ 6ºC	None	48 hours	100 mL
Oil and Grease	Glass	≤ 6ºC	H_2SO_4 or HCl to pH <2	28 days	1 L

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp[14].	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Organic Carbon (TOC)	Plastic/Glass	≤ 6ºC	H_2SO_4 or HCl to pH <2[12]	28 days	250 mL
Orthophosphate	Plastic/Glass	≤ 6ºC	Filter within 15 min.	48 hours	250 mL
Oxygen, Dissolved Probe	Glass[13]	None	None	15 min.[6]	200 mL
Oxygen, Winkler	Glass[13]	None	Fix on site and store in dark.	8 hours	300 mL
Phenols	Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	500 mL
Phosphorus, Elemental	Glass	≤ 6ºC	None	48 hours	250 mL
Phosphorus, Total	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	250 mL
Residue, Total	Plastic/Glass	≤ 6ºC	None	7 days	1 L
Residue, Filterable	Plastic/Glass	≤ 6ºC	None	7 days	1 L
Residue, Non-Filterable	Plastic/Glass	≤ 6ºC	None	7 days	1 L
Residue, Settleable	Plastic/Glass	≤ 6ºC	None	48 hours	1 L
Residue, Volatile	Plastic/Glass	≤ 6ºC	None	7 days	1 L
Silica	Plastic[5]	≤ 6ºC	None	28 days	250 mL
Specific Conductance	Plastic/Glass	≤ 6ºC	None	28 days	250 mL
Sulfate	Plastic/Glass	≤ 6ºC	None	28 days	250 mL
Sulfide	Plastic/Glass	≤ 6ºC	Zinc acetate plus NaOH to pH>9	7 days	500 mL
Sulfite	Plastic/Glass	None	None	15 min.[6]	200 mL
Surfactants	Plastic/Glass	≤ 6ºC	None	48 hours	1 L
Temperature	Plastic/Glass	None	None	N/A	100 mL
Turbidity	Plastic/Glass	≤ 6ºC	None	48 hours	1 L

Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤ 6ºC until compositing and sample splitting is completed.

Key to Table

3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. May also be collected in quartz or PFTE Plastic.
6. For compliance testing, the analysis must be performed in the field at the time of analysis. If transported to the laboratory for analysis, the analysis will be performed as soon as practical and reported qualified.
7. Should only be used in the presence of residual chlorine. (Alternatively, sodium arsenite may be used.)
8. H_2SO_4 to a pH <2 is also acceptable.
9. Except Mercury and Hexavalent Chromium.
10. For dissolved metals, samples must be filtered on site before adding HNO_3 preservative (or before shipping to laboratory).
11. Samples collected for determination of trace level mercury (100 ng/L) using EPA 1631 must be collected in tightly capped Fluor polymer or glad bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipping, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
12. Phosphoric acid (H_3PO_4) may also be used.
13. Should have glass lid or top.
14. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of ‘‘≤ °C’’ is used in place of the ‘‘4 °C’’ and ‘‘<4 °C’’ sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).
15. Holding time is 24 hours if pH adjustment is not performed.
16. In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH. If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered (with sulfide treatment by laboratory) and qualify the results in the final report.
17. It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
18. Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.

Table 22-4
Holding Times, Preservation and Container Requirements: NPDES - Organic

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[15]	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Purgeable Halocarbons	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5]	14 days	40 mL
Purgeable Aromatic Hydrocarbons	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5], HCl to pH<2	14 days[6]	40 mL
Acrolein and Acrylonitrile	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5], adjust pH to 4-5[7]	14 days	40 mL
Phenols[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[8]	1 L
Benzidines[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[8, 11]	1 L
Phthalate esters[9]	Glass[4]	≤ 6ºC	None	7 days[8]	1 L
Nitrosamines[9,12]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
PCBs[9]	Glass[4]	≤ 6ºC	None	1 year[8]	1 L
Nitroaromatics and Isophorone[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
Polynuclear Aromatic Hydrocarbons[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
Haloethers[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[8]	1 L
Chlorinated Hydrocarbons[9]	Glass[4]	≤ 6ºC	None	7 days[8]	1 L
CDD/CDFs[9] – Aqueous: Field/Lab Preservation	Glass	≤ 6ºC	pH <9, 0.0008 % $Na_2S_2O_3$[5]	1 year	1 L
CDD/CDFs[9] – Solids/Mixed Phase/ - Field Preservation	Glass	≤ 6ºC	None	7 days	1 L
CDD/CDFs[9] – Tissue – Field Preservation	Glass	≤ 6ºC	None	24 hours	
CDD/CDFs[9] – Solids/Mixed Phase/Tissue - Lab Preservation	Glass	< -10ºC	None	1 year	1 L
Pesticides[9]	Glass	≤ 6ºC	pH 5-9 [14]	7 days[8]	1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at $\leq 6^{o}C$ until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. With Teflon lined septum.
5. Should only be used in the presence of residual chlorine. Ascorbic may be used instead.
6. Samples receiving no pH adjustments must be analyzed within 7 days. If 2-chlorovinylethylether is a target analyte, the sample should not be acidified.
7. The pH adjustment is not required if acrolein is not being measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.
8. 7 days until extraction, 40 days after extraction. (PCB only – 1 year after extraction)
9. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more categories, the sample may be preserved by cooling to $\leq 6^{o}C$ reducing residual chlorine with 0.0008 % sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine) and footnotes 10 and 11(re the analysis of Benzidine).
10. If 1,2-diphenylhydrazine is likely to be present, adjust pH to of the sample to 4.0 $\pm$ 0.2 to prevent rearrangement to benzidine.
11. Extracts may be stored up to 30 days before analysis if storage temperature is $< 0^{o}C$.
12. For the analysis of diphenylnitrosamine, add 0.008 % $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
13. Store in dark.
14. The pH adjustment may be performed upon receipt in the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.0008 % $Na_2S_2O_3$.
15. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of ‘‘≤ °C’’ is used in place of the ‘‘4 °C’’ and ‘‘<4 °C’’ sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

Table 22-5.
Holding Times, Preservation and Container Requirements: NPDES - Radiological

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Alpha, Beta, Radium	Plastic/Glass	None	HNO_3 to pH<2	6 months	1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Table 22-6.
Holding Times, Preservation and Container Requirements: RCRA - Aqueous

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.[12]	PRESERVATION[2,3] Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Chloride	Plastic/Glass	4ºC	None	28 days	100 mL
Cyanide -Total	Plastic/Glass	4ºC	NaOH to pH >12[5]	14 days	250 mL
Cyanide -Amenable	Plastic/Glass	4ºC	NaOH to pH >12[5]	14 days	250 mL
Hydrogen Ion (pH)	Plastic/Glass	4ºC	None	24 hours[11]	100 mL
Nitrate	Plastic/Glass	4ºC	None	48 hours	28 days
Oil and Grease	Glass	4ºC	HCl	28 days	1 L
Organic carbon (TOC)	Plastic/Glass	4ºC	pH to <2[6] Store in dark	28 days	28 days
Sulfate	Plastic/Glass	4ºC	None	28 days	400 mL
Sulfide	Plastic/Glass	4ºC	Add Zn Acetate	7 days	400 mL
Chromium VI	Plastic/Glass	4ºC	None	24 hours	250 mL
Mercury	Plastic/Glass	None	HNO_3 to pH<2	28 days	250 mL
Other Metals	Plastic/Glass	None	HNO_3 to pH<2[15]	6 months	250 mL
Acrolein and Acrylonitrile	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7] Adjust pH to 4-5[13]	14 days	1 L
Benzidines	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Chlorinated Hydrocarbons	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Dioxins and Furans	Glass[10]	4ºC	None	30 days[8]	1 L
Haloethers	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Nitroaromatics and cyclic ketones	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Nitrosamines	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Organochlorine Pesticides	Glass[10]	4ºC	None	7 days[8]	1 L
Organophosphorus Pesticides	Glass[10]	4ºC	Adjust pH[9]	7 days[8]	1 L
PCBs	Glass[10]	4ºC	None	None[14]	1 L
Phenols	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.[12]	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Phthalate Esters	Glass[10]	4ºC	None	7 days[8]	1 L
Polynuclear Aromatic Hydrocarbons	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Purgeable Hydrocarbons	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7] Adjust pH <2[2]	14 days	40 mL
Purgeable Halocarbons	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	14 days	40 mL
Total Organic Halides (TOX)	Glass[10]	4ºC	Adjust pH to <2 with H_2SO_4	28 days	1 L
Radiological Tests (Alpha, Beta, Radium)	Plastic/Glass	None	HNO_3 to pH<2	6 months	250 mL

Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
5. If oxidizing agents are present, add 5 mL 0.1 N $NaAsO_2$ or 0.06 g of ascorbic acid per L. See Cyanide SOP for additional information about other interferences.
6. Adjust pH to <2 with H_2SO_4, HCl, or solid $NaHSO_4$. Free Chlorine must be removed prior to adjustment.
7. Free Chlorine must be removed by the appropriate addition of $Na_2S_2O_3$.
8. 7 days until extraction. 40 days after extraction.
9. Adjust pH to 5-8 using NaOH or H_2SO_4.
10. With Teflon lined septum.
11. Holding Time is listed as “As Soon as Possible” in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
12. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to $\leq$ 6°C is acceptable.
13. Based on guidance from EPA MICE, if samples are received without pH adjustment, the holding time is 7 days.
14. Analysis to be completed within 40 days after extraction.
15. Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.

Table 22-7.
Holding Times, Preservation and Container Requirements: RCRA – Non-Aqueous

PARAMETER	CONTAINER [1]	PRESERVATION Temp.[7]	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Chloride	Glass	4ºC	None	28 days	50 g
Cyanide -Total	Glass	4ºC	None	14 days	50 g
Cyanide - Amenable	Glass	4ºC	None	14 days	50 g
Hydrogen Ion (pH)	Glass	4ºC	None	7 days[6]	50 g
Nitrate	Glass	4ºC	None	N/A	50 g
Oil and Grease	Glass	4ºC	None	28 days	50 g
Sulfide	Glass	4ºC	Add Zn Acetate, zero headspace	7 days	50 g
Chromium VI	Glass	4ºC	None	30 days	50 g
Mercury	Plastic/Glass	None	None	28 days	50 g
Other Metals	Plastic/Glass	None	None	6 months	50 g
Acrolein and Acrylonitrile	Glass[4]	4ºC	None	14 days	50 g
Benzidines	Glass[4]	4ºC	None	14 days[3]	50 g
Chlorinated Hydrocarbons	Glass[4]	4ºC	None	14 days[3]	50 g
Dioxins and Furans	Glass[4]	4ºC	None	30 days[3]	50 g
Haloethers	Glass[4]	4ºC	None	14 days[3]	50 g
Nitroaromatics and cyclic ketones	Glass[4]	4ºC	None	14 days[3]	50 g
Nitrosamines	Glass[4]	4ºC	None	14 days[3]	50 g
Organochlorine Pesticides	Glass[4]	4ºC	None	14 days[3]	50 g
Organophosphorus Pesticides	Glass[4]	4ºC	None	14 days[3]	50 g
PCBs	Glass[4]	4ºC	None	None[8]	50 g
Phenols	Glass[4]	4ºC	None	14 days[3]	50 g
Phthalate Esters	Glass[4]	4ºC	None	14 days[3]	50 g
Polynuclear Aromatic Hydrocarbons	Glass[4]	4ºC	None	14 days[3]	50 g

PARAMETER	CONTAINER [1]	PRESERVATION Temp.[7]	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Purgeable Hydrocarbons	Glass[4]	4ºC	None	14 days[5]	50 g
Purgeable Halocarbons	Glass[4]	4ºC	None	14 days[5]	50 g
Total Organic Halides (TOX)	Glass[4]	4ºC	None	28 days	50 g

Key to Table

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. 14 days until extraction. 40 days after extraction.
4. With Teflon Lined Septum.
5. See Volatile SOP for more detailed preservation requirements.
6. Holding Time is listed as “As Soon as Possible” in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
7. For samples with a temperature requirement of 4^{o}C, a sample temperature of just above the water freezing temperature to $\leq$ 6^{o}C is acceptable.
8. Analysis to be completed within 40 days after extraction.

Table 22-8.
Holding Times, Preservation and Container Requirements: Air Samples

PARAMETER	CONTAINER [1]	PRESERVATION Temp.	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Volatile Organics	Summa Canister	None	None	30 days	6L or 1L
Volatile Organics	Tedlar Bag	None	None	72 hrs[3,4]	1 L

Key to Table

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. Holding Time is based on SW 846 Method 0040 “SAMPLING OF PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS FROM COMBUSTION SOURCES USING TEDLAR® BAGS”. Some states specifically enforce this holding time (e.g. Florida, New Jersey) and others have not specified this information in their regulatory requirements.
4. The holding time is 72 hours unless the laboratory has a documented validation study that indicates a longer HT is acceptable for the analytes of interest.

SECTION 23 HANDLING OF SAMPLES *(NELAC 5.5.8)*

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, legal COCs will be generated per the Manual for Certification of Laboratories Analyzing Drinking Water, Fifth Edition, January 2005, Appendix A, and SOP No. WS-QA-0003, "Sample Receipt and Procedures".

23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in SOP No. WS-QA-0003, "Sample Receipt and Procedures"

23.2.1 Laboratory Receipt

Laboratory receipt procedures are summarized in SOP No. WS-QA-0003.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

When samples arrive at the laboratory, they are assigned a tracking ID consisting of the Lot ID and a sample number. The lot ID is a 9 character alphanumeric identifier, with the composition as follows:

Character Position	Valid Values	Significance
First	"G"	Assigned to TestAmerica West Sacramento, to distinguish from other laboratories using the Quantims LIMS.
Second	0-9	Last digit of the year in which the samples are received.
Third	A – L	Corresponds to the month in which the samples are

		received. A = January, B= February, etc.
Fourth & Fifth	01 – 31	Corresponds to the day of the month in which samples are received.
Sixth	0	Separator between day and sequence values.
Seventh through Ninth	400 – 999	Sequence value denoting the order in which lots were logged into Quantims on a given day. Each lot will have a unique sequence number, no matter which Quantims Laboratory logged in the lot.

From the table above, the lot ID G0F100544 is for TestAmerica West Sacramento, logged in in 2010, June, the tenth day, and sequence number 544 for that day.

Once a lot ID is assigned and samples are individually logged in, a workorder(5) and bottle number are assigned to each container. In addition, workorder(8) values are assigned for each analysis requested. The workorder(8) is used to distinguish extracts and digestates produced from the sample. The workorder(5) consists of 5 alphanumeric characters assigned by the LIMS. The LIMS assigns these values sequentially as samples are logged in across the network, using 0-9, and letters of the alphabet excluding “B”, “O” and “I”, as these may be mistaken for numbers. In addition, sample containers are labeled with the lot ID and a sample number (from 1 to 99).
The workorder(8) consists of the workorder(5), plus 3 additional alphanumeric characters, beginning with “1AA”. The first digit of the additional characters denotes the analysis number, such that “1” is the first analysis, “2” is a reanalysis, etc. The second and third characters are assigned sequentially as test requests are added to the sample.

23.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. WS-QA-0003.

23.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, are returned to the secure sample control area. Empty sample containers are marked as “DIT” (destroyed in testing) on the sample receiving check out form and are disposed by the analytical staff. All samples are kept in the refrigerators for 30 days past invoicing, unless other arrangements have been made with the client.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: WS-EHS-001, "Waste Disposal"). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

Figure 23-1.

Example: Chain of Custody (COC)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Test America West Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
Tel 916 373-5600 Fax 916 372-1059

Chain of Custody Record

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	____ of ____ COCs
Address	Analysis Turnaround Time			Job No.
City/State/Zip	Calendar (C) or Work Days (W) _____			
(xxx) xxx-xxxx Phone	TAT If different from Below _____			SDG No.
(xxx) xxx-xxxx FAX	☐ Standard			
Project Name:	☐ 1 week			
Site:	☐ 3 days			
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type	# of Matrix Cont.	Filtered Sample	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

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

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

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:

Figure 23-2

Example: Sample Acceptance Policy

NELAC and TestAmerica West Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. TestAmerica West Sacramento will review your sample shipment against those requirements as listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

TestAmerica West Sacramento requirements are as follows:

- ✓ Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples, shall be provided.
- ✓ Samples must be accompanied by written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law.
- ✓ Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- ✓ Drinking waters samples for Method 1613B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- ✓ The samples shall arrive at the laboratory with adequate remaining holding time for the analyses requested.
- ✓ Sufficient sample volume must be available to perform the requested analyses.
- ✓ Received samples must not exhibit obvious signs of damage, contamination or inadequate preservation.
- ✓ For samples undergoing chemical warfare degradate analysis, the sample must be screened for agent prior to shipment in accordance with appendix 10 of our Sample Receipt Procedure (WS-QA-0003).
- ✓ Samples containing mammalian tissue will not be accepted without prior coordination with a project manager. Additional conditions for receipt and handling of tissue are outlined in appendix 11 of our Sample Receipt Procedure (WS-QA-0003).

The laboratory will notify the client/Project Manager upon sample receipt if the samples fail to meet any of the above requirements.

When completing the chain of custody form, please do not forget to sign your name in the "relinquished by" box.

Figure 23-3. Example: Cooler Receipt Forms

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

LOT RECEIPT CHECKLIST
TestAmerica West Sacramento

CLIENT _____ PM_____ LOG # _____

LOT# (QUANTIMS ID) _____ QUOTE#_____ LOCATION_____

Checked (✓)

DATE RECEIVED _____ TIME RECEIVED_____ ☐

DELIVERED BY ☐ FEDEX ☐ ON TRAC ☐ CLIENT
☐ GOLDENSTATE ☐ UPS ☐ GO-GETTERS ☐ OTHER
☐ TAL COURIER ☐ TAL SF ☐ VALLEY LOGISTICS ☐

CUSTODY SEAL STATUS ☐ INTACT ☐ BROKEN ☐ N/A ☐

CUSTODY SEAL #(S) _____

SHIPPPING CONTAINER(S) ☐ TAL ☐ CLIENT ☐ N/A ☐

COC #(S)_____ ☐

TEMPERATURE BLANK Observed:_____ Corrected:_____

SAMPLE TEMPERATURE - (TEMPERATURES ARE IN °C)

Observed:_____ Average_____ Corrected Average_____

LABORATORY THERMOMETER ID:

IR UNIT: #4 ☐ #5☐ ☐ OTHER_____ ☐

Initials Date

==========

pH MEASURED ☐ YES ☐ ANOMALY ☐ N/A ☐
LABELED BY..... ☐
LABELS CHECKED BY..... ☐
PEER REVIEW _____ ☐ NA

SHORT HOLD TEST NOTIFICATION SAMPLE RECEIVING ☐
WETCHEM ☐ N/A ☐
VOA-ENCORES☐ N/A ☐

☐ METALS NOTIFIED OF FILTER/PRESERVE VIA VERBAL & EMAIL ☐ N/A ☐

☐ COMPLETE SHIPMENT RECEIVED IN GOOD CONDITION WITH ☐ N/A ☐
APPROPRIATE TEMPERATURES, CONTAINERS, PRESERVATIVES

☐ CLOUSEA ☐ TEMPERATURE EXCEEDED (2 °C – 6 °C)*1 ☐ N/A
☐ WET ICE ☐ BLUE ICE ☐ GEL PACK ☐ NO COOLING AGENTS USED ☐ PM NOTIFIED

Initials Date

Notes_____

*1 Acceptable temperature range for State of Wisconsin samples is ≤4°C.

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Bottle Lot Inventory

Lot ID:_____

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
VOA*																				
VOAh*																				
AGB																				
AGBs																				
250AGB																				
250AGBs																				
250AGBn																				
500AGB																				
____AGJ																				
500AGJ																				
250AGJ																				
125AGJ																				
____CGJ																				
500CGJ																				
250CGJ																				
125CGJ																				
PJ																				
PJn																				
500PJ																				
500PJn																				
500PJna																				
500PJzn/na																				
250PJ																				
250PJn																				
250PJna																				
250PJzn/na																				
Acetate Tube																				
_____"CT																				
Encore																				
Folder/filter																				
PUF																				
Petri/Filter																				
XAD Trap																				
Ziploc																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

h = hydrochloric acid s = sulfuric acid na = sodium hydroxide n = nitric acid zn = zinc acetate

Number of VOAs with air bubbles present / total number of VOA's

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SECTION 24 ASSURING THE QUALITY OF TEST RESULTS *(NELAC 5.5.9)*

24.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 NEGATIVE CONTROLS

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	Are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
Calibration Blanks	Are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Solvent/Reagent /Consumable Material Blanks	When new lots of solvents, reagents or consumable materials are received, a blank using these new materials must be prepared and shown to be ND to less than ½ the reporting limit. The blank can be a batch Method Blank with the exception of DoD method blanks which cannot be used for this purpose.
Instrument Blanks	Are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1. Example – Negative Controls

Control Type	Details
Trip Blank [1]	Are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	Are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	Are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Holding Blanks	Also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 SAMPLE MATRIX CONTROLS

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exceptions: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable. Method 1694 may have acceptance limits <10% provided the analyte is detectable and identifiable.
- The maximum recommended recovery limit will be 150%. Some specific methods or SOPs may allow for higher recoveries.
- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. See Policy WS-PQA-003 for further details.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- Or, for NELAC and Departement Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).
- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.
- Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25 REPORTING RESULTS *(NELAC 5.5.10)*

25.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

25.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed or prepared electronically, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report For Samples) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.
- The applicable COC is an integral part of the report.

- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (eg. Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.21 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

25.2.22 The laboratory includes a cover letter.

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.24 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.25 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 REPORTING LEVEL OR REPORT TYPE

The laboratory offers three levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level II is a report with the features described in Section 25.2 above plus summary information, including results for the method blank reported to the laboratory MDL if required, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms or instrument print-outs, and relevant calibration information. No raw data is provided unless it is necessary to provide the relevant calibration information.
- Level VI is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in electronic deliverable form, either via e-mail or CD ROM. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. West Sacramento offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP # CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at (916) 373-5600 (or for e-mails: please notify us immediately by e-mail or by phone (916 373-5600) and delete this material from any computer.

25.7 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "Amend". The revised report will have the word "revised" or "amended" next to the date in the footer.

When the report is re-issued, a notation of "Amended" is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. For Example: Report was revised on 11/3/07 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/07.

25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

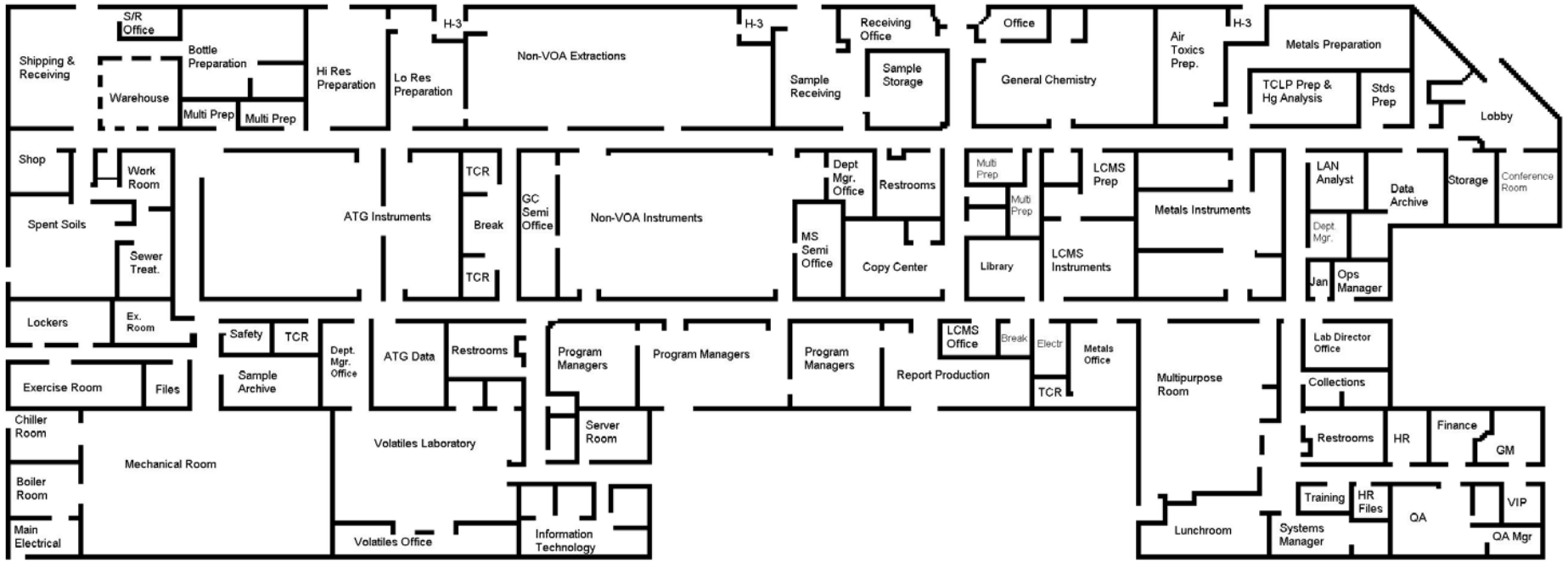
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1.

Laboratory Floor Plan



Facility Size	Square Feet
Total Area	66,000
Lab Area	43,000
Storage Area	5,200
	Linear Feet
Bench Top	3,000
Hoods	500

Appendix 2. Glossary/Acronyms

Glossary:

Acceptance Criteria:

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

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]

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)

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)

Batch:

Environmental samples which 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 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) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include 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. (ASQC)

Blind Sample:

A 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.

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:

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

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 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)

Compromised Samples:

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (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)

Correction:

Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

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 re 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)

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 if performed. (ASQC)

Duplicate Analyses:

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Environmental Detection Limit (EDL):

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement

procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

Equipment Blank:

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

External Standard Calibration:

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

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 Blank:

Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:

NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Holding Times (Maximum Allowable Holding Times):

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard:

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

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 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, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. 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.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1st Order Curve):

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):

An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Matrix:

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as 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: 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):

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.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

Method 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)

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)

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)

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)

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)

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 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 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)

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. (EAP-QAD)

Quality Control:

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

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:

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

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)

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 Standard:

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

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)

Report Limit (RL):

The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

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)

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 Duplicate:

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic):

The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity:

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of 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)

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.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (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)

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.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

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)

Uncertainty:

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

A2LA – American Association for Laboratory Accreditation
ANSI – American National Standards Institute
ASQ – American Society for Quality
ASTM – American Society for Testing and Materials
BS – Blank Spike
BSD – Blank Spike Duplicate
CAR – Corrective Action Report
CCB – Continuing Calibration Blank
CCC – Calibration Check Compound
CCV – Continuing Calibration Verification
CERCLA – Comprehensive Environmental Response, Compensation and Liability Act
CF – Calibration Factor
CFR – Code of Federal Regulations
CLP – Contract Laboratory Program
COC – Chain of Custody
CRS – Change Request Form
DL – Detection Limit
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DU – Duplicate
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICB – Initial Calibration Blank
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LOD- Level of Detection
LOQ- Level of Quantitation
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MDL – Method Detection Limit
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
NIOSH – National Institute for Occupational Safety and Health
NPDES – National Pollutant Discharge Elimination System
NRC – Nuclear Regulatory Commission
NRM – National Reference Material
PT – Performance Testing
PUF – Polyurethane Foam

QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP: Standard Operating Procedure
SPCC – System Performance Check Compound
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound
WS – Water Supply
WP – Water Pollution

Appendix 3. Laboratory Certifications, Accreditations, Validations

West Sacramento maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Lab ID	Authority
A2LA	C581	A2LA
DoD ELAP	2928-01	A2LA
NELAC Primary AB	1119CA	California
NELAC Secondary AB	E87570	Florida
NELAC Secondary AB	200060	Illinois
NELAC Secondary AB	E-10375	Kansas
NELAC Secondary AB	30612	Louisiana
NELAC Secondary AB	CA005	New Jersey
NELAC Secondary AB	11666	New York
NELAC Secondary AB	CA200005	Oregon
NELAC Secondary AB	68-01272	Pennsylvania
NELAC Secondary AB	T104704399-08-TX	Texas
NELAC Secondary AB	QUAN1	Utah
State Program	UST-055	Alaska
State Program	AZ0708	Arizona
State Program	88-0691	Arkansas
State Program	N/A	Colorado
State Program	PH-0691	Connecticut
State Program	960	Georgia
State Program	N/A	Guam
State Program	N/A	Hawaii
State Program	9947	Michigan
State Program	CA44	Nevada
State Program	N/A	New Mexico
State Program	87014	South Carolina
State Program	178	Virginia
State Program	C1281	Washington
State Program	334	West Virginia
State Program	9930C	West Virginia
State Program	998204680	Wisconsin
State Program	8TMS-Q	Wyoming
US Fish & Wildlife	LE148388-0	US Fish and Wildlife
USDA	P330-09-00055	USDA
USEPA	CA00044	USEPA

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the QA office.

Appendix 4: Listing of Methods Performed

Preparation Only Methods

Method	Aqueous	Solid	Waste	Biological	Air
Organics					
Calif. CAM-WET	X	X	X		
EPA 1311	X	X	X		
EPA 3510C	X				
EPA 3520C	X				
EPA 3535	X				
EPA 3540B		X			
EPA 3542					X
EPA 3550B		X		X	
EPA 3580A			X		
EPA 3600C	X	X	X		
EPA 3620B	X	X	X		
EPA 3630C	X	X	X		
EPA 3640A	X	X		X	
EPA 5030B	X	X	X		
EPA 5035	X	X	X		
Inorganics					
Calif. CAM WET	X	X	X		
EPA 1311	X	X	X		
EPA 1312 (W)	X	X	X		
EPA 3005A	X				
EPA 3010A	X				
EPA 3050B		X	X	X	

Organics Methods Performed

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
Volatile Organics	SW846 8260B	X	X	X		
Base Neutrals and Acids (BNAs)	SW846 8270B	X	X	X	X	
	TO-13A					X
	IP-7					X
	EPA 23					X
Organochlorine Pesticides	SW846 8081A	X	X	X	X	
	TO-4A					X
	TO-10A					X
	IP-8					X
	WS-ID-0014	X	X	X	X	
PCBs	EPA 8082	X	X	X	X	
	TO-4A					X
	TO-10A					X
Petroleum Hydrocarbons	EPA 8015B	X	X	X		
	CA LUFT	X	X	X		
	AK101	X	X	X		
	Ak102	X	X	X		
	AK103	X	X	X		
	NWTPH-Gx	X	X	X		
	NWTPH-Dx	X	X	X		
	GRO/DRO	X	X	X		
Nitroaromatics and Nitroamines	EPA 8330	X	X	X		X
	EPA 8330A	X	X	X		
	EPA 8330B	X	X	X		
	EPA 8321A (modified)	X	X	X		
	WS-LC-0001	X	X	X		
	WS-LC-0009	X	X	X		
	WS-LC-0010	X	X	X		
PAHs	EPA 8270C (SIM Isotope dilution)	X	X	X	X	X
	EPA 8270C (SIM)	X	X	X		
	CARB 429	X	X	X	X	X
	TO-13A					X
	IP-7					X
Nonyl Phenols	WS-MS-0013	X	X		X	
CBSA	WS-LC-0013	X	X			
Chemical Warfare Degradates	EPA 8321A (Modified)	X	X			
	WS-LC-0004	X	X			

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
Organosulfur Degradates	EPA 8270C	X	X			
	WS-MS-0003	X	X			
PFOA/PFOS	WS-LC-0020	X	X			
PPCPs (Pharmaceuticals & Personal Care Products)	EPA 1694	X				
Steroids & Hormones	EPA 1698	X				
PCB Congeners	EPA 1668A	X	X	X	X	X
Dioxins & Furans	EPA 1613B	X	X			
	EPA 8290	X	X	X	X	
	EPA 8280A	X	X	X	X	
	NCASI 551	X	X			
	DLFM01.1	X	X	X		
	EPA 0023A					X
	EPA 23					X
	TO-9					X

Metals Methods Performed

Parameter	Methods	Aqueous	Solid	Waste	Biological	Air
Trace Metals	EPA 200.7	X				
	EPA 200.8	X				
	EPA 6010B	X	X	X	X	X
	EPA 6020	X	X	X	X	X
	EPA 0060					X
	EPA 12					X
	CARB 12					X
	EPA 29					X
	CARB 436					X
Hardness	SM 2340B	X				
	EPA 200.7	X				
	EPA 200.8	X				
Mercury	EPA 245.1	X				
	EPA 200.8	X				
	EPA 6020	X				X
	EPA 7470A	X				
	EPA 7471A		X	X	X	X
	EPA 101A					X
	ASTM D6784-02					X
	Ontario-Hydro					X
	EPA 0060					X
	EPA 29					X
	CARB 436					X

Inorganics Methods Performed

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
Alkalinity (Carbonate, Bicarbonate, Total)	SM 2320B	X				
Ammonia	EPA 350.1	X				
Bromide	EPA 300.0	X				
	EPA 9056	X	X			
	EPA 9057					X
	EPA 26A					X
	CARB 421					X
Carbon, Total Inorganic	EPA 9060	X	X			
Carbon, Total Organic	EPA 9060	X	X			
	SM 5310 C	X				
Chloride	EPA 300.0	X				
	EPA 9056	X	X			
	EPA 9057					X
	EPA 26A					X
	CARB 421					X
Chromium, Hexavalent	EPA 7196A	X	X			
	EPA 0061					X
	EPA 306					X
	CARB 426					X
Conductivity	EPA 9050A	X				
	SM 2510 B	X				
Cyanide, Free	EPA 9012A	X	X			
	SM 4500 CN E	X				
Cyanide, Total	EPA 335.4	X				
	EPA 9012A	X	X			
	CARB 426					X
Demand, Chemical Oxygen	EPA 410.4	X				
Flouride	EPA 300.0	X	X			
	EPA 9056	X	X			
	EPA 9214	X	X			
	SM 4500 F C	X				
	EPA 9057					X
	EPA 26A					X
	CARB 421					X
n-Hexane Extractable Materials	EPA 1664A	X				
	EPA 9070A	X				
	EPA 9071B		X			
Moisture	ASTM 2216		X			

Nitrate	EPA 353.2	X				
	EPA 300.0	X				
	EPA 9056	X	X			
	CARB 421					X
Nitrate-Nitrite	EPA 353.2	X				
Nitrite	EPA 353.2	X				
	EPA 300.0	X				
	EPA 9056	X	X			
	CARB 421					X
Nitrocellulose	EPA 353.2	X	X			
	WS-WC-0050	X	X			
Total Kjeldahl Nitrogen	EPA 351.2	X				
Orthophosphate	EPA 365.3	X				
	EPA 300.0	X				
	EPA 9056	X	X			
Particulates in Air	EPA 5					X
	40 CFR Part 50					X
Perchlorate	EPA 314.0	X				
	EPA 331.0	X				
	EPA 6850	X	X			
	WS-LC-0012	X	X			
pH	SM 4500 H+ B	X				
	EPA 150.2	X				
	EPA 9040A	X				
	EPA 9041A	X				
	EPA 9045C		X	X		
Phosphorus, Total	EPA 365.3	X				
	EPA 365.4	X				
Solids, Total	SM 2540 B	X				
Solids, Total Dissolved	SM 2540 C	X				
Solids, Total Suspended	SM 2540 D	X				
Settleable Solids	SM 2540 F	X				
Sulfate	EPA 300.0	X				
	EPA 9065	X				
Sulfide	SM 4500 S2- D	X				
Turbidity	SM 2130 B	X				

Appendix 5 . Data Qualifiers

Qualifier Organic	Qualifier Inorganic	Footnote
U	U	Analyte analyzed for but was not detected.
G	G	Elevated reporting limit. The reporting limit is elevated due to matrix interference.
J	B	Estimated result. Result is less than RL.
E	I	Estimated result. Result concentration exceeds the calibration range.
B	J	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
P	*	Relative percent difference (RPD) is outside stated control limits.
a	N	Spiked analyte recovery is outside stated control limits.
*		Surrogate recovery is outside stated control limits.
PG		The percent difference between the original and confirmation analyses is greater than 40%.

Appendix C

CD-ROM Including: Laboratory Standard Operating Procedures

Legend Technical Services, St. Paul, MN
TestAmerica, West Sacramento, CA

(See CD included in Appendix B)

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**ANALYSIS OF ALKALINITY AS $CaCO_3$ IN WATER**
SOP NO.:	**LABENV-012.7**

Original Information		
Prepared by:	Rose Breiland	Date: 08/18/92
Technical Review:	Corine Goodrich	Date: 08/18/92
QA/QC Coordinator:		Date:
Authorized by:	Cheryl Sykora	Date: 08/18/92

Revision Information		
Supersedes:	LABENV-012.6	Date: 04/04/08
Revised by:	Victoria Bolton	Date: 11/18/10
Signature:	_____	Date: _____
Technical Review:	Dan Brezina	Date: 11/18/10
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 11/18/10
Signature:	_____	Date: _____

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Prepared by:	Rose Breiland	Date: 08/18/92
Technical Review:	Corine Goodrich	Date: 08/18/92
QA/QC Coordinator:		Date:
Authorized by:	Cheryl Sykora	Date: 08/18/92

Revision Information		
Supersedes:	LABENV-012.6	Date: 04/04/08
Revised by:	Victoria Bolton	Date:
Signature:		Date: 11/18/10
Technical Review:	Dan Brezina	Date:
Signature:		Date: 11-18-10
Authorized by:	Cheryl Sykora	Date:
Signature:		Date: 11/18/10

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SOP TITLE: ANALYSIS OF ALKALINITY AS $CaCO_3$ IN WATER

1. PURPOSE

1.1 This document defines the procedure to be followed when determining alkalinity in waters and wastewaters using an end-point pH of 4.5 su. The SOP is applicable to samples typically analyzed by Standard Methods (SM) 2320 B, Online Version, 1997.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is applicable to aqueous samples only.

3.2 This method is not applicable to samples that require results below 20 mg/L. Alkalinity as $CaCO_3$ can be reported less than 20 mg/L only if it has been determined by the low-level method.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 Gloves and safety glasses should be worn.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Samples should be collected in an unpreserved glass/plastic container and stored at 4 ± 2 °C.

5.4 The recommended holding time for samples is 14 days until analysis.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Standard sulfuric acid solution, 0.02 N, commercially prepared and certified

6.2 Bromocresol green-methyl red indicator solution, commercially prepared

6.3 pH meter, Orion EA 940 or equivalent

6.4 50 mL graduated cylinder

6.5 Buret, 50 mL with 0.1 mL graduations

6.6 Assorted laboratory glassware

6.7 Deionized (DI) water (>16 MΩ)

6.8 Check Standard (Na_2CO_3) – dissolve 106-159 mg of Na_2CO_3 to 500 mL of deionized water to produce a 200-300 ppm solution (1 mg/L Na_2CO_3 = 0.9434 mg/L $CaCO_3$) – freshly prepare once a year, sooner if results indicate a problem

6.9 Reporting Limit Verification (RLV) Standard (Na_2CO_3) – dissolve 10.6 mg of Na_2CO_3 to 500 mL of deionized water to produce a 20 ppm solution (1 mg/L Na_2CO_3 = 0.9434 mg/L $CaCO_3$) – freshly prepare once a year, sooner if results indicate a problem

7. PROCEDURE

7.1 Calibration

7.1.1 The pH meter should be calibrated at pH 4, 7, and 10 prior to each use (see SOP 'Analysis of pH by Electrometric Method').

7.2 Analysis

7.2.1 Record the project and sample numbers in the Alkalinity Logbook.

7.2.2 Using a 50 mL graduated cylinder, add 50 mL of DI water (method blank) to a labeled 250 mL Erlenmeyer flask. The method blank will be carried through all steps and used as the end-point color reference (slight pink to red).

7.2.3 Using a 50 mL graduated cylinder, add 50 mL of the check standard to a labeled 250 mL Erlenmeyer flask. Analyze a check standard at the beginning of each batch. Recovery of the check standard should be ± 10% or corrective action is taken.

7.2.4 Corrective action may include but is not limited to reanalyzing the check standard, preparing and analyzing a new check standard, and/or using new reagents.

7.2.5 Using a 50 mL graduated cylinder, add 50 mL of the RLV Standard to a labeled 250 mL Erlenmeyer flask.

7.2.5.1 Reporting limit verification (RLV) is checked monthly at a minimum by analyzing a standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.2.6 Using a 50 mL graduated cylinder, add 50 mL of each sample to a labeled 250 mL Erlenmeyer flask.

7.2.7 One duplicate sample is to be analyzed per batch of 10 or fewer samples.

7.2.8 Record the sample volumes in the Alkalinity Logbook.

7.2.9 Determine the pH of each sample with the pH meter and record in the Alkalinity Logbook.

7.2.10 Add 4-5 drops of bromocresol green-methyl red indicator to each Erlenmeyer flask.

7.2.11 Rinse buret with 0.02N sulfuric acid, fill past the 50 mL mark and deliver acid into a waste container to the zero mark.

7.2.12 While swirling the flask, titrate each sample.

7.2.13 As end-point is approached make smaller additions of acid, and make sure pH equilibrium is attained before adding more titrant.

7.2.14 Record volume of titrant used.

7.2.15 If turbidity or excess suspended solids in the sample interfere with indicator color change, transfer sample into a 200 mL beaker and determine alkalinity using a pH meter to an end-point of 4.5 su.

7.3 Calculation

$$\textit{Alkalinity, mg } CaCO_3/L = \frac{(A)(N)(50{,}000)}{\textit{mL sample}}$$

where:

A = amount of standard acid titrated, mL
N = normality of standard acid

8. WASTE DISPOSAL

8.1 Samples and analysis materials are disposed of in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, on an annual basis. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs can be found in Appendix B. Project specific RLs may override those listed. The RL for alkalinity is set at 20 mg/L based on the reference method.

9.2 Method Blank

9.2.1 The method blank must be less than the reporting limit or the batch is reanalyzed if possible. If it is not possible to reanalyze the batch, the data will be flagged. Do not subtract the blank result from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 The precision limit between the sample/sample duplicate must be $\leq$ 20% RPD or the batch is reanalyzed if possible. If it is not possible to reanalyze the batch, the data will be flagged.

9.3.2 QC calculations are found in the QA Manual

10. REPORTING

10.1 Water sample results are reported in mg/L.

10.2 The reported result is rounded to two significant figures.

10.3 The results are placed in the client file and a final report is sent to the client.

10.4 The reporting limit for this analysis is 20 mg/L based on methodology.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 Standard Methods for the Examination of Water and Wastewater, Method 2320 B, Online Version, 1997

12.2 Orion EA 940 Expandable Ion Analyzer Instruction Manual

Appendix A

Initial Demonstration of Capability (IDC)
Alkalinity as $CaCO_3$ in Water

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four replicate standards between 20-1000 mg/L in lab-grade water and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: 90.0-110%
   Precision: $\leq$20.0% RPD

7. The reagent blank must be less than the reporting limit (RL).

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Alkalinity as $CaCO_3$ in Water

Parameter	Water MDL (mg/L)	Water RL (mg/L)
Total Alkalinity	5.3	20

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-012.7
REVIEWER:	Victoria Bolton
DATE:	11/18/10

SECTION	CHANGE	RATIONALE
7.2.3	Added check standard is analyzed at the beginning of each batch	Clarification
Appendix B	Updated MDLs	Annual update

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

SOP NO.: LABENV-017.10

Original Information		
Prepared by:	Jennifer Nelson	Date: 01/27/94
Technical Review:	Sandra McDonald	Date: 12/12/94
QA/QC Coordinator:		Date:
Authorized by:	Cheryl Sykora	Date: 10/25/94

Revision Information		
Supersedes:	LABENV-017.9	Date: 04/27/09
Revised by:	Henrik Pham	Date: 05/02/11
Signature:	_____	Date: _____
Technical Review:	Van Pham	Date: 05/02/11
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 05/02/11
Signature:	_____	Date: _____

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SOP NO.: LABENV-017.10

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Prepared by:	Jennifer Nelson	Date: 01/27/94
Technical Review:	Sandra McDonald	Date: 12/12/94
QA/QC Coordinator:		Date:
Authorized by:	Cheryl Sykora	Date: 10/25/94

Revision Information		
Supersedes:	LABENV-017.9	Date: 04/27/09
Revised by:	Henrik Pham	Date:
Signature:	Henrik Pham	Date: 5/02/11
Technical Review:	Van Pham	Date:
Signature:	Van Pham	Date: 5/02/11
Authorized by:	Cheryl Sykora	Date:
Signature:	Cheryl Sykora	Date: 5/02/11

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SOP TITLE: DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

1. PURPOSE

1.1 This document defines the procedure to be followed for the preparation and analysis for polychlorinated biphenyls (PCBs) in soil, wipe, and water by Gas Chromatography (GC) using an Electron Capture Detector (ECD). The SOP is applicable to samples typically analyzed by EPA 8082.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the SOP.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is applicable to wastewater, groundwater, soil, wipe, and solid samples.

3.2 Phthalate ester interferences may be removed through the use of sulfuric acid cleanup.

3.3 Elemental sulfur is readily extracted from samples and may cause chromatographic interferences. Sulfur can be removed through the use of copper powder cleanup.

3.4 The Sonication Method may be used when a sample has the potential to be detrimental to the ASE (tar samples, fine sediments, caulk, etc.).

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 A lab coat should be worn.

4.4 When working with organic compounds, wear safety glasses and solvent resistant gloves.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in 1L amber glass bottles with Teflon lined caps and stored at ≤ 6 °C but not freezing.

5.4 The recommended holding time for water samples is 7 days until extraction and analysis within 40 days of extraction.

5.5 Soil samples should be collected in 4 oz. glass jars with Teflon lined caps and stored at ≤ 6 °C but not freezing.

5.6 The recommended holding time for soil samples is 14 days until extraction and analysis within 40 days of extraction.

5.7 Wipe samples should be received on 2" sterile gauze pads that are in 4 oz. glass jars with Teflon lined caps and stored at ≤ 6 °C but not freezing.

5.8 The recommended holding time for wipe samples is 1 year until extraction and analysis within 40 days of extraction.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Gas chromatograph equipped with dual injectors, dual ECDs, and a data processing system

6.2 GC columns – Rtx®-CLPesticides™, 30 m x 0.32 mm, 0.5 µm film (Restek #11139), and Rtx®-CLPesticidesII™, 30 m x 0.32 mm, 0.25 µm film (Restek #11324) or equivalent. Whichever two columns are selected, they must be of dissimilar stationary phases.

6.3 Two liter Teflon separatory funnel, or equivalent

6.4 500 mL Kuderna Danish (K-D) flask

6.5 Steam bath

6.6 Orbital shaker

6.7 Accelerated Solvent Extractor (ASE) and associated parts and glassware

6.8 Turbo Vap II and associated parts and glassware

6.9 100 mm glass funnel

6.10 10 mL Kuderna Danish (K-D) concentrator

6.11 Graduated cylinder, 1000 mL

6.12 pH paper (0-14 Std. Units)

6.13 Disposable glass pasteur pipets and bulb

6.14 Volumetric flasks, 100 mL, 50 mL, 25 mL, 10 mL

6.15 Microliter syringes

6.16 Filter paper – Whatman 41, or equivalent

6.17 2" sterile gauze pads

6.18 Snyder column

6.19 Assorted laboratory glassware

6.20 Glass wool

6.21 PTFE solvent rinsed boiling beads, or equivalent

6.22 Disposable weighing aluminum dishes – prerinsed with hexane and methylene chloride

6.23 Disposable graduated pipets

6.24 2 mL autosampler vials

6.25 1 dram saver vials with Teflon liners

6.26 Kimwipes®, or equivalent

6.27 Hydromatrix® or equivalent – muffle at 400 °C for four hours before using

6.28 Ottawa Sand (20-30 mesh) or equivalent – muffle at 400 °C for four hours before using

6.29 Anhydrous Sodium Sulfate (Na_2SO_4) – muffle at 400 °C for four hours before using

6.30 Acetone – pesticide grade, or equivalent

6.31 Hexane – pesticide grade, or equivalent

6.32 Methylene Chloride (CH_2Cl_2) – pesticide grade, or equivalent

6.33 Organic free water

6.34 Sodium Hydroxide (NaOH) – reagent grade, or equivalent

6.35 Sulfuric Acid (H_2SO_4) – trace metal grade, or equivalent

6.36 Nitric Acid (HNO_3) – trace metal grade, or equivalent

6.37 10N Sodium Hydroxide – dissolve 40 g of the reagent grade NaOH in 100 mL organic free water

6.38 1:1 Sulfuric Acid – slowly add 100 mL of trace metal grade H_2SO_4 to 100 mL organic free water

6.39 1% Nitric Acid – add 1 part HNO_3 to 99 parts organic free water

6.40 Copper powder – (-10 + 40 mesh, Sigma-Aldrich #31,140-5) or equivalent

NOTE: Reactive copper is indicated by a bright, shiny appearance. If the copper is dull, wash with 1% HNO_3, rinse with organic free water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

6.41 Extraction Solvent Mix for Solids – 3:1 Hexane and Acetone

6.42 Aroclor 1221 Stock – 1,000 µg/mL, Supelco #4-8098, or equivalent

6.43 Aroclor 1232 Stock – 1,000 µg/mL, Supelco #4-4805, or equivalent

6.44 Aroclor 1242 Stock – 1,000 µg/mL, Supelco #4-4806, or equivalent

6.45 Aroclor 1248 Stock – 1,000 µg/mL, Supelco #4-4807, or equivalent

6.46 Aroclor 1254 Stock – 1,000 µg/mL, Supelco #4-4808, or equivalent

6.47 Aroclor 1260 Stock – 1,000 µg/mL, Supelco #4-4809, or equivalent

6.48 Aroclor 1016-1260 Stock 1 – 1,000 µg/mL, Restek #32039, or equivalent

6.49 Aroclor 1016-1260 Stock 2 – 1,000 µg/mL, Ultra Scientific #PPM-8082 or equivalent, must be a different vendor or lot number than Aroclor 1016-1260 Stock 1 (used in PCB Second Source Standard)

6.50 Surrogate Stock – Restek #32000, 200 µg/mL for each of the following compounds: 2,4,5,6-Tetrachloro-m-xylene (TCMX), and Decachlorobiphenyl (DCB)

6.51 Surrogate Standard – dilute 0.25 mL of the Surrogate Stock Solution to 50 mL with 1:1 hexane and acetone to produce a 1.0 µg/mL Working Surrogate Standard. Store in a freezer for up to six months.

6.52 PCB Spike Standard – dilute 0.500 mL of the Aroclor 1260 Stock to 100 mL with 1:1 hexane and acetone to produce a 5.0 µg/mL PCB Spike Standard. Store in a freezer for up to six months.

6.53 Aroclor 1016-1260 Intermediate Solution – dilute 0.5 mL of the Aroclor 1016-1260 Stock 1 and 0.25 mL of the Surrogate Stock Solution to 10 mL with hexane to produce a 50 µg/mL Aroclor 1016-1260 and a 5.0 µg/mL Surrogate Standard. Store in a freezer for up to six months.

6.54 PCB Second Source Standard (CCAL/CCVS) – dilute 0.025 mL of the Aroclor 1016-1260 Stock 2 and 0.025 mL of the Surrogate Stock Solution to 25 mL with hexane to produce a 1.0 µg/mL Aroclor 1016-1260 and a 0.20 µg/mL Surrogate Second Source Standard. Store in a freezer for up to six months.

7. PROCEDURE

7.1 Preparation of Water Samples

7.1.1 Pre-rinse all glassware with extraction solvent.

7.1.2 Mark the water level on the outside of the bottle for later determination of volume.

7.1.3 Measure the pH of the sample and transfer to a pre-rinsed 2 L Separatory Funnel. (NOTE: If an evident layer of sediment is present, decant the sample and record on the extraction sheet.) The pH should be 5-9 su. If not, adjust the sample by using 10N NaOH or 1:1 H_2SO_4 and note on the extraction sheet.

7.1.4 Add 1.0 mL of 1.0 µg/mL Surrogate Standard to each sample, blank, and spike sample. Final concentration will be 1.0 µg/L.

7.1.5 For the samples in each analytical batch selected for spiking, add 1.0 mL of the 5.0 µg/mL PCB Spike Standard. Final concentration will be 5.0 µg/L. A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

7.1.6 Add 60 mL methylene chloride to the sample bottle and rinse. Transfer to the separatory funnel with the sample. (NOTE: If the sample was decanted, add 60 mL of methylene chloride directly to the separatory funnel.)

7.1.7 Cap and shake vigorously for 10 seconds and then vent. Cap and shake for 2 minutes. Allow the methylene chloride to separate from the sample.

7.1.7.1 If an emulsion interface between the layers is more than one-third the size of the solvent layer, the analyst should perform a beaker break without the use of Na_2SO_4.

7.1.7.2 Using a beaker, transfer the solvent layer into a glass 100 mm funnel containing a glass wool plug and about 2-3 inches of anhydrous muffled Na_2SO_4. Rinse the beaker with $MeCl_2$ and add to the funnel.

7.1.8 Drain into a 500 mL K-D flask equipped with a 10 mL K-D concentrator for Nitrogen Blowdown.

7.1.9 Repeat steps above with two additional fresh portions of methylene chloride.

7.1.10 After the final extraction, rinse the sodium sulfate with 20-30 mL of methylene chloride to complete the quantitative transfer.

7.1.11 If the emulsion layer is still present after the final shake, the analyst should employ mechanical techniques to complete the phase separation. Refer to the protocol found in Work Instruction (WI) 'Handling Emulsions'

7.1.12 Fill sample bottle with tap water to mark made previously. Transfer to a 1000 mL graduated cylinder and record volume on extraction sheet

7.1.13 K-D Technique / Nitrogen Blowdown

7.1.13.1 Add one solvent rinsed boiling bead to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-85 ºC) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot vapor. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 20 mL, add approximately 20 mL of hexane through the Snyder column.

7.1.13.2 Concentrate to approximately 5 mL, remove the K-D apparatus from the steam bath, and allow it to cool. Remove the Snyder column.

7.1.13.3 Put the concentrator tube in a warm bath (about 35 °C) and evaporate the solvent volume to less than 5 mL using a gentle stream of clean, dry nitrogen. During evaporation, the tube solvent level must be positioned to avoid water condensation (i.e., the solvent level should be below the level of the water bath). Do not allow the extract to go dry. Using a disposable pipet, adjust the final volume to 5.0 mL with hexane and mix the extract completely.

7.1.13.4 Transfer approximately 1 mL to a 2 mL autosampler vial and put the rest in a 4 mL saver vial. Store in freezer until analysis.

7.2 Preparation of Soil Samples – ASE technique

7.2.1 The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.2.2 Place two filters on top of the open end of the cell body. Use the black cylindrical insertion tool to push filter to the bottom of the assembled cell body. Very fine soils may require three filters.

7.2.3 Use a 1:1 Ottawa Sand:Hydromatrix® mixture for the blank and Laboratory Control Sample (LCS). A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

7.2.4 Weigh approximately 15 g of sample into a prerinsed disposable aluminum dish. Record the weight to the nearest 0.01 g.

7.2.5 Mix the sample with Hydromatrix®, or equivalent, using approximately a 1:1 ratio by volume, until free-flowing. Depending on the matrix, the amount of sample may need to be reduced.

7.2.6 Place the extraction cell funnel on open end of cell body. Load sample into cell through funnel. Gently tap cell on hard surface to pack sample evenly and to reduce void volume.

7.2.7 All samples should come within 1 cm of the top of the vessel. If a sample does not, use sand to fill.

7.2.8 When the transfer is complete, remove the funnel. Add 1.0 mL of 1.0 µg/mL Surrogate Standard to each sample, blank, and spike sample. Final concentration will be 0.067 mg/kg.

7.2.9 For the samples in each analytical batch selected for spiking, add 1.0 mL of the 5.0 µg/mL PCB Spike Standard. Final concentration will be 0.33 mg/kg.

7.2.10 Place filter paper on top of the sample. Remove any debris from cell threads and screw the second end cap onto open end of the cell body, and tighten.

7.2.11 Place completed extraction cell into position #1 on ASE. Place the corresponding collection vial into position #1 below.

7.2.12 Repeat steps above for additional samples.

7.2.13 Fill solvent bottles with the 3:1 Hexane to Acetone Extraction Solvent Mix.

7.2.14 Make sure pressure on gas tank is set to 180 psi. Make sure that solvent bottle pressure is 10 psi, system air pressure is 50 psi, and compression oven pressure is 130 psi.

7.2.15 Refer to Equipment SOP entitled ‘ASE’ for equipment set-up and operation.

7.2.16 Attach a K-D concentrator to a 500 mL evaporation flask for Nitrogen Blowdown or a 200 mL Turbo Vap II concentration vial.

7.2.17 Decant extraction solvent from the ASE collection vial through a hexane rinsed funnel with sodium sulfate and glass wool. Rinse the collection vial three times to complete quantitative transfer. Collect the extract in the assembled K-D concentrator flask or a 200 mL Turbo Vap II concentration vial.

7.2.18 K-D Technique / Nitrogen Blowdown

7.2.18.1 Add one solvent rinsed boiling bead to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of hexane to the top of the column. Place the K-D apparatus on a hot water bath (90-95 °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot vapor. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

7.2.18.2 When the apparent volume of liquid reaches 5 mL, remove the K-D apparatus from the water bath and allow it to cool. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of hexane.

7.2.18.3 Put the concentrator tube in a warm bath (about 35 ºC) and evaporate the solvent volume to less than 5 mL using a gentle stream of clean, dry nitrogen. During evaporation, the tube solvent level must be positioned to avoid water condensation (i.e., the solvent level should be below the level of the water bath). Do not allow the extract to go dry. Using a disposable pipet, adjust the final volume to 5.0 mL with hexane and mix the extract completely.

7.2.18.4 Transfer approximately 1 mL to a 2 mL autosampler vial and put the rest in a 4 mL saver vial. Store in freezer until analysis.

7.2.19 Turbo Vap II

7.2.19.1 Place the Turbo Vap collection tube in the Turbo Vap.

7.2.19.2 Set the water bath temperature to 40 °C and the pressure to 8-12 psi.

7.2.19.3 Set the Turbo Vap to monitor by ‘sensor’ to achieve a final volume of approximately 4 mL.

7.2.19.4 Using a disposable pipet, adjust the final volume to 5.0 mL with hexane and mix the extract completely. Transfer approximately 1 mL to a 2 mL autosampler vial and the remaining extract to a 4 mL saver vial. Store in freezer until analysis.

7.3 Preparation of Wipe Samples

7.3.1 For QC samples, label three 4 oz. jars for the Blank, LCS, and LCSD and add one 2” sterile gauze pad to each jar.

7.3.2 Add 1.0 mL of 1.0 µg/mL Surrogate Standard to each sample, blank, and spike sample. Final concentration will be 1.0 µg/wipe.

7.3.3 For the samples in each analytical batch selected for spiking, add 1.0 mL of the 5.0 µg/mL PCB Spike Standard. Final concentration will be 5.0 µg/wipe. A typical batch will have an LCS/LCSD.

7.3.4 Add 25 mL of hexane to all QC and sample jars.

7.3.5 Place on the orbital shaker for 20 minutes.

7.3.6 Remove solvent with a glass disposable pipet and quantitatively transfer into a prerinsed glass funnel containing a filter paper and sitting atop a 500 mL K-D flask equipped with a 10 mL K-D concentrator for Nitrogen Blowdown or a 200 mL Turbo Vap II concentration vial.

7.3.7 K-D Technique / Nitrogen Blowdown

7.3.7.1 Add one solvent rinsed boiling bead to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of hexane to the top of the column. Place the K-D apparatus on a hot water bath (90-95 °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot vapor. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of hexane.

7.3.7.2 Put the concentrator tube in a warm bath (about 35 °C) and evaporate the solvent volume to less than 5 mL using a gentle stream of clean, dry nitrogen. During evaporation, the tube solvent level must be positioned to avoid water condensation (i.e., the solvent level should be below the level of the water bath). Do not allow the extract to go dry. Using a disposable pipet, adjust the final volume to 5.0 mL with hexane and mix the extract completely.

7.3.7.3 Transfer approximately 1 mL to a 2 mL autosampler vial and put the rest in a 4 mL saver vial. Store in freezer until analysis.

7.3.8 Turbo Vap II

7.3.8.1 Place the Turbo Vap collection tube in the Turbo Vap.

7.3.8.2 Set the water bath temperature to 40 °C and the pressure to 8-12 psi.

7.3.8.3 Set the Turbo Vap to monitor by ‘sensor’ to achieve a final volume of approximately 4 mL.

7.3.8.4 Using a disposable pipet, adjust the final volume to 5.0 mL with hexane and mix the extract completely. Transfer approximately 1 mL to a 2 mL autosampler vial and the remaining extract to a 4 mL saver vial. Store in freezer until analysis.

7.4 Calibration

7.4.1 Prepare working standards at a minimum of 5 concentration levels, ranging from 0.4-3.0 µg/mL, by diluting the 50 µg/mL / 5.0 µg/mL Aroclor 1016-1260 Intermediate Solution with hexane. A typical calibration curve would be:

Inter. Solution (mL/25 mL)	Aroclor Conc. (µg/mL)	Surrogate Conc. (µg/mL)
0.2	0.40	0.040
0.3	0.60	0.060
0.4	0.80	0.080
0.5	1.0	0.10
1.0	2.0	0.20
1.5	3.0	0.30

7.4.2 When selecting peaks for quantitation, choose 5-7 peaks that are at least 25 % of the height of the largest Aroclor peak. Assign the concentration to each peak. Concentrations in the standard are determined using the mean value resulting from these peaks.

7.4.3 The average response factor should be calculated for each compound. The percent relative standard deviation (%RSD) should be less than 20% for each compound. If the %RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

7.4.4 If the %RSD of any compound is greater than 20%, construct calibration curves of area versus concentration using a first order or linear fit. Correlation coefficients (r) should be 0.990 or greater.

7.4.5 Calibration curve calculations are found in the QA Manual.

7.4.6 Reporting limit verification (RLV) is checked with each calibration curve or monthly at a minimum, by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.4.7 Use the PCB Second Source Standard as the continuing calibration standard – CCAL/CCVS.

7.4.8 Stock Standards should be stored in a freezer and replaced following manufacturer's expiration date or one year after opening, whichever comes first. Working standards should be stored in a freezer and replaced every 6 months or when analysis of continuing calibration standards indicate degradation or loss.

7.4.9 The RLVs of the other 5 Aroclors are used for pattern recognition. These standards may also be used as a single point calibration if the 1016-1260 is being used for the 5-point calibration and the Aroclor result is within 20% of the applicable RLV. An Aroclor standard may be analyzed to quantitate a sample run in the previous 12 hours.

NOTE: If a sample is expected to contain a specific Aroclor, the analyst may do a minimum 5-point calibration of that Aroclor in place of the 1016-1260. Prepare a calibration curve for the Aroclor of interest.

7.4.10 If a sample contains an Aroclor with an on-column concentration estimated to be between the reporting limit and 1.0 µg/mL, a 1.0 µg/mL standard is available to run as a dilution or as the single point calibration standard. For samples containing estimated on-column concentrations above 1.0 µg/mL, the sample is diluted so that the 1.0 µg/mL standard can be used. With a single point calibration, the sample should be within ± 20% of the concentration of the single-point standard to maximize accuracy.

7.5 Analysis

7.5.1 GC Conditions

7.5.1.1 Columns: Rtx®-CLPesticides™, 30m x 0.32mm, 0.5 µm film (Restek #11139), and Rtx®-CLPesticidesII™, 30 m x 0.32 mm, 0.25 µm film (Restek #11324) or equivalent

7.5.1.2 Injector Temperature: 250 °C

7.5.1.3 Detector Temperature: 310 °C

7.5.1.4 ECD 1 Temp Program: 150 °C for 0.5 min, 10 °C/min ramp to 200 °C, 5.0 °C/min ramp to 310 °C

7.5.1.5 ECD 2 Temp Program: 175 °C for 0 min, 6 °C/min ramp to 300 °C

7.5.1.6 Flow Rate: 1.6 mL/min (ECD 1), 1.0 mL/min (ECD 2); Constant Flow

7.5.1.7 Split Ratio: 40:1 (ECD 1), 60:1 (ECD 2)

7.5.1.8 GC Range: 0

7.5.1.9 Attenuation: 0

7.5.1.10 Injection Volume: 1.0 µL

7.5.2 The 1016-1260 mix may be used to demonstrate that a sample does not contain peaks that represent any Aroclor. As such, it is not necessary to run standards for each of the other 5 Aroclors, but it may be practical.

7.5.3 It is appropriate to perform an area sum on a sample in which the Aroclor pattern is no longer recognizable due to environmental factors. The same integration technique must be performed on standards. Any peaks in the area sum window not identifiable as PCBs on the basis of retention times should be subtracted from the total area. This procedure must be thoroughly documented and described to the data user.

7.5.4 Analyze the PCB Second Source Standard (CCAL/CCVS) at the beginning of each sequence, every 12 hours thereafter and at the end of the sequence. Recoveries should be ± 15% or corrective action should be taken. Corrective action may include reinjection, making a new standard, maintenance, and/or flagging the data.

7.5.5 If the response for a peak exceeds the working range of the system or the highest standard, dilute the extract with hexane and re-analyze.

7.5.6 If the presence of phthalate esters or other interfering compounds are suspected or found, a sulfuric acid cleanup may be performed.

7.5.6.1 Pipet 1.5 – 2 mL of 1:1 H_2SO_4 and 1.5 – 2 mL of extract into a clear glass 5 mL vial.

7.5.6.2 Shake vigorously for two minutes.

7.5.6.3 If a clear separation is not visible between the acid and solvent, centrifuge for 3-5 minutes.

7.5.6.4 If the acid (bottom) layer is colored, repeat steps above until the acid layer is colorless.

7.5.6.5 Remove the solvent (top) layer, place in an autosampler vial, and proceed with the analysis.

7.5.7 If the presence of sulfur is suspected or found, a copper powder cleanup may be performed.

7.5.7.1 To the autosampler vial containing 1 mL of extract, add approximately 2 g of reactive copper powder.

7.5.7.2 Shake vigorously for two minutes; allow to settle.

7.5.7.3 Remove the solvent layer, place in an autosampler vial, and proceed with the analysis.

7.5.7.4 Do not reuse the extract containing the copper powder.

7.6 Calculation

7.6.1 Identify the Aroclor type. For all samples that contain multiple Aroclors, calculate each Aroclor separately. Pattern recognition and experience of the analyst is a major factor in Aroclor identification.

7.6.2 Calculate the concentration of the Aroclor in the sample using one of the following equations:

$$\textit{Water Concentration } (\mu g / L) = \frac{(C_{ex})(V_{ex})(F)}{V_o}$$

$$\textit{Soil Concentration } (mg / kg) = \frac{(C_{ex})(V_{ex})(F)}{(W)(D)}$$

$$\textit{Wipe Concentration } (\mu g / wipe) = (C_{ex})(V_{ex})(F)$$

C_{ex} = extract concentration, µg/mL
V_{ex} = extract volume, mL
F = dilution factor (diluted volume/extract volume)
V_o = volume of sample extracted, L
W = sample weight, g
D = % dry weight of sample/100, or 1 for wet weight basis

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, when there is a change in the test method that may affect how the test is performed or when there is a major change in instrumentation. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the reporting limit or the sample batch is re-extracted if possible. If it is not possible to re-extract, the data will be flagged where appropriate. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Accuracy control limits are set at 70.0-130% for LCS and MS. Surrogate limits are generated semi-annually, using 20 Percent Recovery points, as follows:

9.3.1.1 Upper and Lower Control Limit = Mean ± 3s

9.3.1.2 Upper and Lower Warning Limit = Mean ± 2s

9.3.1.3 s = Standard deviation

9.3.1.4 For Arizona compliance, the surrogate lower control limit can not calculate below the lowest standard on the calibration curve (e.g. lowest standard = 0.04 µg/mL, spike is at 0.2 µg/mL, % can not be below 20.0%). The Minnesota Pollution Control Agency sets a guideline that the lower control limit can not be < 30.0%. For consistency between methods 608, 8081A, and 8082, LEGEND will use 40.0% (8081A limit).

9.3.2 Precision control limits are set at 20.0% RPD for LCS/LCSD and generated for MS/MSD. In-house control charts are generated semi-annually, using 20 RPD points, as follows:

9.3.2.1 Control Limit = Mean + 3s

9.3.2.2 Warning Limit = Mean + 2s

9.3.2.3 s = Standard deviation

9.3.2.4 RPD = Relative Percent Difference

9.3.3 QC calculations are found in the QA Manual

9.3.4 LCS, MS and surrogates are reviewed.

9.3.5 If the LCS data are outside the limits, a method nonconformance is filled out and the sample batch is re-extracted and/or re-analyzed if possible. If the batch cannot be re-analyzed, the data are flagged and/or a case narrative is written for all client reports within the batch.

9.3.6 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS is flagged and/or a case narrative is written.

9.3.7 If a sample surrogate is outside the limits, the sample is re-extracted and/or reanalyzed if possible. If the sample cannot be reanalyzed, the data is flagged next to the actual result in the report.

10. REPORTING

10.1 Soil sample results are reported in mg/kg on a dry weight basis.

10.2 Water sample results are reported in µg/L.

10.3 Wipe sample results are reported in µg/wipe and as a Modified EPA 8082.

10.4 The reported result is rounded to two significant figures.

10.5 The results are placed in the client file and a report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 EPA Methods 3510C, 3545, 3660B, 3665A, 8082, 8000B (MN), 8000C (AZ)

Appendix A

Initial Demonstration of Capability (IDC)
Polychlorinated Biphenyls (PCBs)

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards of all the aroclors in Ottawa sand and/or lab-grade water and a reagent blank. Only the 1260 Aroclor is analyzed for the wipe IDC.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: 70.0-130%
   Precision: ≤ 20% RPD

   If the standards were not extracted, the results must meet the following criteria:

   Accuracy: 85.0-115%
   Precision: ≤ 20% RPD

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the QA/QC Coordinator signs the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

10. For the analysis IDC all aroclors must be analyzed. Only one aroclor is required for the extraction IDC.

11. A minimum of two blind standards containing different aroclors must be analyzed to demonstrate aroclor pattern identification for the analysis IDC unless the analyst has already satisfied this requirement by another PCB method.

Appendix B

Method Detection Limits and Reporting Limits
Polychlorinated Biphenyls (PCBs)

Parameter	Water MDL (µg/L)	Water RL (µg/L)	Soil MDL (mg/kg)	Soil RL (mg/kg)	Wipe MDL (µg/wipe)	Wipe RL (µg/wipe)
Aroclor 1016	0.27	2.0	0.0079	0.20	0.49	2.0
Aroclor 1221	0.51	2.0	0.020	0.20	-----	2.0
Aroclor 1232	0.23	2.0	0.023	0.20	-----	2.0
Aroclor 1242	0.19	2.0	0.010	0.20	-----	2.0
Aroclor 1248	0.28	2.0	0.040	0.20	-----	2.0
Aroclor 1254	0.43	2.0	0.040	0.20	-----	2.0
Aroclor 1260	0.17	2.0	0.0059	0.20	0.35	2.0

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-017.10
REVIEWER:	Henrik Pham
DATE:	04/29/11

SECTION	CHANGE	RATIONALE
5.3, 5.5, 5.7	Changed '4 ± 2 °C' to '≤ 6 °C but above freezing'	40 CFR, Part 136 update
Appendix B	Revised MDLs	Current MDL study

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS SPECTROMETRY (GC/MS)**
SOP NO.:	**LABENV-020.8**

Original Information		
Prepared by:	Sandy McDonald	Date: 03/18/96
Technical Review:	Sharon Cenis	Date: 03/28/96
QA/QC Coordinator:		Date:
Authorized by:	Cheryl Sykora	Date: 04/03/96

Revision Information		
Supersedes:	LABENV-020.7	Date: 11/10/10
Revised by:	Sonny Hang	Date: 03/30/11
Signature:	_____	Date: _____
Technical Review:	Van Pham	Date: 03/30/11
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 03/30/11
Signature:	_____	Date: _____

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Revised by:	Sonny Hang	Date: 03/30/11
Signature:		Date: 3/30/11
Technical Review:	Van Pham	Date: 03/30/11
Signature:		Date: 3/30/11
Authorized by:	Cheryl Sykora	Date: 03/30/11
Signature:		Date: 3/30/11

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SOP TITLE: DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)

1. PURPOSE

1.1 This document defines the preparation and analysis for volatile organic compounds (VOCs) using purge and trap techniques. This document also describes the calibration and analysis of these compounds using a gas chromatograph coupled with a mass selective detector. The SOP is applicable to samples typically analyzed by EPA 8260B.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the SOP.

2.3 An analyst experienced in the use of gas chromatograph/mass spectrometers, skilled in the interpretation of mass spectra, and trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 The method is applicable to water, soil, and hazardous waste.

3.2 Interferences

3.2.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap.

3.2.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.2.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum of the sample container into the samples during shipment and storage. A trip blank prepared from organic-free water and carried through the sampling, handling and storage protocols can serve as a check for such contamination.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 The toxicity or carcinogenicity of most chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

4.3 Benzene has been classified as a known or suspected human or mammalian carcinogen. Pure standard materials and stock solutions of benzene should be handled in a hood.

4.4 Follow standard laboratory safety procedures.

4.5 A lab coat and safety glasses should be worn when preparing standards and samples.

4.6 When working with organic compounds, wear analyte resistant gloves.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in a minimum of three 40 mL VOA vials preserved with HCl to pH < 2 with no headspace and stored at 4 ± 2 °C.

5.4 The recommended holding time for preserved water samples is 14 days. If the sample is not preserved to pH < 2, the holding time is 7 days.

5.5 Soil samples should be collected in 40 mL weighed jars preserved with methanol and stored at 4 ± 2 °C.

5.6 The recommended holding time for soil samples is 14 days.

5.7 Be sure no solid material interferes with the sealing of sample containers and maintain hermetic seal on all sample containers until time of analysis.

5.8 Refrigerate samples upon receipt.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 HP 5890 Series II Gas Chromatograph (GC) with data processing equipment, or equivalent

6.2 Thermo Electron Corporation Focus GC with data processing equipment, or equivalent

6.3 HP 5972 Mass Selective Detector (MSD) with scan range of 35 to 300 amu using 70 volts electron energy in the electron impact ionization mode, or equivalent

6.4 Thermo Electron Corporation DSQ II (MSD) with scan range of 35 to 300 amu using 70 volts electron energy impact ionization mode, or equivalent

6.5 Column – 25 m x 0.20 mm ID 1.1 µm film thickness silicone-coated fused silica capillary column (DB-624 or equivalent)

6.6 Encon sample concentrator connected to an Archon autosampler, or equivalent

6.7 O.I Analytical Eclipse sample concentrator 4660 connected to O.I Analytical auto sampler 4552

6.8 Microliter syringes – 10, 25, 100, 250, 500, and 1000 µL

6.9 VOCARB 3000 trap for Encon sample concentrator, Supelco purging trap #10 for O.I Analytical Eclipse sample concentrator

6.10 Conical vials – 1 mL with mininert valves.

6.11 VOA vials with Teflon lined septa - 20 and 40 mL

6.12 Top loading balance, capable of reading to 0.01 g

6.13 Stainless steel spatula

6.14 Organic free water – purchased from Glenwood Inglewood, or equivalent

NOTE: The water is to be used for standard preparation, method blanks, dilutions, trip and field blanks.

6.15 Methanol – purge and trap grade

6.16 Calibration Stock 1 – 200 µg/mL each of bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, dichlorofluoromethane, trichlorofluoromethane and vinyl chloride, Absolute Standards, Inc. #60011, or equivalent

6.17 Calibration Stock 2 – 2,000 µg/mL of ETBE, Absolute Standards, Inc. #92450, or equivalent

6.18 Calibration Stock 3 – 200 µg/mL each of the compounds listed in Appendix B except for those listed above in Calibration Stocks 1-3, Absolute Standards, Inc. #61005, or equivalent

6.19 Spike Stock 1 – 200 µg/mL each of bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, dichlorofluoromethane, trichlorofluoromethane and vinyl chloride, Absolute Standards, Inc. #60011, or equivalent (must be a different lot number than Calibration Stock 1)

6.20 Spike Stock 2 – 200 µg/mL each of the compounds listed in Appendix B except for those listed above in Calibration Stocks 1-3, Absolute Standards, Inc. #61005, or equivalent (must be a different lot number than Calibration Stock 4)

6.21 GC/MS Tune Check/Surrogate Stock – 2,500 µg/mL each of 4-bromofluorobenzene (BFB), dibromofluoromethane, and toluene-d8, Restek #30073, or equivalent

6.22 Internal Standard Stock – 2,000 µg/mL each of 1,4-difluorobenzene, 2-bromo-1-chloropropane, 1,4-dichlorobenzene-d4, and pentafluorobenzene, Absolute Standards, Inc. #21013, or equivalent

6.23 Calibration Intermediate Solution 1 – dilute 250 µL of the 200 µg/mL Calibration Stock 1, 250 µL of the 200 µg/mL Calibration Stock 3, and 20 µL of the 2,500 µg/mL GC/MS Tune Check/Surrogate Stock to 1 mL with methanol to produce a 50 µg/mL Calibration Intermediate Solution 1

6.24 Calibration Intermediate Solution 2 (ETBE) – dilute 25 µL of the 2,000 µg/mL Calibration Stock 2 to 1mL with methanol to produce a 50 µg/mL Calibration Intermediate Solution 2

6.25 Calibration Intermediate Solution 3 – dilute 100 µL of the 50 µg/mL Calibration Intermediate Standard 1 to 1 mL with methanol to produce a 5.0 µg/mL Calibration Intermediate Solution 3

6.26 GC/MS Tune Check Standard – dilute 20 µL of the 2,500 µg/mL GC/MS Tune Check/Surrogate Stock and 25 µL of the 2,000 µg/mL Internal Standard Stock to 1 mL with methanol to produce a 50 µg/mL GC/MS Tune Check Standard

NOTE: This standard may also be used as a surrogate/internal standard for samples not loaded by the auto samplers but directly loaded by the analyst onto the sample concentrators

6.27 Auto sampler Internal Standard Solution – dilute 625 µL of the 2,000 µg/mL Internal Standard Stock to 5.0 mL with methanol to produce a 250 µg/mL auto sampler Internal Standard Solution

6.28 Auto sampler Surrogate Standard – dilute 500 µL of the 2,500 µg/mL GC/MS Tune Check/Surrogate Stock to 5.0 mL with methanol to produce a 250 µg/mL auto sampler Surrogate Standard

6.29 Spike Standard – dilute 250 µL each of the 200 µg/mL Spike Stock 1 and 2 to 1.0 mL with methanol to produce a 50 µg/mL Spike Standard

6.30 Unopened Calibration Stocks expire according to vendor expiration date. Opened Calibration Stocks expire six months from the opened date. Calibration Intermediate Solutions expire 3 months from the preparation date.

7. PROCEDURE

7.1 Preparation of Water Samples

7.1.1 Water samples are ready for analysis in the 40 mL vials.

7.2 Preparation of Soil Samples

7.2.1 Re-weigh the jar containing the soil and methanol to check sample weight.

7.2.1.1 If a 40 mL VOA vial was used and the sample weighs more than 10 g, add enough methanol to maintain a 1:1 soil to methanol ratio, if possible. Record the sample weight and the amount of methanol added. Flag data for samples containing less than 8 g or more than 20 g of soil, and those where a 1:1 ratio could not be maintained.

7.2.1.2 If a 2 oz. jar was used and the sample weighs more than 25 g, add enough methanol to maintain a 1:1 soil to methanol ratio, if possible. Record the sample weight and the amount of methanol added. Flag data for samples containing less than 20 g or more than 35 g of soil, and those where a 1:1 ratio could not be maintained.

7.2.2 Add 400 µL of methanol extract to 40 mL of organic-free water in a VOA vial.

7.3 Calibration

7.3.1 Initial Calibration

7.3.1.1 The GC/MS must be tuned to meet the criteria in Table 1 for 50 ng of BFB on column by either direct injection or by purging.

7.3.1.2 For 8260 water matrix ICAL: Prepare working standards at a minimum of five concentration levels, ranging from 1-200 µg/L, by diluting the 5.0 µg/mL Calibration Intermediate Solution 3 and the 50 µg/mL Calibration Intermediate Solution 1. A typical calibration curve would be:

Inter. Solution 3 5.0 µg/mL (µL/40 mL)	Inter. Solution 1 50 µg/mL (µL/40 mL)	Final Conc. (µg/L)
8	---	1.0
20	---	2.5
---	4	5.0
---	16	20
---	40	50
---	80	100
---	120	150
---	160	200

7.3.1.3 For 8260 soil matrix ICAL: Prepare working standards at a minimum of five concentration levels, ranging from 2.5-200 µg/L, by diluting the 50 µg/mL Calibration Intermediate Solution 1. Each ICAL level should contain an equal amount of 400uL methanol . A typical calibration curve would be:

Inter. Solution 1 50 µg/mL (µL/40 mL)	Additional methanol (µL/40 mL)	Final Conc. (µg/L)
2	398	2.5
4	396	5.0
16	384	20
40	360	50
80	320	100
120	280	150
160	240	200

7.3.1.4 ETBE: Prepare working standards at a minimum of six concentration levels, ranging from 2.5-200 µg/L, by diluting the 50 µg/mL Calibration Intermediate Solution 2. A typical calibration curve would be:

Inter. Solution 2 50 µg/mL (µL/40 mL)	Final Conc. (µg/L)
2	2.5
4	5.0
16	20
40	50
80	100
160	200

7.3.1.5 Prepare the calibration standards as directed in the table above. Calibration should be done using the same introduction technique that will be used for the samples. If a sample volume larger than 5 mL is to be used, i.e. 25 mL, the curve should be developed at this volume.

7.3.1.6 The average RF must be calculated using the RF values calculated for each compound from the initial calibration curve. Check the five System Performance Check Compounds (SPCCs) to be sure the minimum RF criteria have been met (see Table 3). If the minimum RFs are not met, a new initial calibration curve must be generated. the system must be evaluated and corrective action taken before sample analysis can begin. Examples of corrective action may include inspecting the system for leaks, checking for errors in standard preparation or degradation of the standard mix, or evaluating the chromatography system for active sites.

7.3.1.7 The percent relative standard deviation should be less than 15% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) must be less than 30%. See Table 4 for the CCCs. If a CCC has a %RSD > 30%, a new initial calibration must be generated.

7.3.1.8 If the %RSD of any compound is 15% or less, then the relative RF is assumed to be constant over the calibration range, and the average relative RF may be used for quantitation. A minimum of five calibration points may be used to define the working range.

7.3.1.9 If the %RSD of any compound is greater than 15%, construct calibration curves of area ratio (A/A_{is}) versus concentration using a first order or higher order regression fit of the calibration points. A first order, or linear fit, may be used with a minimum of five calibration points. A second order, or quadratic fit, requires six calibration points. A correlation coefficient of 0.99 or better is required for each curve fit. The analyst should select the regression order that introduces the least error into the quantitation.

7.3.1.10 Calibration curve calculations are found in the QA Manual.

7.3.1.11 Reporting limit verification (RLV) is checked with each calibration curve or monthly at a minimum, by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.3.2 Daily GC/MS Calibration

7.3.2.1 The GC/MS tune check standard containing 50 ng of BFB must meet the Table 1 criteria. The standard must be run and meet the criteria every 12 hours.

7.3.2.2 A mid-level calibration standard must be analyzed every 12 hours. The SPCCs must meet the minimum response criteria on Table 3. If the minimum RFs are not met, the system must be evaluated and corrective action taken before sample analysis can begin. Examples of corrective action may include inspecting the system for leaks, checking for errors in standard preparation or degradation of the standard mix, or evaluating the chromatography system for active sites.

7.3.2.3 Use the Calibration Check Compounds to check the validity of the initial calibration. Calculate the percent drift using:

$$\%Drift = \frac{(C_I - C_c)}{(C_I)}(100)$$

C_I = Calibration Check Compound standard concentration
C_c = Measured concentration using selected quantitation method

7.3.2.4 If the percent difference for each CCC is $\leq$ 20%, the initial calibration is assumed to be valid. If the minimum RFs are not met, the system must be evaluated and corrective action taken before sample analysis can begin. Examples of corrective action may include inspecting the system for leaks, checking for errors in standard preparation or degradation of the standard mix, or evaluating the chromatography system for active sites.

7.3.2.5 Evaluate the internal standard responses and retention times. If the retention time changes by more than 30 seconds from the mid-point of the last initial calibration curve or the Extracted Ion Current Profile (EICP) area for any internal standard changes by a factor of two (- 50% to + 100%) from the mid-point of the last initial calibration curve, the chromatographic system must be inspected for malfunctions and corrections made as required before samples can be analyzed.

7.3.2.6 If any of the daily calibration criteria are not met, minor corrective maintenance may be performed on the system and the calibration check standard re-run. If major corrective action is required, such as cleaning the source or replacing the chromatograph column, a new initial calibration needs to be generated before samples could be analyzed.

7.3.2.7 A method blank must be analyzed prior to the analysis of samples. The method blank should not contain target analytes above the reporting limit. If the method blank does contain analytes above the RL the sample batch is reanalyzed, if possible.

7.4 Analysis

7.4.1 GC/MS Conditions:

7.4.1.1 Mass range: 35-300 amu

7.4.1.2 Scan time: approximately 2.6 scans/sec

7.4.1.3 Initial temperature: 35 °C, hold for 4 minutes

7.4.1.4 Temperature program: 35-180 °C at 8 °C/minute

7.4.1.5 Final temperature: 180 °C, hold until at least one minute after the 1,2,3-trichlorobenzene has eluted

7.4.1.6 Injector temperature: 180-250 °C

7.4.1.7 Interface temperature: 250-300 °C

7.4.1.8 Carrier gas: Helium at approximately 0.6 mL/min at constant flow

7.4.1.9 Purge flow: Approximately 40 mL/minute

7.4.1.10 Desorb flow: Approximately 20 mL/minute

7.4.1.11 Split ratio: Approximately 40:1 (HP 5890 Series II GC), Approximately 100:1 (Thermo Focus GC)

7.4.1.12 Purge time: 11 minutes

7.4.1.13 Desorb time: 0.5 minute (Encon sample concentrator), 1 minute (O.I Analytical Eclipse sample concentrator)

7.4.1.14 Desorb temperature: 250 °C (Encon sample concentrator), 190 °C (O.I Analytical Eclipse sample concentrator)

7.4.1.15 Bake time: 10 minutes

7.4.1.16 Bake temperature: 260 °C (Encon sample concentrator), 210 °C (O.I Analytical Eclipse sample concentrator)

7.4.2 All samples must be allowed to warm to ambient temperature before analysis.

7.4.3 Load vials into the auto sampler. Program the method to analyze a 5 mL sample volume and add 1 µL each of the surrogate and internal standard solutions.

7.4.3.1 The auto sampler adds 1 µL of the 250 µg/mL Internal Standard Solution to each 5 mL calibration standard, blank, sample, MS/MSD, and LCS to obtain a 50 µg/L final concentration.

7.4.3.2 The auto sampler adds 1 µL of the 250 µg/mL auto sampler Surrogate Standard to each 5 mL blank, sample, MS/MSD, and LCS to obtain a 50 µg/L final concentration.

7.4.3.3 The auto sampler does not deliver precisely 1µL of surrogate. This amount tends to be either slightly higher or lower after major maintenance in the auto sampler surrogate valve system. An auto sampler surrogate adjustment calibration must be performed each time the auto sampler surrogate valve system has been serviced.

7.4.3.3.1 Run three blanks with the surrogates delivered by auto sampler and three blanks with the surrogates manually injected into 40ml vials of water at 50ug/L.

7.4.3.3.2 Average the auto sampler and 40 mL vial surrogate concentrations.

7.4.3.3.3 Obtain a correction factor by dividing the auto sampler surrogate concentration average by the manual 40 mL surrogate concentration average.

7.4.3.3.4 Multiply the correction factor by the expected concentration. This yields the actual amount delivered by the auto sampler. Use this factor to calculate the actual surrogate recoveries.

7.4.4 For the samples in each analytical batch selected for spiking, add 40 µL of the 50 µg/mL Spike Standard. Final concentration will be 50 µg/L. A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

NOTE: If running a dual water batch of 624 and 8260B, use 624 spiking amounts.

7.4.5 If the sample concentration exceeds the initial calibration range, dilute the sample and reanalyze.

7.4.6 The pH of water samples is checked after analysis. The pH should be <2. If the pH isn't <2, it should be noted in the client's report.

7.5 Calculation

7.5.1 Qualitative analysis

7.5.1.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. Use a mid-level initial calibration standard to obtain standard reference spectra. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. The relative intensities of the ions should agree within ± 30% between the sample and reference spectrum.

7.5.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification.

7.5.2 Quantitative analysis

7.5.2.1 Quantitate using the internal standard technique. Use the internal standard preceding the analyte (see Table 2). Quantitation is based on the integrated abundance from the EICP of the primary characteristic ion.

7.5.2.2 If the %RSD of a compound's relative response factor is 15% or less, then the concentration in the sample may be determined using the average RF from initial calibration data and the following equation:

$$\textit{Water Concentration}\,(\mu g/L) = \frac{(A_x)(I_{is})(F)}{(A_{IS})(RF)}$$

A_x = Area of characteristic ion being measured
I_{is} = Amount of internal standard injected (µg/L)
F = Dilution factor

A_{is} = Area of characteristic ion for the internal standard
RF = Average response factor for compound being measured

$$Soil\ Concentration\ (mg/kg) = \frac{(A_x)(I_{is})(V_t)}{(A_{is})(RF)(V_i)(W_s)(D)}$$

A_x, I_{is}, A_{is}, RF = Same as for water
V_t = Volume of the total extract (mL)
V_i = Volume of extract added (mL) for purging
W_s = Weight of sample extracted or purged (g)
D = % dry weight of sample/100, or 1 for wet weight basis

7.5.2.3 Alternatively, the regression line fitted to the initial calibration may be used for the determination of the analyte concentration.

7.5.2.4 Where applicable, an estimate of concentration for noncalibrated components (Tentatively Identified Compounds – TIC) in the sample can be made. The concentration should be reported as an estimate assuming a response factor of 1 using the nearest internal standard.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, when there is a change in the test method that may affect how the test is performed or when there is a major change in instrumentation. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the reporting limit or the sample batch is reanalyzed if possible. If it is not possible to reanalyze, the data will be flagged where appropriate. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Accuracy control limits are generated for LCS, MS and surrogates. In-house control charts are generated semi-annually, using 20 Percent Recovery points, as follows:

9.3.1.1 Upper and Lower Control Limit = Mean ± 3s

9.3.1.2 Upper and Lower Warning Limit = Mean ± 2s

9.3.1.3 s = Standard deviation

9.3.2 Precision control limits are generated for MS/MSD. LCS/LCSD will be substituted if there isn't enough sample. In-house control charts are generated semi-annually, using 20 RPD points, as follows:

9.3.2.1 Control Limit = Mean + 3s

9.3.2.2 Warning Limit = Mean + 2s

9.3.2.3 s = Standard deviation

9.3.2.4 RPD = Relative Percent Difference

9.3.3 In-house limits are used for compliance, as the method does not list limits. The limits will be reviewed for reasonableness before using them within the laboratory.

9.3.3.1 For permanent gases and compounds eluting after sec-butylbenzene, in-house limits that calculate narrower than 75.0-125% are set to 75.0-125% (i.e. in-house limits = 79.8-126%, limits are set at 75.0-126%). In-house limits that calculate wider than 70.0-130% are set to 70.0-130% (i.e. in-house limits = 65.8-132%, limits are set at 70.0-130%).

9.3.3.2 For 2,2-dichloropropane, in-house limits that calculate narrower than 70.0-130% are set to 70.0-130% (i.e. in-house limits = 79.8-126%, limits are set at 70.0-130%). In-house limits that calculate wider than 60.0-140% are set to 60.0-140% (i.e. in-house limits = 55.8-142%, limits are set at 60.0-140%).

9.3.3.3 For all other parameters, in-house limits that calculate narrower than 80.0-120% are set to 80.0-120% (i.e. in-house limits = 85.8-122%, limits are set at 80.0-122%). In-house limits that calculate wider than 75.0-125% are set to 75.0-125% (i.e. in-house limits = 75.8-132%, limits are set at 75.8-125%).

9.3.3.4 In-house precision limits that calculate narrower than 20% RPD are set to 20% (i.e. in-house limits = 15.3%, limits are set at 20%). In-house precision limits that calculate wider than 25% are set to 25% (i.e. in-house limits = 28.5%, limits are set at 25%).

9.3.4 QC calculations are found in the QA Manual.

9.3.5 LCS, MS/MSD and surrogates are reviewed.

9.3.6 If the LCS data are outside the limits, a method nonconformance is filled out and the sample batch is re-extracted and/or re-analyzed if possible. If the batch cannot be re-analyzed, the data are flagged and/or a case narrative is written for all client reports within the batch.

9.3.7 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS may be flagged in the case narrative of the report.

9.3.8 If a sample surrogate is outside the limits, the sample is reanalyzed if possible. If the sample cannot be reanalyzed, the data is flagged next to the actual result in the report.

10. REPORTING

10.1 Soil samples results are reported in mg/kg on a dry weight basis.

10.2 Water sample results are reported in µg/L.

10.3 The reported result is rounded to two significant figures.

10.4 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 EPA 5000, 5030B, 5035, 8000B (MN), 8000C (AZ), and 8260B

12.2 Vendor equipment manuals

TABLE 1 - BFB Key Ions and Ion Abundance Criteria

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	<2% of Mass 174
174	>50% of Mass 95
175	5-9% of Mass 174
176	>95% But <101% of Mass 174
177	5-9% of Mass 176

TABLE 2 – Volatile Compounds

Compounds	Retention Time (min.)	Primary Ion
Pentafluorobenzene (IS)	7.61	168
Dichlorodifluoromethane	1.90	85
Chloromethane	2.09	50
Vinyl chloride	2.25	62
Bromomethane	2.61	94
Chloroethane	2.73	64
Trichlorofluoromethane	3.13	101
Dichlorofluoromethane	3.04	67
Ethyl Ether	3.50	59
1,1-Dichloroethene	3.83	96
1,1,2-Trichlorotrifluoroethane	3.88	151
Acetone	3.90	58
Allyl chloride	4.41	76
Methylene chloride	4.62	84
trans-1,2-Dichloroethene	5.08	96
MTBE	5.12	73
1,1-Dichloroethane	5.75	63
Ethyl-t-butylether	6.33	87
2,2-Dichloropropane	6.69	77
cis-1,2-Dichloroethene	6.71	96
2-Butanone	6.74	72
Bromochloromethane	7.08	128
THF	7.16	72
Chloroform	7.24	83
1,1,1-Trichloroethane	7.51	97
Carbon tetrachloride	7.78	117
1,1-Dichloropropene	7.77	75
1,4-Difluorobenzene (IS)	8.74	114

TABLE 2 – Volatile Compounds (continued)

Compounds	Retention Time (min.)	Primary Ion
Dibromofluoromethane (surr.)	7.49	113
Toluene-d8 (surr.)	11.11	98
4-Bromofluorobenzene (surr.)	15.53	95
Benzene	8.08	78
1,2-Dichloroethane	8.12	62
Trichloroethene	9.14	95
1,2-Dichloropropane	9.47	63
Dibromomethane	9.66	93
Bromodichloromethane	9.95	83
cis-1,3-Dichloropropene	10.68	75
Methyl Isobutyl Ketone	10.95	85
Toluene	11.21	92
trans-1,3-Dichloropropene	11.59	75
1,1,2-Trichloroethane	11.88	83
Tetrachloroethene	12.12	166
2-Bromo-1-chloropropane (IS)	11.70	77
1,3-Dichloropropane	12.15	76
Dibromochloromethane	12.53	129
1,2-Dibromoethane	12.68	107
Chlorobenzene	13.54	112
1,1,1,2-Tetrachloroethane	13.69	131
Ethylbenzene	13.75	91
m&p-Xylene	13.96	106
o-Xylene	14.63	106
Styrene	14.65	104
Bromoform	14.94	173
1,4-Dichlorobenzene-d4 (IS)	17.56	152
Isopropylbenzene	15.28	105
Bromobenzene	15.76	156
1,1,2,2-Tetrachloroethane	15.81	83
1,2,3-Trichloropropane	15.86	75
n-Propylbenzene	16.01	91
2-Chlorotoluene	16.13	91
4-Chlorotoluene	16.32	91
1,3,5-Trimethylbenzene	16.33	105
tert-Butylbenzene	16.90	119
1,2,4-Trimethylbenzene	16.98	105
sec-Butylbenzene	17.29	105
1,3-Dichlorobenzene	17.44	146
4-Isopropyltoluene	17.56	119
1,4-Dichlorobenzene	17.61	146

TABLE 2 – Volatile Compounds (continued)

Compounds	Retention Time (min.)	Primary Ion
1,2-Dichlorobenzene	18.25	146
n-Butylbenzene	18.29	91
1,2-Dibromo-3-chloropropane	19.64	75
1,2,4-Trichlorobenzene	21.16	180
Hexachlorobutadiene	21.52	225
Naphthalene	21.58	128
1,2,3-Trichlorobenzene	22.02	180

TABLE 3 - System Performance Check Compounds

Compounds	Minimum Response Factor
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

TABLE 4 – Calibration Check Compounds

Compounds	%RSD
1,1-Dichloroethene	<30
Chloroform	<30
1,2-Dichloropropane	<30
Toluene	<30
Ethylbenzene	<30
Vinyl chloride	<30

Appendix A

Initial Demonstration of Capability (IDC)
Determination of Volatile Organic Compounds by GC/MS

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four replicate standards of the LCS in Ottawa sand and/or lab-grade water and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using form 'IDC 4 rep with RPD', the individual recoveries in concentration and %, the mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: LCS limits
   Precision: LCS limits

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Determination of Volatile Organic Compounds by GC/MS

Parameter	Water MDL (µg/L)	Water RL (µg/L)	Soil MDL (mg/kg)	Soil RL (mg/kg)
Dichlorodifluoromethane	0.25	5.0	0.099	0.50
Chloromethane	0.24	2.5	0.061	0.25
Vinyl chloride	0.21	1.0	0.065	0.25
Bromomethane	0.32	5.0	0.11	0.50
Chloroethane	0.27	2.5	0.037	0.25
Trichlorofluoromethane	0.29	1.0	0.035	0.25
*Dichlorofluoromethane	0.21	1.0	0.038	0.25
*Ethyl Ether	0.27	5.0	0.038	0.50
1,1-Dichloroethene	0.24	1.0	0.025	0.25
*1,1,2-Trichlorotrifluoroethane	0.25	1.0	0.068	0.25
*Acetone	3.8	20	0.19	2.0
*Allyl chloride	0.51	5.0	0.058	0.50
Methylene chloride	0.85	5.0	0.058	1.0
trans-1,2-Dichloroethene	0.056	1.0	0.030	0.25
*MTBE	0.16	1.0	0.016	0.25
1,1-Dichloroethane	0.15	1.0	0.023	0.25
^ETBE	---	5.0	---	0.50
2,2-Dichloropropane	0.66	5.0	0.060	0.50
cis-1,2-Dichloroethene	0.12	1.0	0.026	0.25
*2-Butanone	0.65	20	0.088	2.0
Bromochloromethane	0.20	1.0	0.037	0.25
*THF	0.75	20	0.12	2.0
Chloroform	0.15	1.0	0.021	0.25
1,1,1-Trichloroethane	0.16	1.0	0.032	0.25
Carbon tetrachloride	0.15	1.0	0.034	0.25
1,1-Dichloropropene	0.14	1.0	0.021	0.25
Benzene	0.071	1.0	0.015	0.25
1,2-Dichloroethane	0.26	1.0	0.027	0.25
Trichloroethene	0.18	1.0	0.041	0.25
1,2-Dichloropropane	0.19	1.0	0.031	0.25
Dibromomethane	0.24	2.5	0.029	0.25
Bromodichloromethane	0.23	1.0	0.027	0.25
cis-1,3-Dichloropropene	0.18	1.0	0.022	0.25
*Methyl Isobutyl Ketone	0.40	5.0	0.082	0.50

* = Additional compounds from Minnesota list
^ = Additional compounds not found in typical LEGEND 8260B list

Appendix B (continued)

Method Detection Limits and Reporting Limits
Determination of Volatile Organic Compounds by GC/MS

Parameter	Water MDL (µg/L)	Water RL (µg/L)	Soil MDL (mg/kg)	Soil RL (mg/kg)
Toluene	0.10	1.0	0.016	0.25
trans-1,3-Dichloropropene	0.14	1.0	0.023	0.25
1,1,2-Trichloroethane	0.19	1.0	0.034	0.25
Tetrachloroethene	0.28	1.0	0.028	0.25
1,3-Dichloropropane	0.16	1.0	0.022	0.25
Dibromochloromethane	0.26	2.5	0.034	0.25
1,2-Dibromoethane	0.15	2.5	0.022	0.25
Chlorobenzene	0.20	1.0	0.019	0.25
1,1,1,2-Tetrachloroethane	0.29	1.0	0.059	0.25
Ethylbenzene	0.28	1.0	0.011	0.25
m&p-Xylene	0.57	2.0	0.047	0.50
o-Xylene	0.19	1.0	0.025	0.25
Styrene	0.21	1.0	0.020	0.25
Bromoform	0.26	5.0	0.027	0.50
Isopropylbenzene	0.12	1.0	0.019	0.25
Bromobenzene	0.082	1.0	0.027	0.25
1,1,2,2-Tetrachloroethane	0.16	1.0	0.026	0.25
1,2,3-Trichloropropane	0.22	2.5	0.024	0.25
n-Propylbenzene	0.094	1.0	0.021	0.25
2-Chlorotoluene	0.081	1.0	0.014	0.25
4-Chlorotoluene	0.11	1.0	0.021	0.25
1,3,5-Trimethylbenzene	0.11	1.0	0.020	0.25
tert-Butylbenzene	0.091	1.0	0.018	0.25
1,2,4-Trimethylbenzene	0.072	1.0	0.018	0.25
sec-Butylbenzene	0.11	1.0	0.017	0.25
1,3-Dichlorobenzene	0.15	1.0	0.026	0.25
p-Isopropyltoluene	0.14	2.5	0.014	0.25
1,4-Dichlorobenzene	0.081	1.0	0.017	0.25
1,2-Dichlorobenzene	0.12	1.0	0.020	0.25
n-Butylbenzene	0.15	2.5	0.027	0.25
1,2-Dibromo-3-chloropropane	0.40	5.0	0.069	0.50
1,2,4-Trichlorobenzene	0.30	5.0	0.027	0.50
Hexachlorobutadiene	0.42	10	0.069	1.0
Naphthalene	0.30	5.0	0.055	0.50
1,2,3-Trichlorobenzene	0.35	5.0	0.061	0.50

DOCUMENT REVIEW

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REVIEWER:	Sonny Hang
DATE:	03/30/11

SECTION	CHANGE	RATIONALE
All	No changes made	Annual review

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**DETERMINATION OF SEMI-VOLATILE COMPOUNDS IN SOIL/SOLID BY GC/MS**
SOP NO.:	**LABENV-021.10**

Original Information		
Prepared by:	Sandy McDonald	Date: 03/13/96
Technical Review:		Date:
QA/QC Coordinator:	Sharon Cenis	Date: 03/25/96
Authorized by:	Cheryl Sykora	Date: 03/29/96

Revision Information		
Supersedes:	LABENV-021.9	Date: 03/17/09
Revised by:	Van Pham	Date: 05/06/10
Signature:	_____	Date: _____
Technical Review:	Triet Le	Date: 05/06/10
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 05/06/10
Signature:	_____	Date: _____

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Revised by:	Van Pham	Date:
Signature:	Van Pham	Date: 5/6/10
Technical Review:	Triet Le	Date:
Signature:	T. L	Date: 05/06/10
Authorized by:	Cheryl Sykora	Date:
Signature:	Cheryl Sykora	Date: 5/10/10

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SOP TITLE: DETERMINATION OF SEMI-VOLATILE COMPOUNDS IN SOIL BY GC/MS

1. PURPOSE

1.1 This document defines the preparation and analysis for semi-volatile compounds in soil and solid matrices by Gas Chromatography/Mass Spectrometry (GC/MS). This procedure can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused silica capillary column coated with a slightly polar silicone. The SOP is applicable to samples typically analyzed by EPA 8270C.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in this SOP.

2.3 An analyst experienced in the use of gas chromatograph/mass spectrometers, skilled in the interpretation of mass spectra, and trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is applicable to soil/solid samples only.

3.2 The Sonication Method may be used when a sample has the potential to be detrimental to the ASE (e.g. tar samples, fine sediments, etc.).

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard and exposure to these chemicals minimized.

4.3 Follow standard laboratory safety procedures.

4.4 A lab coat and safety glasses should be worn during sample and standard preparation.

4.5 When working with organic compounds, wear chemical resistant gloves.

4.6 Prepare stock and standard solutions in a hood.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Soil samples should be collected in unweighed 4 oz. glass jars with Teflon-lined caps and stored at 4 ± 2 ºC.

5.3.1 Soil/sediment samples – Decant and discard any water layer. Mix thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

5.3.2 Dry waste samples – Grind so that sample may pass through a 1 mm sieve. Grind enough sample to yield at least 15 grams.

5.4 The recommended holding time for soil samples is 14 days until extraction and analysis within 40 days of extraction.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 HP 5890 Series II Gas Chromatograph (GC) with data processing equipment, or equivalent

6.2 HP 5972A Mass Selective Detector (MSD) with scan range of 35 to 500 amu using 70 volts electron energy in the electron impact ionization mode, or equivalent

6.3 Column – 30m x 0.25 mm ID (or 0.32 mm ID) 0.25 µm film thickness silicone-coated fused silica capillary column (DB-5MS or equivalent)

6.4 Nitrogen evaporator – N-EVAP, or equivalent

6.5 Ultrasonic Disrupter - Bronson Sonifier 450, or equivalent

6.6 Microliter syringes – 10, 25, 100, 250, 500, and 1000 µL

6.7 Volumetric flasks – 5, 10, 25, and 50 mL

6.8 Serum Bottles – amber glass with Teflon-lined crimp tops

6.9 Beaker – 400mL

6.10 Glass funnel with Pyrex glass wool at bottom

6.11 Side arm vacuum flask – 500 mL

6.12 Buchner funnel

6.13 Filter paper - Whatman No. 41 or equivalent

6.14 Kuderna-Danish (K-D) concentrator – 10 mL, graduated

6.15 Kuderna-Danish (K-D) flask – 500 mL

6.16 Snyder column – three ball macro

6.17 PTFE solvent rinsed boiling beads, or equivalent

6.18 Water bath

6.19 Autosampler vials – 2 mL amber glass with Teflon lined crimp tops

6.20 Graduated disposable pipets – 1 or 2 mL

6.21 Mortar and pestle, or equivalent

6.22 Balance, capable of reading to 0.01 g

6.23 Turbo Vap II and associated parts and glassware

6.24 Accelerated Solvent Extractor (ASE) and associated parts and glassware

6.25 Disposable aluminum weighing dishes – prerinsed with hexane and methylene chloride

6.26 Anhydrous Sodium Sulfate (Na_2SO_4) – muffle at 400 °C for four hours before using

6.27 Ottawa Sand (20-30 mesh) or equivalent – muffle at 400 °C for four hours before using

6.28 Hydromatrix® – muffle at 400 °C for four hours before using

6.29 Methanol – pesticide grade, or equivalent

6.30 Methylene chloride, CH_2Cl_2 – pesticide grade, or equivalent

6.31 Acetone – pesticide grade, or equivalent

6.32 Methylene chloride/Acetone (1:1) (v/v) – for sonication preparation technique

6.33 Methylene chloride/Acetone (3:1) (v/v) – for ASE preparation technique

6.34 GC/MS Tune Check Stock – 500 µg/mL each of DFTPP, Benzidine, 4.4'-DDT, and pentachlorophenol, Absolute Standards, Inc. #43030, or equivalent

6.35 Calibration Stock 1 – 2000 µg/mL each of bis(2-chloroethoxy) methane, bis(2-chloroethyl) ether, bis(2-ethylhexyl) phthalate, bis(2-chloroisopropyl)ether, 4-bromophenyl phenyl ether, butyl benzyl phthalate, 4-chlorophenyl phenyl ether, diethyl phthalate, dimethyl phthalate, di-n-butyl phthalate, di-n-octyl phthalate, n-nitrosodimethylamine, n-nitrosodi-n-propylamine, and n-nitrosodiphenylamine, Absolute #10001, or equivalent

6.36 Calibration Stock 2 – 2000 µg/mL each of acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, carbazole, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene Absolute #10007, or equivalent

6.37 Calibration Stock 3 – 2000 µg/mL each of azobenzene (1,2-diphenylhydrazine), 2-chloronaphthalene, 1,2-dichlorobenzene, 1,4-dichlorobenzene, 1,3 dichlorobenzene, 2,6-dinitrotoluene, 2,4-dinitrotoluene, hexachlorobenzene, hexachlorobutadiene, hexachlorocyclopentadiene, hexachloroethane, isophorone, nitrobenzene, and 1,2,4-trichlorobenzene, Absolute #10002, or equivalent

6.38 Calibration Stock 4 – 2,000 µg/mL each of aniline, benzyl alcohol, 4-chloroaniline, dibenzofuran, 2-methylnaphthalene, 2-nitroaniline, 3-nitroaniline, and 4-nitroaniline, Absolute #10005, or equivalent

6.39 Calibration Stock 5 – 2,000 µg/mL each of benzoic acid, 2-methylphenol, 4-methylphenol, and 2,4,5-trichlorophenol, Absolute #10004, or equivalent

6.40 Calibration Stock 6 – 2,000 µg/mL each of 4-chloro-3-methylphenol, 2-chlorophenol, 2,4-dichlorophenol, 2,6-dichlorophenol, 2,4-dimethylphenol, 2,4-dinitrophenol, 2-methyl-4,6-dinitrophenol, 2-nitrophenol, 4-nitrophenol, pentachlorophenol, phenol, 2,4,6-trichlorophenol, and 2,3,4,6-tetrachlorophenol, Absolute #10018, or equivalent

6.41 Calibration Stock 7 – 2,000 µg/mL each of benzidine and 3,3'-dichlorobenzidine, Absolute #10006, or equivalent

6.42 Calibration Stock 8 – 1,000 µg/mL of pyridine, Absolute #70260, or equivalent

6.43 Internal Standard Stock – 4,000 µg/mL each of acenaphthene-d_{10}, chrysene-d_{12}, 1,4-dichlorobenzene-d_4, naphthalene-d_8, perylene-d_{12}, and phenanthrene-d_{10}, Absolute #10009, or equivalent

6.44 Calibration Surrogate Stock 1 – 10,000 µg/mL each of 2-flurophenol, phenol-d6, and 2,4,6-tribromophenol, Absolute #21015, or equivalent

6.45 Calibration Surrogate Stock 2 – 5,000 µg/mL each of nitrobenzene-d_5, 2-fluorobiphenyl, and terphenyl-$d_{14,}$ Absolute #21016, or equivalent

6.46 Sample/Second Source Surrogate Stock 1 – 10,000 µg/mL each of 2-flurophenol, phenol-d6, and 2,4,6-tribromophenol, Restek #31087, or equivalent

6.47 Sample/Second Source Surrogate Stock 2 – 5,000 µg/mL each of nitrobenzene-d_5, 2-fluorobiphenyl, and terphenyl-$d_{14,}$ Restek #31086, or equivalent

6.48 Spike Stock 1 – 10,000 µg/mL each of pentachlorophenol, phenol, 2-chlorophenol, 4-chloro-3-methylphenol, and 4-nitrophenol, Restek #31071, or equivalent

6.49 Spike Stock 2 – 5,000 µg/mL each of 1,2,4-trichlorobenzene, acenaphthene, 2,4-dinitrotoluene, pyrene, n-nitrosodi-n-propylamine, and 1,4-dichlorobenzene, Restek #31084, or equivalent

6.50 Spike Stock 3 – 2,000 µg/mL each of acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, 2-methylnaphthalene, naphthalene, phenanthrene, and pyrene, Absolute #50003, or equivalent

6.51 Second Source Stock 9 - 1000ug/ml of all 8270 components except for benzidine, 3,3'-dichlorobenzidine, 2,6-dichlorophenol, surrogates, and benzoic acid , Restek # 31850 or equivalent

6.52 Second Source Stock 10 - 2000ug/ml each of benzidine and 3,3'-dichlorobenzidine, Restek # 31834 or equivalent

6.53 Second Source Stock 11 - 1000ug/ml of 2,6-dichlorophenol, Restek # 31409 or equivalent

6.54 Second source stock 12 - 1000ug/ml of benzoic acid, Absolute #70034, or equivalent

6.55 Calibration Intermediate Solution – combine 300 µL of the 2,000 µg/mL Calibration Stocks 1-7, 600 µL of the 1,000 µg/mL Calibration Stock 8, 60 µL of the 10,000 µg/mL Surrogate Stock 1, 120 µL of the 5,000 µg/mL Surrogate Stock 2, and 120 µL of methylene chloride (3 mL final volume) to produce a 200 µg/mL Calibration Intermediate Solution

6.56 GC/MS Tune Check Standard – dilute 100 µL of the 500 µg/mL GC/MS Tune Check Stock with 900 µL of methylene chloride to produce a 50 µg/mL GC/MS Tune Check Standard

6.57 Sample Surrogate Standard – dilute 0.5 mL of the 10,000 µg/mL Surrogate Stock 1 and 1 mL of the 5,000 µg/mL Surrogate Stock 2 to a final volume of 50 mL with methanol to produce a 100 µg/mL Sample Surrogate Standard

6.58 Spike Standard – dilute 0.5 mL of the 10,000 µg/mL Spike Stock 1, 1 mL of the 5,000 µg/mL Spike Stock 2, and 2.5 mL of the 2,000 µg/mL Spike Stock 3 to a final volume of 50 mL with methanol to produce a 100 µg/mL Spike Standard

6.59 Second Source ICV - combine 50 µL of the 1,000 µg/mL Stocks 9, 11 and 12, 25 µL of the 2000 µg/mL Stock 10, 5.0 µL of the 10,000 µg/mL Sample Surrogate Stock 1, 10 µL of the 5,000 µg/mL Sample Surrogate Stock 2, and 810 µL of methylene chloride (1 mL final volume) to produce a 50 µg/mL Second Source Calibration Solution. Add 10 µL of the 4,000 µg/mL Internal Standard Stock prior to analysis to produce an ISTD concentration of 40 µg/mL.

6.60 All solutions and standards should be stored in a freezer at $\leq$ -10 ºC and should be freshly prepared each year, or sooner if check standards or continuing calibration standards indicate a problem.

7. PROCEDURE

7.1 Preparation of Samples – ASE Technique

7.1.1 The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.1.2 Assemble extraction cell by screwing a cell end cap onto cell body

7.1.3 Place 2 filters on top of the open end of the cell body. Use the black cylindrical insertion tool to push the filters to the bottom of the assembled cell body. Very fine soils may require three filters.

7.1.4 Weigh approximately 15 g of sample into a prerinsed disposable aluminum dish. Record the weight to the nearest 0.01g on the extraction sheet.

7.1.5 Use a 1:1 Ottawa Sand:Hydromatrix® mixture for the blank and Laboratory Control Sample (LCS). A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

7.1.6 Mix the sample with Hydromatrix®, or equivalent, using approximately a 1:1 ratio by volume, until free-flowing. Depending on the matrix, the amount of sample may need to be reduced.

7.1.7 Place the extraction cell funnel on open end of cell body. Load sample into cell through funnel. Gently tap cell on hard surface to pack sample evenly, and to reduce void volume.

7.1.8 All samples should come within 1 cm of the top of the vessel. If a sample does not, use sand to fill.

7.1.9 When the transfer is complete, remove the funnel. Add 1.0 mL of the 100 µg/mL Sample Surrogate Standard to each sample, blank, and spike sample. Final concentration will be 6.67 mg/kg, assuming a 1.0 mL final volume, 1 µL injection, and 15 g sample amount.

7.1.10 For the samples in each analytical batch selected for spiking, add 1.0 mL of the 100 µg/mL Spike Standard. Final concentration will be 6.67 mg/kg, assuming a 1.0 mL final volume, 1 µL injection, and 15 g sample amount.

7.1.11 Place filter paper on top of the sample. Remove any debris from cell threads and screw the second end cap onto open end of the cell body.

7.1.12 Place completed extraction cell into position #1 on the ASE. Place the corresponding collection vial into position #1 below.

7.1.13 Repeat the above steps for additional samples.

7.1.14 Fill solvent bottle with the 3:1 methylene chloride:acetone. Record their lot numbers on the solvent bottle.

7.1.15 Make sure pressure on gas tank is set to 180 psi. Make sure that solvent bottle pressure is 10 psi, system air pressure is 50 psi, and compression oven pressure is 130 psi. Record the pressure readings in the soil extraction log.

7.1.16 After the ASE extraction, assemble a K-D apparatus by attaching a 10 mL K-D concentrator to a 500 mL K-D flask for Nitrogen Blowdown or a 200 mL Turbo Vap II concentration vial. The Nitrogen Blowdown technique is used when there is over 200 mL of extract.

7.1.17 Decant extraction solvent through a methylene chloride rinsed funnel filled one-third full with sodium sulfate and plugged with glass wool. Rinse the collection vial three times with methylene chloride to complete quantitative transfer. Collect the extract in the assembled K-D apparatus or a 200 mL Turbo Vap II concentration vial.

7.1.18 K-D Technique / Nitrogen Blowdown

7.1.18.1 Add one solvent rinsed boiling bead to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-85 °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot vapor. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

7.1.18.2 When the apparent volume of liquid reaches 5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool. Remove the Snyder column.

7.1.18.3 Put the K-D concentrator in a warm bath (35 ºC) and evaporate the solvent volume to less than 1 mL using a gentle stream of clean, dry nitrogen. During evaporation, the tube solvent level must be positioned to avoid water condensation (i.e. the solvent level should be below the level of the water bath). Do not allow the extract to go dry as this may result in a loss of analytes. Using a disposable graduated pipet, adjust the final volume to 1.0 mL with methylene chloride and mix the extract completely.

7.1.18.4 Transfer to a 2 mL autosampler vial. Store in freezer until analysis.

7.1.19 Turbo Vap II

7.1.19.1 Place the Turbo Vap collection tube in the Turbo Vap.

7.1.19.2 Set the water bath temperature to 40 °C and the pressure to 8-12 psi.

7.1.19.3 Set the Turbo Vap to monitor by 'sensor' to achieve a final volume of approximately 1 mL.

7.1.19.4 Using a graduated disposable pipet, adjust the final volume to 1.0 mL with methylene chloride and mix the extract completely.

7.1.19.5 Transfer to a 2 mL autosampler vial. Store in freezer until analysis.

7.2 Preparation of Samples – Sonication Technique

7.2.1 The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.2.2 Weigh approximately 15 g of sample into a prerinsed 250 mL beaker. Record the weight to the nearest 0.01 g on the extraction sheet.

7.2.3 Add approximately 15 g of anhydrous muffled Na_2SO_4 and mix the sample well. More sodium sulfate may be added until the sample is free flowing.

7.2.4 Add 1.0 mL of the 100 µg/mL Sample Surrogate Standard to each sample, blank, and spike sample. Final concentration will be 6.67 mg/kg, assuming a 1.0 mL final volume, 1 µL injection, and 15 g sample amount.

7.2.5 For the samples in each analytical batch selected for spiking, add 1.0 mL of the 100 µg/mL Spike Standard. Final concentration will be 6.67 mg/kg, assuming a 1.0 mL final volume, 1 µL injection, and 15 g sample amount. A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

7.2.6 Immediately add 100 mL of 1:1 methylene chloride/acetone.

7.2.7 Place the bottom surface of the tip of the 2" disrupter horn about 2" below the surface of the solvent, but above the soil/sediment layer.

7.2.8 Extract ultrasonically for about 3 minutes, with output control knob set at 10, mode switch on Pulse and percent-duty cycle knob set at 50%.

7.2.9 Quantitatively transfer the solvent into a glass funnel containing a filter paper and sitting atop a 500 mL K-D apparatus. Repeat steps above with two additional fresh portions of 1:1 methylene chloride/acetone.

7.2.10 K-D Technique / Nitrogen Blowdown

7.2.10.1 Add one solvent rinsed boiling bead to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-85 ºC) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot vapor. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

7.2.10.2 When the apparent volume of liquid reaches 5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool. Remove the Snyder column.

7.2.10.3 Put the concentrator tube in a warm bath (35 °C) and evaporate the solvent volume to less than 1 mL using a gentle stream of clean, dry nitrogen. During evaporation, the tube solvent level must be positioned to avoid water condensation (i.e. the solvent level should be below the level of the water bath). Do not allow the extract to go dry as this may result in a loss of analytes. Using a disposable graduated pipet, adjust the final volume to 1.0 mL with methylene chloride and mix the extract completely.

7.2.10.4 Transfer to a 2 mL autosampler vial. Store in freezer until analysis.

7.3 Calibration

7.3.1 Initial Calibration

7.3.1.1 The GC/MS must be tuned to meet the criteria in Table 1 for a 50 ng injection of DFTPP. The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and pentachorophenol should be present at their normal responses, and no peak tailing should be visible.

7.3.1.2 Use the base peak ion from the specific internal standard as the primary ion for quantitation, unless interferences are noted.

7.3.1.3 Prepare working standards at a minimum of 5 concentration levels, ranging from 5.0-150 µg/mL (except benzo(k)fluoranthene and benzo(g,h,i)perylene range from 5.0-100 µg/mL, and pentachlorophenol ranges from 10-150 µg/mL), by diluting the 200 µg/mL Calibration Intermediate Solution with methylene chloride. A typical calibration curve would be:

Calib. Inter. Solution (µL/1 mL)	Concentration (µg/mL)
25	5.0
50	10
100	20
250	50
500	100
750	150

7.3.1.4 Add 10 µL of the 4,000 µg/mL Internal Standard Stock to each calibration standard prior to analysis to produce an ISTD concentration of 40 µg/mL.

7.3.1.5 Calculate response factors (RFs) for each compound at each level relative to the preceding internal standard (see Table #2).

7.3.1.6 The average response factor should be calculated for each compound. The percent relative standard deviation (%RSD) should be less than 15% for each compound. However, the %RSD for each Calibration Check Compound (CCC), (see Table 3), must be less than 30%. If these criteria cannot be met, corrective action must be taken and the system recalibrated. Possible problems include standard mixture degradation, injection port inlet contamination, contamination of the front end of the column, or active sites in the column or chromatographic system.

7.3.1.7 System Performance Check Compounds (SPCC) must meet minimum average response factor criteria (see Table 4) or corrective action must be taken. For examples of corrective action, see above.

7.3.1.8 If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation. A minimum of five calibration points may be used to define the working range.

7.3.1.9 If the %RSD of any compound is greater than 15%, construct calibration curves of area ratio (A/A_{is}) versus concentration using first order regression fit. Second order (quadratic) curves may be constructed for some compounds that respond poorly in the chromatographic system (e.g. benzyl alcohol, benzoic acid, benzidine, phenol, 4-nitrophenol, 2,3,4,6-tetrachlorophenol, 2,4-dinitrophenol, 4,6-dinitro-2-methylphenol, 2,4,6-tribromophenol, pentachlorophenol, hexachlorocyclopentadiene, acenaphthylene, diethyl phthalate, naphthalene, 2-methylnaphthalene, 2-chloronaphthalene, fluorene, benzo[b] & [k]fluoranthene, indeno[1,2,3-cd]pyrene, dibenz[a,h]anthracene, and benzo[g,h,i]perylene). Second order fit may not be used in place of instrument maintenance. A correlation coefficient of 0.99 or better is required for each curve fit.

7.3.1.10 Immediately after an initial calibration curve is generated it must be verified by a second source verification standard. Acceptance criteria will be set at 70.0 – 130%.

7.3.1.11 Calibration curve calculations are found in the QA Manual.

7.3.1.12 Reporting limit verification (RLV) is checked with each calibration curve or monthly at a minimum, by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% (± 50% for AZ samples) or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.3.2 Daily GC/MS calibration

7.3.2.1 The GC/MS tuning standard containing 50 ng of DFTPP must meet the Table 1 criteria. This standard must be run and meet these criteria every 12 hours.

7.3.2.2 A mid-level calibration standard must be analyzed every 12 hours. Two different concentrations of CCVs will be used to verify quadratic calibration curves when analyzing WI compliance samples. The SPCCs must meet the minimum response criteria on Table 4.

7.3.2.3 Use the Calibration Check Compounds (CCCs), found in Table 3, to check the validity of the initial calibration. Calculate the percent drift using:

$$\%Drift = \frac{(C_I - C_c)}{(C_I)}(100)$$

C_I = Calibration Check Compound standard concentration
C_c = Measured concentration using selected quantitation method

If the percent difference for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. Non-CCC must meet the acceptance criteria of $\leq$ 20% for Arizona compliance samples.

7.3.2.4 Evaluate the internal standard responses and retention times. If the retention time changes by more than 30 seconds from the mid-point of the last initial calibration curve or the Extracted Ion Current Profile (EICP) area for any internal standard changes by a factor of two (- 50% to + 100%) from the mid-point of the last initial calibration curve, the chromatographic system must be inspected for malfunctions and corrections made as required before samples can be analyzed.

7.3.2.5 If any of the daily calibration criteria are not met, minor corrective maintenance may be performed on the system and the calibration check standard re-run. If major corrective action were required, such as cleaning the source or replacing the chromatographic column, a new initial calibration would need to be generated before samples could be analyzed.

7.4 Analysis

7.4.1 GC/MS Conditions

7.4.1.1 Mass Range: 35-500 amu

7.4.1.2 Scan Time: approximately 1 scan/sec

7.4.1.3 Initial Temperature: 40 °C, hold for 4 minutes

7.4.1.4 Temp. Program: 40-320 °C at 10 °C/min

7.4.1.5 Final Temperature: 320 °C, hold until at least one minute after benzo(g,h,i)perylene has eluted

7.4.1.6 Injector Temperature: 250-300 °C

7.4.1.7 Interface Temperature: 250-300 °C

7.4.1.8 Injector: Split/Splitless

7.4.1.9 Sample volume: 1 µL

7.4.1.10 Carrier gas: Helium at 1 mL/min

7.4.2 Add 10 µL of the 4,000 µg/mL Internal Standard Stock to each sample extract to produce an ISTD concentration of 40 µg/mL.

7.4.3 Inject 1 µL of the 1 mL sample extract. If the response for any quantitation ion exceeds the initial calibration curve, make an appropriate dilution, add additional internal standard as required to maintain 40 µg/mL of each internal standard. Reanalyze the diluted extract.

7.4.4 Recap samples prior to storing in freezer.

7.5 Calculation

7.5.1 Qualitative analysis

7.5.1.1 The retention time of the sample compound must fall within ± 30 seconds of the retention time of the standard compound run within the last 12 hours.

7.5.1.2 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. Use the continuing calibration standard to obtain standard reference spectra.

7.5.1.3 The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. The relative intensities of the ions should agree within ± 30% between the sample and reference spectrum.

7.5.1.4 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification.

7.5.2 Quantitative analysis

7.5.2.1 Quantitate using the internal standard technique. Use the internal standard preceding the analyte (see Table 2). Quantitation is based on the integrated abundance from the EICP of the primary characteristic ion.

7.5.2.2 If the %RSD of a compound's relative response factor is 15% or less, then the concentration in the extract may be determined using the average response factor (RF) from initial calibration data and the following equation:

$$Concentration\ (mg/kg) = \frac{(A_x)(C_{is})(F)(V_{ex})}{(A_{is})(RF)(W_s)(D)}$$

A_x = Area of characteristic ion being measured
C_{is} = Amount of internal standard injected (µg/mL)
F = Dilution factor
V_{ex} = Volume of extract, mL
A_{is} = Area of characteristic ion for the internal standard
RF = Mean response factor for compound being measured
W_s = Weight of sample extracted, g
D = % dry weight of sample/100, or 1 for wet weight basis

7.5.2.3 Alternatively, the regression line fitted to the initial calibration may be used for the determination of the analyte concentration. Compute the concentration of the analyte in the sample using the following equation:

$$Concentration\ (mg/kg) = \frac{(C_{ex})(V_{ex})}{(W_s)(D)}$$

C_{ex} = extract concentration, µg/mL
V_{ex} = extract volume, mL
W_s = sample weight, g
D = % dry weight of sample/100, or 1 for wet weight basis

7.5.2.4 Where applicable, an estimate of concentration for noncalibrated components in the sample can be made. The concentration should be reported as an estimate assuming a response factor of 1 using the nearest internal standard.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, when there is a change in the test method that may affect how the test is performed or when there is a major change in instrumentation. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the reporting limit or the sample batch is re-extracted if possible. If it is not possible to re-extract, the data will be flagged where appropriate. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Accuracy control limits are generated for LCS, MS and surrogates. In-house control charts are generated semi-annually, using 20 Percent Recovery points, as follows:

9.3.1.1 Upper and Lower Control Limit = Mean ± 3s

9.3.1.2 Upper and Lower Warning Limit = Mean ± 2s

9.3.1.3 s = Standard deviation

9.3.1.4 For Arizona compliance, the lower control limit can not calculate below the lowest standard on the calibration curve (e.g. lowest standard = 5.0 µg/mL, spike is at 100 µg/mL, % can not be below 5.0%). The Minnesota Pollution Control Agency sets a guideline that the lower control limit can not be < 30.0%. LEGEND will use the greater of these two; 30.0% in this example.

9.3.2 Precision control limits are set at 20.0% RPD for LCS/LCSD and generated for MS/MSD. In-house control charts are generated semi-annually, using 20 RPD points, as follows:

9.3.2.1 Control Limit = Mean + 3s

9.3.2.2 Warning Limit = Mean + 2s

9.3.2.3 s = Standard deviation

9.3.2.4 RPD = Relative Percent Difference

9.3.3 QC calculations are found in the QA Manual.

9.3.4 LCS, MS and surrogates are reviewed.

9.3.5 If the LCS data are outside the limits, a method nonconformance is filled out and the sample batch is re-extracted and/or re-analyzed if possible. If the batch cannot be re-extracted and/or re-analyzed, the data are flagged and/or a case narrative is written for all client reports within the batch.

9.3.6 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS is flagged and/or a case narrative is written.

9.3.7 If a sample surrogate is outside the limits, the sample is re-extracted and/or reanalyzed if possible. If the sample cannot be re-extracted and/or reanalyzed, the data is flagged next to the actual result in the report.

10. REPORTING

10.1 Soil samples results are reported in mg/kg on a dry weight basis.

10.2 The reported result is rounded to two significant figures.

10.3 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 EPA 3500B, 3550B, 3545, 8270C, 8000B (MN), 8000C (AZ)

12.2 Vendor equipment manuals

TABLE 1 - DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	10-80% of mass 198
197	< 2% of mass 198
198	Base peak or >50% of 442
199	5-9% of mass 198
275	10-60% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	Base peak or > 50% of mass 198
443	15-24% of mass 442

TABLE 2 – Semi-Volatile Compounds

Compound	Primary Ion
1,4-Dichlorobenzene-d_4(IS)	152
n-Nitrosodimethylamine	74
2-Fluorophenol (surr.)	112
Aniline	93
bis(2-Chloroethyl)ether	93
Phenol-d_6 (surr.)	99
Phenol	94
2-Chlorophenol	128
1,3-Dichlorobenzene	146
1,4-Dichlorobenzene	146
1,2-Dichlorobenzene	146
Benzyl alcohol	108
bis(2-Chloroisopropyl)ether	45
2-Methylphenol	108
Hexachloroethane	117
N-Nitrosodi-n-propylamine	70
4-Methylphenol	108
Naphthalene-d_8 (IS)	136
Nitrobenzene-d_5 (surr.)	82

TABLE 2 – Semi-Volatile Compounds (continued)

Compound	Primary Ion
Nitrobenzene	77
Isophorone	82
2-Nitrophenol	139
2,4-Dimethylphenol	107
bis(2-Chloroethoxy)methane	93
2,4-Dichlorophenol	162
1,2,4-Trichlorobenzene	180
Naphthalene	128
Benzoic acid	122
2,6-Dichlorophenol	162
4-Chloroaniline	127
Hexachlorobutadiene	225
4-Chloro-3-methylphenol	107
2-Methylnaphthalene	142
Acenaphthene-d_{10} (IS)	164
Hexachlorocyclopentadiene	237
2,4,6-Trichlorophenol	196
2,4,5-Trichlorophenol	196
2-Fluorobiphenyl (surr.)	172
2-Chloronaphthalene	162
2-Nitroaniline	65
Acenaphthylene	152
Dimethylphthalate	163
2,6-Dinitrotoluene	165
Acenaphthene	153
3-Nitroaniline	138
2,4-Dinitrophenol	184
Dibenzofuran	168
2,4-Dinitrotoluene	165
4-Nitrophenol	109
2,3,4,6-Tetrachlorophenol	232
Fluorene	166
4-Chlorophenyl-phenylether	204
Diethylphthalate	149
4-Nitroaniline	138

TABLE 2 – Semi-Volatile Compounds (continued)

Compound	Primary Ion
Phenanthrene-d_{10} (IS)	188
4,6-Dinitro-2-methylphenol	198
N-Nitrosodiphenylamine	169
Azobenzene	77
2,4,6-Tribromophenol (surr.)	330
4-Bromophenyl-phenylether	248
Hexachlorobenzene	284
Pentachlorophenol	266
Phenanthrene	178
Anthracene	178
Carbazole	167
Di-n-butylphthalate	149
Fluoranthene	202
Chrysene-d_{12} (IS)	240
Benzidine	184
Pyrene	202
Terphenyl-d_{14} (surr.)	244
Butylbenzylphthalate	149
3,3'-Dichlorobenzidine	252
Benzo[a]anthracene	228
Chrysene	228
bis(2-Ethylhexyl)phthalate	149
Perylene-d_{12} (IS)	264
Di-n-octylphthalate	149
Benzo[b]fluoranthene	252
Benzo[k]fluoranthene	252
Benzo[a]pyrene	252
Indeno[1,2,3-cd]pyrene	276
Dibenz[a,h]anthracene	278
Benzo[g,h,i]perylene	276

TABLE 3 - Calibration Check Compounds

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 4 - System Performance Check Compounds

Compounds	Minimum Response Factor
N-Nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

Appendix A

Initial Demonstration of Capability (IDC)
Semi-volatile Organic Compounds (SVOC)

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards in Ottawa Sand and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: LCS limits
   Precision: LCS limits

   If the standards were not extracted, the results must meet the following criteria:

   Accuracy: 80.0-120%
   Precision: ≤ 20% RPD

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Semi-volatile Organic Compounds (SVOC)

Parameter	MDL (mg/kg)	RL (mg/kg)	Parameter	MDL (mg/kg)	RL (mg/kg)
1,2,4-Trichlorobenzene	0.031	0.33	Benzo(b)fluoranthene	0.043	0.33
1,2-Dichlorobenzene	0.033	0.33	Benzo(k)fluoranthene	0.043	0.33
1,3-Dichlorobenzene	0.031	0.33	Benzo(g,h,i)perylene	0.048	0.33
1,4-Dichlorobenzene	0.029	0.33	Benzoic Acid	0.024	0.33
2,3,4,6-Tetrachlorophenol	0.078	0.67	Benzyl Alcohol	0.095	0.67
2,4,5-Trichlorophenol	0.037	0.67	bis(2-Chloroethoxy)methane	0.034	0.33
2,4,6-Trichlorophenol	0.080	0.67	bis(2-Chloroethyl)ether	0.035	0.33
2,4-Dichlorophenol	0.075	0.67	bis(2-Chloroisopropyl)ether	0.034	0.33
2,4-Dimethylphenol	0.080	0.67	bis(2-Ethylhexyl)phthalate	0.048	0.33
2,4-Dinitrophenol	0.032	0.67	Butylbenzylphthalate	0.042	0.33
2,4-Dinitrotoluene	0.035	0.33	Carbazole	0.035	0.33
2,6-Dichlorophenol	0.069	0.67	Chrysene	0.044	0.33
2,6-Dinitrotoluene	0.039	0.33	Dibenz(a,h)anthracene	0.043	0.33
2-Chloronaphthalene	0.034	0.33	Dibenzofuran	0.033	0.33
2-Chlorophenol	0.060	0.67	Diethylphthalate	0.031	0.33
2-Methylnaphthalene	0.036	0.33	Dimethylphthalate	0.030	0.33
2-Methylphenol	0.034	0.67	Di-n-butylphthalate	0.044	0.33
2-Nitroaniline	0.038	0.33	Di-n-octylphthalate	0.048	0.33
2-Nitrophenol	0.085	0.67	Fluoranthene	0.039	0.33
3&4-Methylphenol	0.032	0.67	Fluorene	0.035	0.33
3,3'-Dichlorobenzidine	0.15	1.6	Hexachlorobenzene	0.036	0.33
3-Nitroaniline	0.027	0.33	Hexachlorobutadiene	0.030	0.33
4,6-Dinitro-2-methylphenol	0.034	0.67	Hexachlorocyclopentadiene	0.024	0.33
4-Bromophenyl-phenylether	0.033	0.33	Hexachloroethane	0.030	0.33
4-Chloro-3-methyl phenol	0.066	0.67	Indeno(1,2,3-cd)pyrene	0.040	0.33
4-Chloroaniline	0.027	0.67	Isophorone	0.031	0.33
4-Chlorophenyl-phenylether	0.036	0.33	Naphthalene	0.033	0.33
4-Nitroaniline	0.034	0.33	Nitrobenzene	0.033	0.33
4-Nitrophenol	0.093	0.67	n-Nitrosodimethylamine	0.022	0.33
Acenaphthene	0.038	0.33	n-Nitrosodi-n-propylamine	0.033	0.33
Acenaphthylene	0.041	0.33	n-Nitrosodiphenylamine	0.026	0.33
Aniline	0.031	0.67	Pentachlorophenol	0.078	0.67
Anthracene	0.036	0.33	Phenanthrene	0.036	0.33
Azobenzene (1,2-Diphenylhydrazine)	0.036	0.33	Phenol	0.069	0.67
Benzidine	0.19	2.5	Pyrene	0.041	0.33
Benzo(a)anthracene	0.041	0.33	Pyridine (for LTS AZ)	0.10	0.67
Benzo(a)pyrene	0.042	0.33			

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-021.10
REVIEWER:	Van Pham
DATE:	05/06/10

SECTION	CHANGE	RATIONALE
Appendix B	Updated MDLs	Annual update
Appendix B	Changed 4-methylphenol to 3&4 methylphenol	Compounds co-elute
Appendix B	Added pyridine	LTS AZ needs for some clients

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF SEMI-VOLATILE ORGANIC COMPOUNDS IN WATER BY GC/MS

SOP NO.: LABENV-022.11

Original Information		
Prepared by:	Sandy McDonald	Date: 03/13/96
Technical Review:		Date:
QA/QC Coordinator:	Sharon Cenis	Date: 03/25/96
Authorized by:	Cheryl Sykora	Date: 03/29/96

Revision Information		
Supersedes:	LABENV-022.10	Date: 04/27/09
Revised by:	Van Pham	Date: 05/10/10
Signature:	_____	Date: _____
Technical Review:	Triet Le	Date: 05/10/10
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 05/10/10
Signature:	_____	Date: _____

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Signature:		Date: 05/10/10
Authorized by:	Cheryl Sykora	Date:
Signature:	Cheryl Sykora	Date: 5/10/10

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SOP TITLE: DETERMINATION OF SEMI-VOLATILE ORGANIC COMPOUNDS IN WATER BY GC/MS

1. PURPOSE

1.1 This document defines the preparation and analysis for semi-volatile compounds in water by Gas Chromatography/Mass Spectrometry (GC/MS). This procedure can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused silica capillary column coated with a slightly polar silicone. The SOP is applicable to samples typically analyzed by EPA 8270C.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the SOP.

2.3 An analyst experienced in the use of gas chromatograph/mass spectrometers, skilled in the interpretation of mass spectra, and trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is applicable to surface water and groundwater.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

4.3 Follow standard laboratory safety procedures.

4.4 A lab coat and safety glasses should be worn during sample and standard preparation.

4.5 When working with organic compounds, wear chemical resistant gloves.

4.6 Prepare stock and standard solutions in a hood.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Samples should be collected in 1L amber bottles with Teflon lined caps and stored at 4 ± 2 °C.

5.4 The recommended holding time is 7 days until extraction and analysis within 40 days of extraction.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 HP 5890 Series II Gas Chromatograph (GC) with data processing equipment, or equivalent

6.2 HP 5972A Mass Selective Detector (MSD) with scan range of 35 to 500 amu using 70 volts electron energy in the electron impact ionization mode, or equivalent

6.3 Column – 30m x 0.25 mm ID (or 0.32 mm ID) x 0.25 µm film thickness silicone-coated fused silica capillary column (DB-5MS or equivalent)

6.4 Nitrogen evaporator – N-EVAP, or equivalent

6.5 Microliter syringes – 10, 25, 100, 250, 500, and 1000 µL

6.6 Volumetric flask – 5, 10, 25, and 50 mL

6.7 Serum Bottles – amber glass with Teflon-lined crimp tops

6.8 Two liter Teflon separatory funnel

6.9 Glass funnel with Pyrex glass wool at bottom

6.10 Kuderna-Danish (K-D) concentrator – 10 mL, graduated

6.11 Kuderna-Danish (K-D) flask – 500 mL

6.12 Snyder Column – three ball macro

6.13 PTFE solvent rinsed boiling beads, or equivalent

6.14 Water bath

6.15 Autosampler vials – 2 mL amber glass with Teflon lined crimp tops

6.16 pH paper (0-14 Std. Units)

6.17 Graduated cylinder – 1 liter

6.18 Graduated disposable pipets – 1 or 2 mL

6.19 Organic free water

6.20 Sodium Hydroxide (NaOH) – reagent grade

6.21 10N Sodium Hydroxide – dissolve 40 g of the NaOH in 100 mL organic free water

6.22 Sulfuric Acid (H_2SO_4) – reagent grade

6.23 1:1 Sulfuric Acid – slowly add 50 mL of H_2SO_4 (sp. gr. 1.84) to 50 mL of organic free water

6.24 Anhydrous Sodium Sulfate (Na_2SO_4) – muffle at 400 °C for four hours before using

6.25 Methylene Chloride (CH_2Cl_2) – pesticide grade, or equivalent

6.26 Acetone – pesticide grade, or equivalent

6.27 Methanol – pesticide grade, or equivalent

6.28 GC/MS Tune Check Stock – 500 µg/mL each of DFTPP, Benzidine, 4.4'-DDT, and pentachlorophenol, Absolute Standards, Inc. #43030, or equivalent

6.29 Calibration Stock 1 – 2000 µg/mL each of bis(2-chloroethoxy) methane, bis(2-chloroethyl) ether, bis(2-ethylhexyl) phthalate, bis(2-chloroisopropyl)ether, 4-bromophenyl phenyl ether, butyl benzyl phthalate, 4-chlorophenyl phenyl ether, diethyl phthalate, dimethyl phthalate, di-n-butyl phthalate, di-n-octyl phthalate, n-nitrosodimethylamine, n-nitrosodi-n-propylamine, and n-nitrosodiphenylamine, Absolute #10001, or equivalent

6.30 Calibration Stock 2 – 2000 µg/mL each of acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, carbazole, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene Absolute #10007, or equivalent

6.31 Calibration Stock 3 – 2000 µg/mL each of azobenzene (1,2-diphenylhydrazine), 2-chloronaphthalene, 1,2-dichlorobenzene, 1,4-dichlorobenzene, 1,3 dichlorobenzene, 2,6-dinitrotoluene, 2,4-dinitrotoluene, hexachlorobenzene, hexachlorobutadiene, hexachlorocyclopentadiene, hexachloroethane, isophorone, nitrobenzene, and 1,2,4-trichlorobenzene, Absolute #10002, or equivalent

6.32 Calibration Stock 4 – 2,000 µg/mL each of aniline, benzyl alcohol, 4-chloroaniline, dibenzofuran, 2-methylnaphthalene, 2-nitroaniline, 3-nitroaniline, and 4-nitroaniline, Absolute #10005, or equivalent

6.33 Calibration Stock 5 – 2,000 µg/mL each of benzoic acid, 2-methylphenol, 4-methylphenol, and 2,4,5-trichlorophenol, Absolute #10004, or equivalent

6.34 Calibration Stock 6 – 2,000 µg/mL each of 4-chloro-3-methylphenol, 2-chlorophenol, 2,4-dichlorophenol, 2,6-dichlorophenol, 2,4-dimethylphenol, 2,4-dinitrophenol, 2-methyl-4,6-dinitrophenol, 2-nitrophenol, 4-nitrophenol, pentachlorophenol, phenol, 2,4,6-trichlorophenol, and 2,3,4,6-tetrachlorophenol, Absolute #10018, or equivalent

6.35 Calibration Stock 7 – 2,000 µg/mL each of benzidine and 3,3'-dichlorobenzidine, Absolute #10006, or equivalent

6.36 Calibration Stock 8 – 1,000 µg/mL of pyridine, Absolute #70260, or equivalent

6.37 Internal Standard Stock – 4,000 µg/mL each of acenaphthene-d_{10}, chrysene-d_{12}, 1,4-dichlorobenzene-d_4, naphthalene-d_8, perylene-d_{12}, and phenanthrene-d_{10}, Absolute #10009, or equivalent

6.38 Calibration Surrogate Stock 1 – 10,000 µg/mL each of 2-flurophenol, phenol-d6, and 2,4,6-tribromophenol, Absolute #21015, or equivalent

6.39 Calibration Surrogate Stock 2 – 5,000 µg/mL each of nitrobenzene-d_5, 2-fluorobiphenyl, and terphenyl-$d_{14,}$ Absolute #21016, or equivalent

6.40 Sample/Second Source Surrogate Stock 1 – 10,000 µg/mL each of 2-flurophenol, phenol-d6, and 2,4,6-tribromophenol, Restek #31087, or equivalent

6.41 Sample/Second Source Surrogate Stock 2 – 5,000 µg/mL each of nitrobenzene-d_5, 2-fluorobiphenyl, and terphenyl-$d_{14,}$ Restek #31086, or equivalent

6.42 Spike Stock 1 – 10,000 µg/mL each of pentachlorophenol, phenol, 2-chlorophenol, 4-chloro-3-methylphenol, and 4-nitrophenol, Restek #31071, or equivalent

6.43 Spike Stock 2 – 5,000 µg/mL each of 1,2,4-trichlorobenzene, acenaphthene, 2,4-dinitrotoluene, pyrene, n-nitrosodi-n-propylamine, and 1,4-dichlorobenzene, Restek #31084, or equivalent

6.44 Spike Stock 3 – 2,000 µg/mL each of acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, 2-methylnaphthalene, naphthalene, phenanthrene, and pyrene, Absolute #50003, or equivalent

6.45 Second Source Stock 9 - 1000ug/ml of all 8270 components except for benzidine, 3,3'-dichlorobenzidine, 2,6-dichlorophenol, surrogates, and benzoic acid , Restek # 31850 or equivalent

6.46 Second Source Stock 10 - 2000ug/ml each of benzidine and 3,3'-dichlorobenzidine, Restek # 31834 or equivalent

6.47 Second Source Stock 11 - 1000ug/ml of 2,6-dichlorophenol, Restek # 31409 or equivalent

6.48 Second source stock 12 - 1000ug/ml of benzoic acid, Absolute #70034, or equivalent

6.49 Calibration Intermediate Solution – combine 300 µL of the 2,000 µg/mL Calibration Stocks 1-7, 600 µL of the 1,000 µg/mL Calibration Stock 8, 60 µL of the 10,000 µg/mL Surrogate Stock 1, 120 µL of the 5,000 µg/mL Surrogate Stock 2, and 120 µL of methylene chloride (3 mL final volume) to produce a 200 µg/mL Calibration Intermediate Solution

6.50 GC/MS Tune Check Standard – dilute 100 µL of the 500 µg/mL GC/MS Tune Check Stock with 900 µL of methylene chloride to produce a 50 µg/mL GC/MS Tune Check Standard

6.51 Sample Surrogate Standard – dilute 0.5 mL of the 10,000 µg/mL Surrogate Stock 1 and 1 mL of the 5,000 µg/mL Surrogate Stock 2 to a final volume of 50 mL with methanol to produce a 100 µg/mL Sample Surrogate Standard

6.52 Spike Standard – dilute 0.5 mL of the 10,000 µg/mL Spike Stock 1, 1 mL of the 5,000 µg/mL Spike Stock 2, and 2.5 mL of the 2,000 µg/mL Spike Stock 3 to a final volume of 50 mL with methanol to produce a 100 µg/mL Spike Standard

6.53 Second Source ICV - combine 50 µL of the 1,000 µg/mL Stocks 9, 11 and 12, 25 µL of the 2000 µg/mL Stock 10, 5.0 µL of the 10,000 µg/mL Sample Surrogate Stock 1, 10 µL of the 5,000 µg/mL Sample Surrogate Stock 2, and 810 µL of methylene chloride (1 mL final volume) to produce a 50 µg/mL Second Source Calibration Solution. Add 10 µL of the 4,000 µg/mL Internal Standard Stock prior to analysis to produce an ISTD concentration of 40 µg/mL.

6.54 All solutions and standards should be stored in a freezer at $\leq$ -10 ºC and should be freshly prepared each year, or sooner if check standards or continuing calibration standards indicate a problem.

7. PROCEDURE

7.1 Preparation of Water Samples

7.1.1 Pre-rinse all glassware twice with acetone, once with hexane, and three times with $MeCl_2$.

7.1.2 Mark the water level on the outside of the bottle for later determination of volume.

7.1.3 Measure the pH of the sample and transfer to a pre-rinsed two-liter separatory funnel. (Note: If an evident layer of sediment is present, decant the sample and record on the extraction sheet.)

7.1.4 If analysis of acid and base/neutral compounds is required, first extract the sample three times with the pH being <2 and then three times with the pH being >11.

7.1.5 If the analysis of acid compounds ONLY is required, adjust the pH to <2 with 1:1 H_2SO_4.

7.1.6 If the analysis of base/neutral compounds ONLY is required, adjust the pH to >11 with 10N NaOH.

7.1.7 Add 50 mL of acetone to all samples, spikes, and blanks.

7.1.8 Add 1.0 mL of the 100 µg/mL Sample Surrogate Standard to all samples, spikes, and blanks. Final concentration will be 100 µg/L assuming a 1.0 mL final volume, 1 µL injection, and 1,000 mL sample volume.

7.1.9 For the samples in each analytical batch selected for spiking, add 1.0 mL of the 100 µg/mL Spike Standard. Final concentration will be 100 µg/L assuming a 1.0 mL final volume, 1 µL injection, and 1,000 mL sample volume. A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

7.1.10 Add approximately 60 mL of methylene chloride to the sample bottle and rinse. Transfer to the separatory funnel with the sample. (Note: If the sample was decanted, add the 60 mL of methylene chloride directly to the separatory funnel.)

7.1.11 Cap and shake vigorously for 10 seconds then vent. Continuously shake for an additional 2 minutes. Allow the methylene chloride to separate from the sample.

7.1.12 If an emulsion interface between layers is more than one-third the size of the solvent layer, the analyst should perform a beaker break without the use of Na_2SO_4.

7.1.12.1 Using a beaker, transfer the solvent layer to a glass 100 mm funnel containing a glass wool plug and about 2-3 inches of anhydrous muffled Na_2SO_4. Rinse the beaker with $MeCl_2$ and add to the funnel.

7.1.13 Drain into a 500 mL K-D flask equipped with a 10 mL K-D concentrator for Nitrogen Blowdown.

7.1.14 Repeat steps above with two additional fresh portions of methylene chloride.

7.1.15 After the final extraction, rinse the Na_2SO_4 with 20-30 mL of methylene chloride to complete the quantitative transfer.

7.1.16 If the emulsion layer is still present after the final shake, the analyst should employ mechanical techniques to complete the phase separation. Refer to the protocol found in Work Instruction (WI) 'Handling Emulsions'

7.1.17 Fill sample bottle with tap water to mark made previously. Transfer to a 1000 mL graduated cylinder and record volume on extraction sheet.

7.1.18 K-D Technique / Nitrogen Blowdown

7.1.18.1 Add one solvent rinsed boiling bead to the flask, 10 mL of acetone, and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-85 °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot vapor. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches approximately 20 mL, add 10 mL of acetone through the Snyder column.

7.1.18.2 Concentrate to approximately 5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool. Remove the Snyder column.

7.1.18.3 Put the K-D concentrator in a warm bath (35 ºC) and evaporate the solvent volume to less than 1 mL using a gentle stream of clean, dry nitrogen. During evaporation, the tube solvent level must be positioned to avoid water condensation (i.e. the solvent level should be below the level of the water bath). Do not allow the extract to go dry at any point. This may result in a loss of analytes, especially the phenols.

7.1.18.4 Using a disposable graduated pipet, adjust the final volume to 1.0 mL with methylene chloride and mix the extract completely.

7.1.18.5 Transfer to a 2 mL autosampler vial. Store in freezer until analysis.

7.2 Calibration

7.2.1 Initial Calibration

7.2.1.1 The GC/MS must be tuned to meet the criteria in Table 1 for a 50 ng injection of DFTPP. The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and pentachorophenol should be present at their normal responses, and no peak tailing should be visible.

7.2.1.2 Use the base peak ion from the specific internal standard as the primary ion for quantitation, unless interferences are noted.

7.2.1.3 Prepare working standards at a minimum of 5 concentration levels, ranging from 5.0-150 µg/mL (except benzo(k)fluoranthene and benzo(g,h,i)perylene range from 5.0-100 µg/mL, and pentachlorophenol ranges from 10-150 µg/mL), by diluting the 200 µg/mL Calibration Intermediate Solution with methylene chloride. A typical calibration curve would be:

Calib. Inter. Solution (µL/1 mL)	Concentration (µg/mL)
25	5.0
50	10
100	20
250	50
400	80
500	100
750	150

7.2.1.4 Add 10 µL of the 4,000 µg/mL Internal Standard Stock to each calibration standard prior to analysis to produce an ISTD concentration of 40 µg/mL.

7.2.1.5 Calculate response factors (RFs) for each compound at each level relative to the preceding internal standard (see Table 2).

7.2.1.6 The average response factor should be calculated for each compound. The percent relative standard deviation (%RSD) should be less than 15% for each compound. However, the %RSD for each Calibration Check Compound (CCC), (see Table 3), must be less than 30%. If these criteria cannot be met, corrective action must be taken and the system recalibrated. Possible problems include standard mixture degradation, injection port inlet contamination, contamination of the front end of the column, or active sites in the column or chromatographic system.

7.2.1.7 System Performance Check Compounds (SPCC) must meet minimum average response factor criteria (see Table 4). For examples of corrective action, see above.

7.2.1.8 If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation. A minimum of five calibration points may be used to define the working range.

7.2.1.9 If the %RSD of any compound is greater than 15%, construct calibration curves of area ratio (A/A_{is}) versus concentration using first order regression fit. Second order (quadratic) curves may be constructed for some compounds that respond poorly in the chromatographic system (e.g. benzyl alcohol, benzoic acid, benzidine, phenol, 4-nitrophenol, 2,3,4,6-tetrachlorophenol, 2,4-dinitrophenol, 4,6-dinitro-2-methylphenol, 2,4,6-tribromophenol, pentachlorophenol, hexachlorocyclopentadiene, acenaphthylene, diethyl phthalate, naphthalene, 2-methylnaphthalene, 2-chloronaphthalene, fluorene, benzo[b] & [k]fluoranthene, indeno[1,2,3-cd]pyrene, dibenz[a,h]anthracene, benzo[g,h,i]perylene, fluorene, and benzo[b] & [k]fluoranthene). Second order fit may not be used in place of instrument maintenance. A correlation coefficient of 0.99 or better is required for each curve fit.

7.2.1.10 Immediately after an initial calibration curve is generated it must be verified by a second source verification standard. Acceptance criteria will be set at 70.0 – 130%.

7.2.1.11 Calibration curve calculations are found in the QA Manual.

7.2.1.12 Reporting limit verification (RLV) is checked with each calibration curve or monthly at a minimum, by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% (± 50% for AZ samples) or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.2.2 Daily GC/MS calibration

7.2.2.1 The GC/MS tuning standard containing 50 ng of DFTPP must meet the Table 1 criteria. This standard must be run and meet these criteria every 12 hours.

7.2.2.2 A mid-level calibration standard must be analyzed every 12 hours. The SPCCs must meet the minimum response criteria on Table 4.

7.2.2.3 Use the Calibration Check Compounds (CCCs), found in Table 3, to check the validity of the initial calibration. Calculate the percent drift using:

$$\%Drift = \frac{(C_I - C_c)}{(C_I)}(100)$$

C_I = Calibration Check Compound standard concentration
C_c = Measured concentration using selected quantitation method

If the percent difference for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. Non-CCC must meet the acceptance criteria of $\leq$ 20% for Arizona compliance samples.

7.2.2.4 Evaluate the internal standard responses and retention times. If the retention time changes by more than 30 seconds from the mid-point of the last initial calibration curve or the Extracted Ion Current Profile (EICP) area for any internal standard changes by a factor of two (- 50% to + 100%) from the mid-point of the last initial calibration curve, the chromatographic system must be inspected for malfunctions and corrections made as required before samples can be analyzed.

7.2.2.5 If any of the daily calibration criteria are not met, minor corrective maintenance may be performed on the system and the calibration check standard re-run. If major corrective action was required, such as cleaning the source or replacing the chromatographic column, a new initial calibration would need to be generated before samples could be analyzed.

7.3 Analysis

7.3.1 GC/MS Conditions

7.3.1.1 Mass Range: 35-500 amu

7.3.1.2 Scan Time: 1 scan/sec

7.3.1.3 Initial Temperature: 40 °C, hold for 4 minutes

7.3.1.4 Temp. Program: 40- 320 °C at 10 °C/min

7.3.1.5 Final Temperature: 320 °C, hold until at least 1 minute after benzo(g,h,i)perylene has eluted

7.3.1.6 Injector Temperature: 250-300 °C

7.3.1.7 Interface Temperature: 250-300 °C

7.3.1.8 Injector: Split/Splitless

7.3.1.9 Sample volume: 1 µL

7.3.1.10 Carrier gas: Helium at 1 mL/min

7.3.2 Add 10 µL of the 4,000 µg/mL Internal Standard Stock to each sample extract to produce an ISTD concentration of 40 µg/mL.

7.3.3 Inject 1 µL of the 1 mL sample extract. If the response for any quantitation ion exceeds the initial calibration curve, make an appropriate dilution, add additional internal standard as required to maintain 40 µg/mL of each internal standard. Reanalyze the diluted extract.

7.3.4 Recap sample vials prior to storing in freezer.

7.4 Calculation

7.4.1 Qualitative analysis

7.4.1.1 The retention time of the sample compound must fall within ± 30 seconds of the retention time of the standard compound run within the last 12 hours.

7.4.1.2 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. Use a mid-level initial calibration standard to obtain standard reference spectra.

7.4.1.3 The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. The relative intensities of the ions should agree within ± 30% between the sample and reference spectrum.

7.4.1.4 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification.

7.4.2 Quantitative analysis

7.4.2.1 Quantitate using the internal standard technique. Use the internal standard preceding the analyte (see Table 2). Quantitation is based on the integrated abundance from the EICP of the primary characteristic ion.

7.4.2.2 If the %RSD of a compound's relative response factor is 15% or less, then the concentration in the extract may be determined using the average response factor (RF) from initial calibration data and the following equation:

$$Concentration\ (\mu g / L) = \frac{(A_x)(C_{is})(F)(V_{ex})}{(A_{is})(RF)(V_s)}$$

A_x = Area of characteristic ion being measured
C_{is} = Amount of internal standard injected (µg/mL)
F = Dilution factor
V_{ex} = Volume of extract, mL
A_{is} = Area of characteristic ion for the internal standard
RF = Mean response factor for compound being measured
V_s = Volume of sample, L

7.4.2.3 Alternatively, the regression line fitted to the initial calibration may be used for the determination of the analyte concentration.

7.4.2.4 Where applicable, an estimate of concentration for noncalibrated components (Tentatively Identified Compounds – TIC) in the sample can be made. The concentration should be reported as an estimate assuming a response factor of 1 using the nearest internal standard.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, when there is a change in the test method that may affect how the test is performed or when there is a major change in instrumentation. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the reporting limit or the sample batch is re-extracted if possible. If it is not possible to re-extract, the data will be flagged where appropriate. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Accuracy control limits are generated for LCS, MS and surrogates. In-house control charts are generated semi-annually, using 20 Percent Recovery points, as follows:

9.3.1.1 Upper and Lower Control Limit = Mean ± 3s

9.3.1.2 Upper and Lower Warning Limit = Mean ± 2s

9.3.1.3 s = Standard deviation

9.3.1.4 For Arizona compliance, the lower control limit cannot calculate below the lowest standard on the calibration curve (e.g. lowest standard = 5.0 µg/mL, spike is at 100 µg/mL, % can not be below 5.0%). The Minnesota Pollution Control Agency sets a guideline that the lower control limit can not be < 30.0%. LEGEND will use the greater of these two; 30.0% in this example.

9.3.2 Precision control limits are set at 20.0% RPD for LCS/LCSD and generated for MS/MSD. In-house control charts are generated semi-annually, using 20 RPD points, as follows:

9.3.2.1 Control Limit = Mean + 3s

9.3.2.2 Warning Limit = Mean + 2s

9.3.2.3 s = Standard deviation

9.3.2.4 RPD = Relative Percent Difference

9.3.3 QC calculations are found in the QA Manual.

9.3.4 LCS, MS and surrogates are reviewed.

9.3.5 If the LCS data are outside the limits, a method nonconformance is filled out and the sample batch is re-extracted and/or re-analyzed if possible. If the batch cannot be re-extracted and/or re-analyzed, the data are flagged and/or a case narrative is written for all client reports within the batch.

9.3.6 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS is flagged and/or a case narrative is written.

9.3.7 If a sample surrogate is outside the limits, the sample is re-extracted and/or reanalyzed if possible. If the sample cannot be re-extracted and/or reanalyzed, the data is flagged next to the actual result in the report.

10. REPORTING

10.1 Sample results are reported in µg/L.

10.2 The reported result is rounded to two significant figures.

10.3 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Report Limits

12. REFERENCES

12.1 EPA Methods 3500B, 3510C, 8270C, 8000B (MN), 8000C (AZ)

12.2 Vendor equipment manuals

TABLE 1 – DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	10-80% of mass 198
197	< 2% of mass 198
198	Base peak or >50% of 442
199	5-9% of mass 198
275	10-60% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	Base peak or > 50% of mass 198
443	15-24% of mass 442

TABLE 2 – Semi-Volatile Compounds

Parameter	Primary Ion
1,4-Dichlorobenzene-d_4(IS)	152
n-Nitrosodimethylamine	74
2-Fluorophenol (surr.)	112
Aniline	93
bis(2-Chloroethyl)ether	93
Phenol-d_6 (surr.)	99
Phenol	94
2-Chlorophenol	128
1,3-Dichlorobenzene	146
1,4-Dichlorobenzene	146
1,2-Dichlorobenzene	146
Benzyl alcohol	108
bis(2-Chloroisopropyl)ether	45
2-Methylphenol	108
Hexachloroethane	117
N-Nitrosodi-n-propylamine	70
4-Methylphenol	108
Naphthalene-d_8 (IS)	136
Nitrobenzene-d_5 (surr.)	82

TABLE 2 – Semi-Volatile Compounds (continued)

Parameter	Primary Ion
Nitrobenzene	77
Isophorone	82
2-Nitrophenol	139
2,4-Dimethylphenol	107
Bis(2-Chloroethoxy)methane	93
2,4-Dichlorophenol	162
1,2,4-Trichlorobenzene	180
Naphthalene	128
Benzoic acid	122
2,6-Dichlorophenol	162
4-Chloroaniline	127
Hexachlorobutadiene	225
4-Chloro-3-methylphenol	107
2-Methylnaphthalene	142
Acenaphthene-d_{10} (IS)	164
Hexachlorocyclopentadiene	237
2,4,6-Trichlorophenol	196
2,4,5-Trichlorophenol	196
2-Fluorobiphenyl (surr.)	172
2-Chloronaphthalene	162
2-Nitroaniline	65
Acenaphthylene	152
Dimethylphthalate	163
2,6-Dinitrotoluene	165
Acenaphthene	153
3-Nitroaniline	138
2,4-Dinitrophenol	184
Dibenzofuran	168
2,4-Dinitrotoluene	165
4-Nitrophenol	109
2,3,4,6-Tetrachlorophenol	232
Fluorene	166
4-Chlorophenyl-phenylether	204
Diethylphthalate	149
4-Nitroaniline	138

TABLE 2 – Semi-Volatile Compounds (continued)

Parameter	Primary Ion
Phenanthrene-d_{10} (IS)	188
4,6-Dinitro-2-methylphenol	198
N-Nitrosodiphenylamine	169
Azobenzene	77
2,4,6-Tribromophenol (surr.)	330
4-Bromophenyl-phenylether	248
Hexachlorobenzene	284
Pentachlorophenol	266
Phenanthrene	178
Anthracene	178
Carbazole	167
Di-n-butylphthalate	149
Fluoranthene	202
Chrysene-d_{12} (IS)	240
Benzidine	184
Pyrene	202
Terphenyl-d_{14} (surr.)	244
Butylbenzylphthalate	149
3,3'-Dichlorobenzidine	252
Benzo(a)anthracene	228
Chrysene	228
bis(2-Ethylhexyl)phthalate	149
Perylene-d_{12} (IS)	264
Di-n-octylphthalate	149
Benzo(b)fluoranthene	252
Benzo(k)fluoranthene	252
Benzo(a)pyrene	252
Indeno(1,2,3-cd)pyrene	276
Dibenz(a,h)anthracene	278
Benzo(g,h,i)perylene	276

TABLE 3 – Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 4 – System Performance Check Compounds (SPCC)

Compounds	Minimum Response Factor
N-Nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

Appendix A

Initial Demonstration of Capability (IDC)
Semi-volatile Organic Compounds (SVOC)

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards in lab-grade water and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: LCS limits
   Precision: LCS limits

   If the standards were not extracted, the results must meet the following criteria:

   Accuracy: 80.0-120%
   Precision: ≤ 20% RPD

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Semi-Volatile Organic Compounds (SVOC)

Parameter	MDL (µg/L)	RL (µg/L)	Parameter	MDL (µg/L)	RL (µg/L)
1,2,4-Trichlorobenzene	0.30	10	Benzo(b)fluoranthene	0.44	10
1,2-Dichlorobenzene	0.33	10	Benzo(k)fluoranthene	0.46	10
1,3-Dichlorobenzene	0.31	10	Benzo(g,h,i)perylene	0.49	10
1,4-Dichlorobenzene	0.29	10	Benzoic Acid	0.52	10
2,3,4,6-Tetrachlorophenol	0.96	10	Benzyl Alcohol	1.2	10
2,4,5-Trichlorophenol	0.59	10	bis(2-Chloroethoxy)methane	0.27	10
2,4,6-Trichlorophenol	0.59	10	bis(2-Chloroethyl)ether	0.22	10
2,4-Dichlorophenol	0.63	10	bis(2-Chloroisopropyl)ether	0.23	10
2,4-Dimethylphenol	0.57	10	bis(2-Ethylhexyl)phthalate	0.81	10
2,4-Dinitrophenol	1.2	10	Butylbenzylphthalate	0.45	10
2,4-Dinitrotoluene	0.45	10	Carbazole	0.33	10
2,6-Dichlorophenol	0.50	10	Chrysene	0.61	10
2,6-Dinitrotoluene	0.53	10	Dibenz(a,h)anthracene	0.55	10
2-Chloronaphthalene	0.60	10	Dibenzofuran	0.59	10
2-Chlorophenol	0.39	10	Diethylphthalate	0.30	10
2-Methylnaphthalene	0.88	10	Dimethylphthalate	0.33	10
2-Methylphenol	0.48	10	Di-n-butylphthalate	0.39	10
2-Nitroaniline	0.60	10	Di-n-octylphthalate	0.67	10
2-Nitrophenol	0.93	10	Fluoranthene	0.61	10
3&4-Methylphenol	0.41	10	Fluorene	0.52	10
3,3'-Dichlorobenzidine	7.2	25	Hexachlorobenzene	0.40	10
3-Nitroaniline	1.1	10	Hexachlorobutadiene	0.41	10
4,6-Dinitro-2-methylphenol	1.0	10	Hexachlorocyclopentadiene	0.35	10
4-Bromophenyl-phenylether	0.42	10	Hexachloroethane	0.33	10
4-Chloro-3-methyl phenol	0.48	10	Indeno(1,2,3-cd)pyrene	0.39	10
4-Chloroaniline	1.3	10	Isophorone	0.26	10
4-Chlorophenyl-phenylether	0.31	10	Naphthalene	0.52	10
4-Nitroaniline	0.90	10	Nitrobenzene	0.37	10
4-Nitrophenol	0.78	10	n-Nitrosodimethylamine	0.48	10
Acenaphthene	0.60	10	n-Nitrosodi-n-propylamine	0.25	10
Acenaphthylene	0.55	10	n-Nitrosodiphenylamine	0.38	10
Aniline	1.6	10	Pentachlorophenol	0.90	10
Anthracene	0.56	10	Phenanthrene	0.35	10
Azobenzene (1,2-Diphenylhydrazine)	0.26	10	Phenol	0.41	10
Benzidine	7.6	100	Pyrene	0.66	10
Benzo(a)anthracene	0.50	10	Pyridine (for LTS AZ)	2.1	10
Benzo(a)pyrene	0.41	10	---	---	---

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-022.11
REVIEWER:	Van Pham
DATE:	05/10/10

SECTION	CHANGE	RATIONALE
7.1.1	Changed from once to twice	To remove water residue better
7.1.15	Changed from sodium sulfate to Na_2SO_4	To keep format consistent throughout the SOP
Appendix B	Updated MDLs	Annual update
Appendix B	Changed 4-methylphenol to 3&4-methylphenol	Compounds co-elute

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**DETERMINATION OF ORGANOCHLORINATED PESTICIDES IN SOIL AND WATER SAMPLES**
SOP NO.:	**LABENV-025.9**

Original Information		
Prepared by:	Jennifer Nelson	Date: 11/03/95
Technical Review:		Date:
QA/QC Coordinator:	Sharon Cenis	Date: 03/28/96
Authorized by:	Cheryl Sykora	Date: 03/29/96

Revision Information		
Supersedes:	LABENV-025.8	Date: 08/28/09
Revised by:	Henrik Pham	Date: 05/03/11
Signature:	_____	Date: _____
Technical Review:	Van Pham	Date: 05/03/11
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 05/03/11
Signature:	_____	Date: _____

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Revised by:	Henrik Pham	Date:
Signature:	Henrik Pham	Date: 5/3/11
Technical Review:	Van Pham	Date:
Signature:	Van Pham	Date: 5/3/11
Authorized by:	Cheryl Sykora	Date:
Signature:	Cheryl Sykora	Date: 5/3/11

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SOP TITLE: DETERMINATION OF ORGANOCHLORINATED PESTICIDES IN SOIL AND WATER SAMPLES

1. PURPOSE

1.1 This document defines the procedure to be followed for the preparation and analysis for organochlorine pesticides in soil and water by gas chromatography (GC) using an electron capture detector (ECD). The SOP is applicable to samples typically analyzed by EPA Method 8081A.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 An analyst experienced in GC techniques and trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is applicable to solid, wastewater, groundwater, and aqueous samples.

3.2 The Sonication Method may be used when a sample has the potential to be detrimental to the ASE (tar samples, fine sediments, etc.).

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 A lab coat and safety glasses should be worn.

4.4 When working with organic compounds, wear solvent resistant gloves.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in 1L amber bottles with Teflon lined caps and stored at ≤ 6 °C but not freezing.

5.4 The recommended holding time for water samples is 7 days until extraction and analysis within 40 days of extraction.

5.5 Soil samples should be collected in unweighed 4 oz. glass jars with Teflon lined caps and stored at ≤ 6 °C but not freezing.

5.6 The recommended holding time for soil samples is 14 days until extraction and analysis within 40 days of extraction.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Gas chromatograph equipped with dual injectors, dual ECDs, and a data processing system

6.2 GC columns – Rtx®-CLPesticides™, 30 m x 0.32 mm, 0.5 µm film (Restek #11139), and Rtx®-CLPesticidesII™, 30 m x 0.32 mm, 0.25 µm film (Restek #11324) or equivalent. Whichever two columns are selected, they must be of dissimilar stationary phases.

6.3 Two liter Teflon separatory funnel, or equivalent

6.4 500 mL Kuderna Danish (K-D) flask

6.5 Steam bath

6.6 100 mm glass funnel

6.7 10 mL K-D concentrator

6.8 Snyder column

6.9 pH paper (0-14 Std. Units)

6.10 Graduated cylinders, 1000 mL, 100 mL

6.11 Volumetric flasks, 50 mL, 25 mL, 10 mL

6.12 Microliter syringes

6.13 Disposable glass pasteur pipets and bulb

6.14 Turbo Vap II and associated parts and glassware

6.15 Accelerated Solvent Extractor (ASE) and associated parts and glassware

6.16 Ultrasonic Disrupter - Bronson Sonifier 450, or equivalent

6.17 Balance, capable of reading to 0.01 g

6.18 Filter paper- Dionex Corporation 19.8 mm cellulose or equivalent

6.19 Assorted laboratory glassware

6.20 Beakers, 250 mL

6.21 Glass wool

6.22 2 mL autosampler vials

6.23 PTFE solvent rinsed boiling beads, or equivalent

6.24 Disposable weighing aluminum dishes – prerinsed with hexane and methylene chloride

6.25 Disposable graduated pipets

6.26 4 mL saver vials with Teflon liners

6.27 Anhydrous Sodium Sulfate (Na_2SO_4) - muffle at 400 °C for four hours before using

6.28 Sodium Hydroxide (NaOH) – reagent grade

6.29 10N Sodium Hydroxide – dissolve 40 g of the reagent grade NaOH in 100 mL organic free water

6.30 Sulfuric Acid (H_2SO_4) – reagent grade

6.31 1:1 Sulfuric Acid – slowly add 50 mL of the reagent grade H_2SO_4 to 50 mL organic free water

6.32 Hydromatrix® or equivalent – muffle at 400 °C for four hours before using

6.33 Ottawa Sand (20-30 mesh) or equivalent – muffle at 400 °C for four hours before using

6.34 Organic free water

6.35 Methylene chloride (CH_2Cl_2) - pesticide grade, or equivalent

6.36 Acetone – pesticide grade, or equivalent

6.37 Hexane – pesticide grade, or equivalent

6.38 Hexane/Acetone (3:1) (v/v) – for ASE Method

6.39 Hexane/Acetone (1:1) (v/v) – for Sonication Method

6.40 Pesticide Stock 1 – 2000 µg/mL, Supelco #47426-U or equivalent

6.41 Pesticide Stock 2 – 2000 µg/mL, Restek #32415 or equivalent, must be a different vendor or lot number than Pesticide Stock 1 (used in Pesticide Second Source Standard Solution)

6.42 Surrogate Stock – Restek #32000, 200 µg/mL for each of the following compounds: 2,4,5,6-Tetrachloro-m-xylene (TCMX), and Decachlorobiphenyl (DCB)

6.43 Toxaphene Stock – 1000 µg/mL, Supelco #4-8103 or equivalent

6.44 4,4-DDT and Endrin Breakdown Stock – 100 µg/mL each, Restek #32093 or equivalent

6.45 Pesticide Intermediate Solution – dilute 50 µL of 2000 µg/mL Pesticide Stock 1 and 500 µL of the 200 µg/mL Surrogate Stock into a 10 mL volumetric flask with hexane to produce a 10 µg/mL Pesticide and Surrogate Standard. Store in a freezer for up to six months.

6.46 Pesticide Spike Intermediate Solution – dilute 125 µL of the 2,000 µg/mL Pesticide Stock 1 into a 10 mL volumetric flask with 1:1 hexane:acetone to produce a 25 µg/mL Pesticide Spike Intermediate Solution. Store in a freezer for up to six months.

6.47 Pesticide Spike Standard – dilute 1.25 mL of the 25 µg/mL Pesticide Spike Intermediate Solution into a 25 mL volumetric flask with 1:1 hexane:acetone to produce a 1.25 µg/mL Pesticide Spike Standard. Store in a freezer for up to six months.

6.48 Surrogate Standard – dilute 250 µL of the 200 µg/mL Surrogate Stock into a 50 mL volumetric flask with 1:1 hexane:acetone to produce a 1.0 µg/mL Working Surrogate Standard. Store in a freezer for up to six months.

6.49 Toxaphene Intermediate Solution – dilute 10 µL of the 1,000 µg/mL Toxaphene Stock into a 10 mL volumetric flask with hexane to produce a 1.0 µg/mL Toxaphene Intermediate Solution. Store in a freezer for up to six months.

6.50 4,4'-DDT and Endrin Breakdown Standard – dilute 1-2 drops of the 100 µg/mL 4,4-DDT and Endrin Breakdown Stock with approximately 50 mL of hexane to produce the 4,4'-DDT and Endrin Breakdown Standard. Store in a freezer for up to six months.

NOTE: The actual concentration of the breakdown standard is not needed; the calculation uses responses only.

6.51 Pesticide Second Source Standard (CCAL/CCVS) – dilute 5 µL of the 2,000 µg/mL Pesticide Stock 2 and 50 µL of the 200 µg/mL Surrogate Stock to 50 mL with hexane to produce a 0.20 µg/mL Pesticide and Surrogate Second Source Standard. Store in a freezer for up to six months.

7. PROCEDURE

7.1 Preparation of Water Samples

7.1.1 Pre-rinse all glassware once with acetone, once with hexane, and three times with methylene chloride.

7.1.2 Mark the water level on the outside of bottle for later determination of volume.

7.1.3 Measure the pH of the sample and transfer to a pre-rinsed 2 L Teflon separatory funnel. (NOTE: If an evident layer of sediment is present, decant the sample and record on the extraction sheet.) The pH should be 5 - 9. If not, adjust the sample by using 10N NaOH or 1:1 H_2SO_4 and note on the extraction sheet.

7.1.4 Add 1.0 mL of the 1.0 µg/mL Surrogate Standard to all samples and QC. The final concentration will be 1.0 µg/L.

7.1.5 Add 1.0 mL of the 1.25 µg/mL Pesticide Spike Standard to samples selected for pesticide spiking. The final concentration will be 1.25 µg/L.

7.1.6 A typical batch will have an LCS and MS/MSD. An LCSD will be substituted if enough sample is not provided for a MSD.

7.1.7 Add approximately 60 mL methylene chloride to the sample bottle and rinse. Transfer to the separatory funnel with the sample. (NOTE: If the sample was decanted, add 60 mL of methylene chloride directly to the separatory funnel.)

7.1.8 Cap and shake vigorously for 10 seconds, and then vent. Cap and shake for two minutes. Allow the methylene chloride to separate from the sample.

7.1.8.1 If an emulsion interface between the layers is more than one-third the size of the solvent layer, the analyst should perform a beaker break without the use of Na_2SO_4.

7.1.8.2 Rinse the beaker with methylene chloride and quantitatively transfer. Drain the solvent layer into a glass 100 mm funnel containing a glass wool plug and about 2-3 inches of anhydrous muffled Na_2SO_4. Rinse funnel with methylene chloride.

7.1.9 Drain into a 500 mL K-D flask equipped with a 10 mL K-D concentrator for Nitrogen Blowdown.

7.1.10 Repeat with two additional fresh portions of methylene chloride.

7.1.11 After the final extraction, rinse the sodium sulfate with 20-30 mL of methylene chloride to complete the quantitative transfer.

7.1.12 If the emulsion layer is still present after the final shake, the analyst should employ mechanical techniques to complete the phase separation. Refer to the protocol found in Work Instruction (WI) 'Handling Emulsions'.

7.1.13 Fill sample bottle with tap water to mark made previously. Transfer to a graduated cylinder and record volume on extraction sheet.

7.1.14 K-D Technique / Nitrogen Blowdown

7.1.14.1 Add one solvent rinsed boiling bead to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-85 °C) so that the K-D concentrator is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot vapor. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 20 mL, add 20 mL of hexane through the Snyder column.

7.1.14.2 Concentrate to approximately 5 mL, remove the K-D apparatus from the steam bath, and allow it to cool. Remove the Snyder column.

7.1.14.3 Put the concentrator tube in a warm bath (about 35°C) and evaporate the solvent volume to less than 5 mL using a gentle stream of clean, dry nitrogen. During evaporation, the tube solvent level must be positioned to avoid water condensation (i.e., the solvent level should be below the level of the water bath). Do not allow the extract to go dry. Using a disposable pipet, adjust the final volume to 5.0 mL with hexane and mix the extract completely.

7.1.14.4 Transfer approximately 1 mL to a 2 mL autosampler vial and put the rest in a 4 mL saver vial. Store in freezer until analysis.

7.2 Preparation of Soil Samples – ASE method

7.2.1 The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.2.2 Place 2 filters paper on top of the open end of the cell body. Use the black cylindrical insertion tool to push filter to the bottom of the assembled cell body. Very fine soils may require three filters.

7.2.3 Use a 1:1 Ottawa Sand:Hydromatrix® mixture for the blank and Laboratory Control Sample (LCS). A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

7.2.4 Weigh approximately 15 g of sample into a prerinsed disposable aluminum dish. Record the weight to the nearest 0.01 g.

7.2.5 Mix the sample with Hydromatrix®, or equivalent, using approximately a 1:1 ratio by volume, until free-flowing. Depending on the matrix, the amount of sample may need to be reduced.

7.2.6 Place the extraction cell funnel on open end of cell body. Load sample into cell through funnel. Gently tap cell on hard surface to pack sample evenly, and to reduce void volume.

7.2.7 All samples should come within 1 cm of the top of the vessel. If a sample does not, use sand to fill.

7.2.8 When the transfer is complete, remove the funnel. Add 1.0 mL of 1.0 µg/mL Surrogate Standard to each sample, blank, and spike sample. Final concentration will be 0.067 mg/kg.

7.2.9 Add 1.0 mL of the 1.25 µg/mL Pesticide Spike Standard to samples selected for pesticide spiking. The final concentration will be 0.083 mg/kg.

7.2.10 Place filter paper on top of the sample. Either wipe cell threads with Kimwipes® or blow away any visible particles and screw the second end cap onto open end of the cell body, and tighten.

7.2.11 Place completed extraction cell into position #1 on ASE. Place the corresponding collection vial into position #1 below.

7.2.12 Repeat steps above for additional samples.

7.2.13 Fill solvent bottles with the 3:1 Hexane to Acetone Extraction Solvent Mix.

7.2.14 Make sure pressure on gas tank is set to 180 psi. Make sure that solvent bottle pressure is 10 psi, system air pressure is 50 psi, and compression oven pressure is 130 psi.

7.2.15 Refer to Equipment SOP entitled ‘ASE’ for equipment set-up and operation.

7.2.16 Assemble a K-D apparatus by attaching a 10 mL K-D concentrator to a 500 mL K-D flask for Nitrogen Blowdown or a 200 mL Turbo Vap II concentration vial.

7.2.17 Decant extraction solvent through a hexane rinsed funnel with sodium sulfate and glass wool. Rinse the collection vial three times with hexane to complete quantitative transfer. Collect the extract in the assembled K-D apparatus or a 200 mL Turbo Vap II concentration vial.

7.2.18 KD Technique / Nitrogen Blowdown

7.2.18.1 Add one solvent rinsed boiling bead to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of hexane to the top of the column. Place the K-D apparatus on a hot water bath (80-85 °C) so that the K-D concentrator is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot vapor. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

7.2.18.2 When the apparent volume of liquid reaches 5 mL, remove the K-D apparatus from the water bath and allow it to cool. Remove the Snyder column.

7.2.18.3 Put the K-D concentrator in a warm bath (about 35 °C) and evaporate the solvent volume to less than 5 mL using a gentle stream of clean, dry nitrogen. During evaporation, the tube solvent level must be positioned to avoid water condensation (i.e., the solvent level should be below the level of the water bath). Do not allow extract to go dry. Using a disposable pipet, adjust the final volume to 5.0 mL with hexane and mix the extract completely.

7.2.18.4 Transfer approximately 1 mL to a 2 mL autosampler vial and put the rest in a 4 mL saver vial. Store in freezer until analysis.

7.2.19 Turbo Vap II

7.2.19.1 Place the Turbo Vap collection tube in the Turbo Vap.

7.2.19.2 Set the water bath temperature to 40 °C and the pressure to 8-11 psi.

7.2.19.3 Set the Turbo Vap to monitor by ‘sensor’ to achieve a final volume of approximately 4 mL.

7.2.19.4 Using a disposable pipet, adjust the final volume to 5.0 mL with hexane and mix the extract completely. Transfer approximately 1 mL to a 2 mL autosampler vial and the remaining extract to a 4 mL saver vial. Store in freezer until analysis.

7.3 Preparation of Samples – Sonication Method

7.3.1 The following steps should be performed rapidly to avoid loss of the more volatile extractables.

7.3.2 Weigh approximately 15 g of sample into a prerinsed 250 mL beaker. Record the weight to the nearest 0.01 g on the extraction sheet.

7.3.3 Add approximately 15 g of anhydrous muffled Na_2SO_4 and mix the sample well. More sodium sulfate may be added until the sample is free flowing.

7.3.4 Add 1.0 mL of 1.0 µg/mL Surrogate Standard to each sample, blank, and spike sample. Final concentration will be 0.067 mg/kg, assuming a 5.0 mL final volume, 1 µL injection, and 15 g sample amount.

7.3.5 For the samples in each analytical batch selected for spiking, add 1.0 mL of the 1.25 µg/mL Pesticide Spike Standard. Final concentration will be 0.083 mg/kg, assuming a 5.0 mL final volume, 1 µL injection, and 15 g sample amount. A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

7.3.6 Immediately add 100 mL of 1:1 hexane/acetone.

7.3.7 Place the bottom surface of the tip of the 2" disrupter horn about 2" below the surface of the solvent, but above the soil/sediment layer.

7.3.8 Extract ultrasonically for 3 minutes, with output control knob set at 10, mode switch on Pulse and percent-duty cycle knob set at 50%.

7.3.9 Quantitatively transfer the solvent through a hexane-rinsed funnel with sodium sulfate and glasswool sitting atop a 500 mL K-D apparatus. Repeat steps above with two additional fresh portions of 1:1 hexane/acetone.

7.3.10 After the third aliquot of 1:1 hexane/acetone has been transferred, proceed with the KD Technique/Nitrogen Blowdown method described in the ASE method section.

7.4 Calibration

7.4.1 Prepare pesticide working standards at a minimum of five concentration levels, ranging from 0.080-0.40 µg/mL, by diluting the 10 µg/mL Pesticide Intermediate Solution with hexane. A typical calibration curve would be:

Inter. Solution (mL/25 mL)	Pest. Conc. (µg/mL)	Surr. Conc. (µg/mL)
0.20	0.080	0.080
0.25	0.10	0.10
0.50	0.20	0.20
0.75	0.30	0.30
1.0	0.40	0.40

7.4.2 Prepare toxaphene working standards at a minimum of three concentration levels, ranging from 0.20-1.0 µg/mL, by diluting the 1.0 µg/mL Toxaphene Intermediate Solution with hexane. A typical calibration curve would be:

Inter. Solution (mL/1.0 mL)	Conc. (µg/mL)
0.20	0.20
0.50	0.50
1.0	1.0

7.4.3 The average response factor should be calculated for each compound. The percent relative standard deviation (%RSD) should be < 20% for each compound. If the %RSD of any compound is < 20%, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

7.4.4 If the %RSD of any compound is > 20%, construct calibration curves of area versus concentration using a first order or linear fit. Correlation coefficients should be 0.990 or greater.

7.4.5 Calibration curve calculations are found in the QA Manual.

7.4.6 Reporting limit verification (RLV) is checked with each calibration curve or monthly at a minimum, by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.4.7 Use the Pesticide Second Source Standard as the continuing calibration standard – CCAL/CCVS.

7.4.8 Stock Standards should be stored in a freezer and replaced following manufacturer's expiration date or one year after opening, whichever comes first. Working standards should be stored in a freezer and replaced every 6 months, or sooner if analyses of continuing calibration standards indicate degradation or loss.

7.5 Analysis

7.5.1 GC Conditions

7.5.1.1 Columns: Rtx®-CLPesticides™, 30m x 0.32mm, 0.5 µm film (Restek #11139), and Rtx®-CLPesticidesII™, 30 m x 0.32 mm, 0.25 µm film (Restek #11324) or equivalent

7.5.1.2 Injector Temperature: 250 °C

7.5.1.3 Detector Temperature: 310 °C

7.5.1.4 ECD 1 Temp Program: 150 °C for 0.5 min, 10 °C/min ramp to 200 °C, 5.0 °C/min ramp to 310 °C

7.5.1.5 ECD 2 Temp Program: 175 °C for 0 min, 6 °C/min ramp to 300 °C

7.5.1.6 Flow Rate: 1.6 mL/min (ECD 1), 1.0 mL/min (ECD 2); Constant Flow

7.5.1.7 Split Ratio: 40:1 (ECD 1), 60:1 (ECD 2)

7.5.1.8 GC Range: 0

7.5.1.9 Attenuation: 0

7.5.1.10 Injection Volume: 1.0 µL

7.5.2 4,4'-DDT and endrin are easily degraded in the injection port. Analyze the 4,4'-DDT and Endrin Breakdown Standard at the start of a daily run and every 12 hours thereafter.

7.5.3 Using the 'DDT Endrin Breakdown' form, calculate the breakdown of 4,4'-DDT and endrin as given below on both the front and back detectors. Include the breakdown report in the daily file.

$$4,4'\text{-}DDT\ Breakdown = \frac{(DDE\ response + DDD\ response)}{(DDE\ response + DDD\ response + DDT\ response)}(100)$$

$$Endrin\ Breakdown = \frac{(Endrin\ aldehyde\ response + Endrin\ ketone\ response)}{(Endrin\ aldehyde\ response + Endrin\ ketone\ response + Endrin\ response)}(100)$$

7.5.4 Calculated breakdown must be < 15% for both 4,4'-DDT and endrin. If not, corrective action should be taken. Corrective action may include reinjection, making a new standard, performing maintenance and/or flagging data.

7.5.5 Analyze the Pesticide Second Source Standard (CCAL/CCVS) at the start of a daily run, every 12 hours or 20 samples after that, which ever comes first, and at the end of a sequence. Recoveries should be ± 15% for each analyte. For MN projects, recoveries can be ± 15% for all analytes collectively. When using the collective analyte list, individual compounds that exceeded ± 15% must be flagged on the report.

7.5.6 If recoveries are not met, corrective action should be taken. Corrective action may include reinjection, making a new standard, performing maintenance and/or flagging data.

7.5.7 Typical run order

7.5.7.1 Solvent blank

7.5.7.2 DDT/Endrin breakdown

7.5.7.3 Pesticide CCVS

7.5.7.4 Samples and QC

7.5.7.5 Pesticide CCVS

7.5.8 If the response for a peak exceeds the working range of the system or the highest standard, dilute the extract with hexane and re-analyze.

7.5.9 When identification is confirmed by a second column, calculate the %RPD of the quantitative results. The %RPD should be < 40%. If it isn't, corrective action should be taken. Corrective action may include checking for overlapping peaks, examining peak integration, and/or flagging data.

7.5.10 Various sample cleanup procedures are destructive to chlorinated pesticides. A first step in cleanup should simply be allowing sediment to settle and siphon off the top solvent layer. See 8081A for specific cleanup procedures and their advantages and disadvantages.

7.6 Calculation

7.6.1 Calculate the concentration of the analyte in the sample using one of the following equations:

$$Water\ Concentration\ (\mu g / L) = \frac{(C_{ex})(V_{ex})(F)}{V_o}$$

$$Soil\ Concentration\ (mg / kg) = \frac{(C_{ex})(V_{ex})(F)}{(W)(D)}$$

C_{ex} = extract concentration, µg/mL
V_{ex} = extract volume, mL
F = dilution factor (diluted volume/extract volume)
V_o = volume of sample extracted, L
W = sample weight, g
D = % dry weight of sample/100, or 1 for wet weight basis

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, when there is a change in the test method that may affect how the test is performed or when there is a major change in instrumentation. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the reporting limit or the sample batch is re-extracted if possible. If it is not possible to re-extract, the data will be flagged where appropriate. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Accuracy control limits are set at 70.0-130% for LCS and MS. Surrogate limits are generated semi-annually, using 20 Percent Recovery points, as follows:

9.3.1.1 Upper and Lower Control Limit = Mean ± 3s

9.3.1.2 Upper and Lower Warning Limit = Mean ± 2s

9.3.1.3 s = Standard deviation

9.3.1.4 For Arizona compliance, the surrogate lower control limit can not calculate below the lowest standard on the calibration curve (e.g. lowest standard = 0.08 µg/mL, spike is at 0.2 µg/mL, % can not be below 40.0%). The Minnesota Pollution Control Agency sets a guideline that the lower control limit can not be < 30.0%. For consistency between methods 608, 8081A, and 8082, LEGEND will use the greater of these which is 40.0% (8081A limit).

9.3.2 Precision control limits are set at 20.0% RPD for LCS/LCSD and generated for MS/MSD. In-house control charts are generated semi-annually, using 20 RPD points, as follows:

9.3.2.1 Control Limit = Mean + 3s

9.3.2.2 Warning Limit = Mean + 2s

9.3.2.3 s = Standard deviation

9.3.2.4 RPD = Relative Percent Difference

9.3.3 QC calculations are found in the QA Manual

9.3.4 LCS, MS and surrogates are reviewed.

9.3.5 If the LCS data are outside the limits, a method nonconformance is filled out and the sample batch is re-extracted and/or re-analyzed if possible. If the batch cannot be re-analyzed, the data are flagged and/or a case narrative is written for all client reports within the batch.

9.3.6 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS is flagged and/or a case narrative is written.

9.3.7 If a sample surrogate is outside the limits, the sample is re-extracted and/or reanalyzed if possible. If the sample cannot be reanalyzed, the data is flagged next to the actual result in the report.

10. REPORTING

10.1 Soil samples results are reported in mg/kg on a dry weight basis.

10.2 Toxaphene results for soil and water samples will only be calculated using data from the front column only.

10.3 Water sample results are reported in µg/L.

10.4 The reported result is rounded to two significant figures.

10.5 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 EPA Methods 3510C, 3545, 8081A, 8000B (MN), 8000C (AZ)

Appendix A

Initial Demonstration of Capability (IDC)
Organochlorinated Pesticides in Soil and Water Samples

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards in Ottawa sand and/or lab-grade water and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the %RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: 70.0-130%
   Precision: $\leq$ 20.0%

   If the standards were not extracted, the results must meet the following criteria:

   Accuracy: 85.0-115%
   Precision: ≤ 20% RPD

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Organochlorine Pesticides – Method 8081A

Parameter	Water MDL (µg/L)	Water RL (µg/L)	Soil MDL (mg/kg)	Soil RL (mg/kg)
α-BHC	0.028	0.40	0.0020	0.040
α-Chlordane	0.032	0.40	0.0020	0.040
Aldrin	0.033	0.40	0.0021	0.040
β-BHC	0.031	0.40	0.0022	0.040
δ-BHC	0.030	0.40	0.0020	0.040
Dieldrin	0.031	0.40	0.0020	0.040
Endosulfan I	0.032	0.40	0.0022	0.040
Endosulfan II	0.032	0.40	0.0021	0.040
Endosulfan sulfate	0.031	0.40	0.0021	0.040
Endrin	0.033	0.40	0.0020	0.040
Endrin aldehyde	0.040	0.40	0.0014	0.040
Endrin ketone	0.031	0.40	0.0021	0.040
γ-BHC (Lindane)	0.029	0.40	0.0021	0.040
γ-Chlordane	0.032	0.40	0.0021	0.040
Heptachlor	0.031	0.40	0.0021	0.040
Heptachlor epoxide	0.031	0.40	0.0020	0.040
Methoxychlor	0.029	0.40	0.0021	0.040
4,4'-DDD	0.031	0.40	0.0020	0.040
4,4'-DDE	0.031	0.40	0.0020	0.040
4,4'-DDT	0.031	0.40	0.0020	0.040
Toxaphene	0.29	1.0	0.016	0.080

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-025.9
REVIEWER:	Henrik Pham
DATE:	04/29/11

SECTION	CHANGE	RATIONALE
5.3, 5.5	Changed '4 ± 2 °C' to '≤ 6 °C but above freezing'	40 CFR, Part 136 update
Appendix B	Revised MDLs	Current MDL study

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**ANIONS IN AQUEOUS AND SOLID SAMPLES BY ION CHROMATOGRAPHY (IC)**
SOP NO.:	**LABENV-026.11**

Original Information		
Prepared by:	Jennifer Nelson	Date: 03/12/96
Technical Review:		Date:
QA/QC Coordinator:	Sharon Cenis	Date: 03/28/96
Authorized by:	Cheryl Sykora	Date: 03/29/96

Revision Information		
Supersedes:	LABENV-026.10	Date: 02/17/11
Revised by:	Scott Creekmur	Date: 05/03/11
Signature:	_____	Date: _____
Technical Review:	Tom Barrett	Date: 05/03/11
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 05/03/11
Signature:	_____	Date: _____

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Technical Review:	Tom Barrett	Date: 05/03/11
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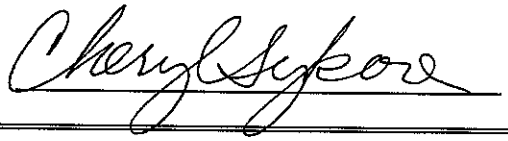
Authorized by:	Cheryl Sykora	Date: 05/03/11
Signature:		Date: 5/3/11

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SOP TITLE: ANIONS IN AQUEOUS AND SOLID SAMPLES BY ION CHROMATOGRAPHY (IC)

1. PURPOSE

1.1 This document defines the procedure to be followed for analysis of various anions in aqueous and solid samples by ion chromatography. A portion of the sample is injected onto an analytical column where the anions are separated in discrete bands. These discrete bands are then passed through a conductivity cell where the relative responses are measured and used for quantitation. The SOP is applicable to samples typically analyzed for anions by EPA 300.0 or EPA 9056A and perchlorate by a modified EPA 9056A.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This SOP is applicable to aqueous and solid samples.

3.2 Perchlorate analysis is limited to aqueous samples only.

3.3 Any species with a retention time similar to that of the desired ion will interfere. Large quantities of ions eluting close to the ion of interest will also result in an interference. Sample dilution and/or the use of the method of standard additions can also be used. For example, high levels of organic acids may be present in industrial wastes, which may interfere with inorganic anion analysis. Two common interference species, formate and acetate, elute between fluoride and chloride.

3.4 High levels of carbonate might interfere with both nitrite and sulfate and would require a dilution in order to obtain accurate results.

3.5 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water and solid samples may be collected in unpreserved plastic or glass bottles and stored at $\leq$ 6 °C but not freezing.

5.4 The recommended holding time for water samples and soil extracts is 48 hours from the date of collection for nitrate, nitrite, and phosphate. All other analytes are set at 28 days.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Dionex ICS-2100 with Anion Self-Regenerating Suppressor, or equivalent

6.2 Dionex IonPac AG18 Guard Column, or equivalent

6.3 Dionex IonPac AS18 Analytical Column, or equivalent

6.4 Dionex IonPac AG20 Guard Column, or equivalent for Perchlorate analysis only

6.5 Dionex IonPac AS20 Analytical Column, or equivalent for Perchlorate analysis only

6.6 KOH II Eluent Generator Cartridge, or equivalent

6.7 Polyvial 5 mL Disposable Autosampler Vials, or equivalent

6.8 Plaincaps for 5 mL Autosampler Vials, or equivalent

6.9 Analytical balance, capable of measuring to 0.1 mg

6.10 Calibrated Autopipetter with 200 µL to 1000 µL range, or equivalent

6.11 Calibrated Autopipetter with 20 µL to 200 µL range, or equivalent

6.12 Sonicating Bath

6.13 20 mL VOA Vial, or equivalent

6.14 Deionized (DI) Water (>16.3 MΩ)

6.15 Sodium Perchlorate, ≥ 98.0 %

6.16 Potassium Perchlorate, ≥ 98.0 %

6.17 Calibration Stock A –Anions Mixture 1 (Alltech Cat# 269106), or equivalent

6.17.1 Fluoride (F) = 1.0 mg/L

6.17.2 Chloride (Cl) = 5.0 mg/L

6.17.3 Nitrite (NO_2) = 5.0 mg/L

6.17.4 Sulfate (SO_4) = 5.0 mg/L

6.17.5 Bromide (Br) = 5.0 mg/L

6.17.6 Nitrate (NO_3) = 5.0 mg/L

6.17.7 Phosphate (PO_4) = 5.0 mg/L

6.18 Calibration Stock B – Seven Anion Standard II (Dionex Cat# 57590), or equivalent

6.18.1 Fluoride (F) = 20.0 mg/L

6.18.2 Chloride (Cl) = 100.0 mg/L

6.18.3 Nitrite (NO_2) = 100.0 mg/L

6.18.4 Sulfate (SO_4) = 100.0 mg/L

6.18.5 Bromide (Br) = 100.0 mg/L

6.18.6 Nitrate (NO_3) = 100.0 mg/L

6.18.7 Phosphate (PO_4) = 200.0mg/L

6.19 Spiking Solution – Anion Mix A (Alltech Cat# 26910200), or equivalent

6.19.1 Fluoride (F) = 10 mg/L

6.19.2 Chloride (Cl) = 20 mg/L

6.19.3 Nitrite (NO_2) = 20 mg/L

6.19.4 Sulfate (SO_4) = 30 mg/L

6.19.5 Bromide (Br) = 20 mg/L

6.19.6 Nitrate (NO_3) = 20 mg/L

6.19.7 Phosphate (PO_4) = 30 mg/L

6.20 Perchlorate Calibration Solution – weigh approximately 6.2 mg of sodium perchlorate into a 100 mL volumetric flask and bring to volume with DI water to produce a 50.4 mg/L solution of perchlorate. Shake to completely dissolve and mix the solution.

6.21 Perchlorate Spike Solution – weigh out approximately 5.6 mg of potassium perchlorate into a 100 mL volumetric flask and bring to volume with DI water to produce a 40.2 mg/L solution. Shake to completely dissolve and mix the solution.

NOTE: The final concentrations of the perchlorate calibration and spike solutions may vary depending on the exact amount of the standard weighed out.

7. PROCEDURE

7.1 Preparation of Aqueous Samples

7.1.1 Each sample should be filtered prior to analysis.

7.1.2 A typical batch for environmental applications will have a blank, a laboratory control spike (LCS) and matrix spike/matrix spike duplicate (MS/MSD). A laboratory control spike/laboratory control spike duplicate (LCS/LCSD) will be substituted if enough sample is not provided.

7.1.3 A typical batch for industrial chemistry applications will have a blank and a LCS/LCSD.

7.1.4 For the preparation of the LCS/LCSD, combine 400 µL of Anion Mixture A and 1,600 µL of DI water in an autosampler vial and mix. Final concentration will be 2.0 mg/L for fluoride, 6.0 mg/L for phosphate and sulfate, and 4.0 mg/L for the other anions.

7.1.5 For the preparation of the MS/MSD, combine 400 µL of Anion Mixture A and 1,600 µL of the sample selected for QC and mix. Final spiked concentration will be 2.0 mg/L for fluoride and 6.0mg/L for phosphate and sulfate, and 4.0 mg/L for the other anions.

7.1.6 For the preparation of the LCS/LCSD for perchlorate analysis, combine 100 µL of the 40 mg/L Perchlorate Spike Solution and 1,900 µL of DI water in an autosampler vial and mix. Final concentration will be 2.0 mg/L.

7.1.7 For the preparation of the MS/MSD for perchlorate analysis, combine 100 µL of the 40 mg/L Perchlorate Spike Solution and 1,900 µL of the sample selected for QC and mix. Final spiked concentration will be 2.0 mg/L.

7.2 Preparation of Solid Samples

7.2.1 Transfer 2.5 g of sample and 5.0 mL of DI water into a 20 mL VOA vial. If not enough sample is available, maintain a 1:2 sample:DI water ratio. Ensure all the sample is wetted and place into the sonicating bath for a minimum of 10 minutes and then allow the solids to settle.

7.2.2 Filter the liquid portion through a suitable syringe filter into an autosampler vial. Transfer a portion of this filtrate into a separate autosampler vial for analysis. Approximately 1.5 mL is needed. The remainder of the filtrate is retained for dilutions if needed.

7.2.3 A typical batch for environmental applications will have a blank, laboratory control spike (LCS) and matrix spike/matrix spike duplicate (MS/MSD). A laboratory control spike/laboratory control spike duplicate (LCS/LCSD) will be substituted if enough sample is not provided.

7.2.4 A typical batch for industrial chemistry applications will have a blank and an LCS/LCSD.

7.2.5 For the blank, add 5.0 mL of DI water into a 20 mL VOA vial, and place into the sonicating bath for a minimum of 10 minutes.

7.2.6 Follow the procedure above for filtering and transferring the sample.

7.2.7 For the preparation of the LCS/LCSD, add 1.0 mL Anion Mixture A and 4.0 mL of DI water to each into a 20 mL VOA vial and mix. Place into the sonicating bath for a minimum of 10 minutes.

7.2.8 Follow the procedure above for filtering and transferring the sample.

7.2.9 Final concentration, based on 2.5 g, will be 4.0 mg/kg for fluoride, 12 mg/kg for phosphate and sulfate and 8.0 mg/kg for the other anions.

7.2.10 For the preparation of the MS/MSD, transfer two aliquots of 2.5 g of the sample selected for spiking into 20 mL VOA vials. Add 1.0 mL Anion Mixture A and 4.0 mL of DI water to each and mix. Ensure all the sample is wetted and place into the sonicating bath for a minimum of 10 minutes and then allow the solids to settle.

7.2.11 Follow the procedure above for filtering and transferring the sample.

7.2.12 Final spiked concentration will be 4.0 mg/kg for fluoride, 12 mg/kg for phosphate and sulfate and 8.0 mg/kg for the other anions.

7.3 Calibration

7.3.1 Prepare anion working standards at a minimum of five concentration levels by diluting Calibration Stock A, Calibration Stock B, and Level 6 of the calibration curve. Typical calibration curves would be:

Level	Stock A mL/2 mL	Stock B mL/2mL	L6 mL/2mL	F	Cl	Br	NO_3	PO_4	SO_4
L1	0.040	-----	-----	0.020	0.10	0.10	0.10	0.10	0.10
L2	-----	-----	0.050	0.050	0.25	0.25	0.25	0.50	0.25
L3	-----	-----	0.150	0.15	0.75	0.75	0.75	1.5	0.75
L4	-----	0.050	-----	0.50	2.5	2.5	2.5	5.0	2.5
L5	-----	0.100	-----	1.0	5.0	5.0	5.0	10	5.0
L6	-----	0.200	-----	2.0	10	10	10	20	10

NOTE: Solution volumes may be modified, provided final concentrations are maintained.

7.3.2 Prepare perchlorate working standards at a minimum of five concentration levels by diluting the 50 mg/L Perchlorate Calibration Solution. Typical calibration curves would be:

Level	Level 4 mL/2mL	Calibration Solution mL/2mL	Final Conc. mg/L
L1	0.20	-----	0.10
L2	0.50	-----	0.25
L3	-----	0.020	0.50
L4	-----	0.040	1.0
L5	-----	0.120	3.0
L6	-----	0.240	6.0

NOTE: The exact concentrations will vary depending on the Perchlorate Calibration Solution concentration, but the lowest curve concentration must be at or below 0.10 mg/L and the highest curve concentration should not exceed 6.0 mg/L.

7.3.3 Prepare calibration curves of Concentration vs. Response. Correlation Coefficient (r^2) should be 0.990 or greater.

7.3.4 Calibration curve calculations are found in the QA Manual.

7.3.5 Reporting limit verification (RLV) is checked with each calibration curve or monthly at a minimum, by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.4 Analysis

7.4.1 Recommended IC conditions for Anions

7.4.1.1 Mobile Phase – 28.0 mM Potassium Hydroxide

7.4.1.2 Dionex AG18 and AS18 columns

7.4.1.3 Flow Rate – 1.0 mL/min

7.4.1.4 Column Temperature – 30 °C

7.4.1.5 Injection Amount– 20 µL

7.4.1.6 Suppressor Current – 70 mA

7.4.1.7 Cell Temperature – 35 °C

7.4.2 Recommended IC conditions for Perchlorate

7.4.2.1 Mobile Phase – 40.0 mM Potassium Hydroxide

7.4.2.2 Dionex AG20 and AS20 columns

7.4.2.3 Flow Rate – 1.0 mL/min

7.4.2.4 Column Temperature – 30 °C

7.4.2.5 Injection Amount – 20 µL

7.4.2.6 Suppressor current – 100 mA

7.4.2.7 Cell Temperature – 35 °C

7.4.3 A mid-level or calibration verification standard (CVS) is analyzed at the beginning of the run (if an initial calibration curve was not analyzed), after every twenty samples, and at the end of the run. Recoveries should be ± 10% or corrective action should be taken.

7.4.4 Corrective action may include reanalyzing the CVS and/or flagging the data in the daily file.

7.4.5 If the response for a peak exceeds the working range of the system or the highest standard, dilute the sample with water and re-analyze.

7.5 Calculation

7.5.1.1 Compute the concentration of the analyte in the sample using the following equation:

$$\textit{Water Concentration}\ (mg/L) = (C)(F)$$

$$\textit{Soil Concentration}\ (mg/Kg) = \frac{(C)(V)(F)}{(W)(D)}$$

C = on-column, mg/L (prior to dilutions)
F = dilution factor
V = volume of DI used, mL
W = sample weight, g
D = % dry weight of sample/100, or 1 for wet weight basis

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

9. QA/QC

9.1 Method Blank

9.1.1 A method blank is analyzed with each batch of samples prepared at the same time. The method blank must be less than the reporting limit or the data will be flagged, where appropriate. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.2 MDL, PQL, RL

9.2.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, when there is a change in the test method that may affect how the test is performed or when there is a major change in instrumentation. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDL and RL values can be found in Appendix B. Project specific RLs may override those listed.

9.3 Control Limits

9.3.1 Method accuracy and precision limits are set at 80.0 – 120% and 15 % RPD for both LCS and MS. Sample duplicate precision limit is set at 15 % RPD.

9.3.2 QC calculations are found in the QA Manual.

9.3.3 LCS and MS data are reviewed.

9.3.4 If the LCS data are outside the limits, a method nonconformance is filled out and the sample batch is re-analyzed if possible. If the batch cannot be re-analyzed, the information is placed in the daily and project files, the data are flagged and/or a case narrative is written for all client reports within the batch.

9.3.5 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS is flagged and/or a case narrative is written.

9.3.6 If the sample duplicate data are outside the limits, the data for that specific duplicate is flagged and/or a case narrative is written.

10. REPORTING

10.1 Aqueous sample results are reported in mg/L.

10.2 Solid sample results are reported in mg/kg on a dry weight basis.

10.3 The reported result is rounded to two significant figures.

10.4 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 Manufacturer's Operating Manuals

12.2 EPA Method 9056A

12.3 EPA Method 300.0

Appendix A

Initial Demonstration of Capability (IDC)
Anions in Aqueous and Solid Samples by IC

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards of all the parameters in Ottawa sand and/or lab-grade water and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: LCS limits
   Precision: LCS limits

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Anions by IC

Parameter	Aqueous MDL (mg/L)	Aqueous RL (mg/L)	Solid MDL (mg/kg)	Solid RL (mg/kg)
Fluoride	0.0019	0.020	0.0038	0.040
Chloride	0.0029	0.10	0.0058	0.20
Nitrite as NO_2	0.0075	0.10	0.015	0.20
Nitrite as N (calculated)	0.0023	0.030	0.0046	0.061
Sulfate as SO_4	0.024	0.10	0.048	0.20
Bromide	0.016	0.10	0.032	0.20
Nitrate as NO_3	0.020	0.10	0.040	0.20
Nitrate as N (calculated)	0.0045	0.023	0.0090	0.045
Phosphate as PO_4	0.021	0.10	0.042	0.20
Perchlorate	0.019	0.10	NA	NA

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-026.11
REVIEWER:	Scott Creekmur
DATE:	05/03/11

SECTION	CHANGE	RATIONALE
1.1	Removed 'modified' and added 'A' to 9056	Current protocol
3.2	Added reference to perchlorate analysis in aqueous samples only.	New analyte limitations
5.3	Changed '4 ± 2 °C' to '≤ 6 °C but above freezing'	40 CFR, Part 136 update
5.4	Added holding time for nitrate, nitrite, and phosphate as 48 hours, rest are set at 28 days	Per method 9056A for the 48 hours, lab set for 28 days
6.4 & 6.5	Added dionex AG20 and AS20 columns	Column for perchlorate analysis
6.15 & 6.16	Added perchlorate standards	New analyte reference standards
6.20	Added perchlorate calibration intermediate prep	New analyte
6.21	Added perchlorate spike intermediate prep	New analyte
7.1.6	Included procedure on preparation of LCS/LCSD for perchlorate analysis only	New protocol
7.1.7	Included procedure on preparation of MS/MSD for perchlorate analysis only	New protocol
7.3.2	Added calibration table for perchlorate	New analyte
7.4.1	Added "Anions"	Delineated run conditions for the Anions analysis
7.4.1.2	Added "AG18 and AS18 columns"	Specified Anions analysis columns to use
7.4.2	Added perchlorate run condtions	New protocol
Appendix B	Added perchlorate mdl and rl	New protocol
9.3.1	Added EPA 9056A method limits	New reference method
9.3.3-9.3.5	Deleted	Method limits, not calculated limits
9.3.6	Added sample duplicate protocol	Current protocol

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF NITROAROMATICS AND NITROAMINES IN SOIL BY UPLC

SOP NO.: LABENV-036.7

Original Information		
Prepared by:	Tom Barrett	Date: 09/17/99
Technical Review:		Date: 09/17/99
QA/QC Coordinator:	Sharon Dahl	Date:
Authorized by:	Cheryl Sykora	Date: 09/17/99

Revision Information		
Supersedes:	LABENV-036.6	Date: 05/27/09
Revised by:	Scott Creekmur	Date: 05/03/11
Signature:	_____	Date: _____
Technical Review:	Tom Barrett	Date: 05/03/11
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 05/03/11
Signature:	_____	Date: _____

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LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF NITROAROMATICS AND NITROAMINES IN SOIL BY UPLC

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Revision Information		
Supersedes:	LABENV-036.6	Date: 05/27/09
Revised by:	Scott Creekmur	Date: 05/03/11
Signature:		Date: 5-3-11
Technical Review:	Tom Barrett	Date: 05/03/11
Signature:		Date: 5/3/11
Authorized by:	Cheryl Sykora	Date: 05/03/11
Signature:		Date: 5/3/11

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SOP TITLE: DETERMINATION OF NITROAROMATICS AND NITROAMINES IN SOIL BY UPLC

1. PURPOSE

1.1 This document defines the procedure to be followed for the determination of nitroaromatics and nitroamines in soil and solids by an Ultra High Performance Liquid Chromatograph (UPLC) using a photo diode array (PDA) detector. The SOP is applicable to samples typically analyzed by a modified EPA 8330.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 An analyst trained by LEGEND Technical Services, Inc. shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 The procedure is limited to soil and solid samples.

3.2 Degradation products of tetryl appear as a shoulder on the 2,4,6-Trinitrotoluene peak. Peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-Trinitrotoluene.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 When working with organic compounds, wear solvent resistant gloves.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Soil samples should be collected in unweighed 4 oz. glass jars with Teflon lined caps and stored at $\leq$ 6 °C but not freezing. Samples must be protected from light.

5.4 The recommended holding time for soil samples is 14 days until extraction and analysis within 40 days of extraction.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 UPLC with photodiode array detector (PDA)

6.2 Waters Acquity UPLC BEH C18 column, 100 mm x 2.1 mm x 1.7 µm, or equivalent

6.3 Balance – capable of reading 0.1 g

6.4 Chilled Ultrasonic Water Bath

6.5 10 mL amber serum vials with PTFE septa

6.6 Amber autosampler vials – 2 mL

6.7 Acrodisc (0.45 µm) – PTFE, or equivalent

6.8 Disposable Syringes with luer-lock tip – 3 mL, or equivalent

6.9 25 µL, 50 µL, 250 µL, 500 µL and 1.0 mL syringes

6.10 Sand – Ottawa, or equivalent

6.11 Deionized (DI) Water (>16.3 MΩ)

6.12 Acetonitrile (ACN), HPLC grade

6.13 Methanol (MeOH), HPLC grade

6.14 8330 Calibration/Spike Stock – 1,000 µg/mL EPA 8330 Kit, Ultra# NAIM-833E, or equivalent

6.15 Surrogate Stock – 1,000 µg/mL 1,2-dinitrobenzene, Ultra# IST-600, or equivalent

6.16 Second Source 8330 Stock – 1,000 µg/mL, different vendor or different lot number than stock used for calibration

6.17 Second Source Surrogate Stock – 1,000 µg/mL, different vendor or different lot number than stock used for calibration

6.18 8330 Continuing Calibration (CCAL) Solution – dilute 125 µL of the 1,000 µg/mL Second Source 8330 Stock and 1,000 µg/mL Second Source Surrogate Stock to 50 mL with acetonitrile to produce a 2.5 µg/mL 8330 Continuing Calibration (CCAL) Solution

6.19 8330 Spike Working Solution – dilute 0.5 mL of the 1,000 µg/mL 8330 Calibration/Spike Stock to 25 mL with acetonitrile to produce a 20 µg/mL 8330 Spike Working Solution

6.20 Surrogate Working Solution – dilute 0.5 mL of the 1,000 µg/mL Surrogate Stock to 25 mL with acetonitrile to produce a 20 µg/mL Surrogate Working Solution

7. PROCEDURE

7.1 Preparation of Samples

7.1.1 Weigh 2.0 g of sand into two 10 mL serum vials for the method blank and Laboratory Control Spike (LCS).

7.1.2 Weigh 2.0 g of each sample into a 10 mL serum vial. In each analytical batch, choose a sample and weigh out two additional aliquots, one for the Matrix Spike (MS) and one for the Matrix Spike Duplicate (MSD).

7.1.3 Add 0.5 mL of the 20 µg/mL Surrogate Working Solution to each sample, blank, LCS, and MS/MSD. The final concentration will be 5.0 mg/Kg.

7.1.4 For the samples in each analytical batch selected for spiking, add 0.5 mL of the 20 µg/mL 8330 Spike Working Solution. A typical batch will have an LCS and MS/MSD (an LCS/LCSD will be substituted if insufficient sample is provided). The final concentration will be 5.0 mg/Kg.

7.1.5 Add 4.5 mL of HPLC grade acetonitrile to the Blank and samples and 4.0 mL to the LCS and MS/MSD and close the vial with Teflon lined closure.

7.1.6 Place all the vials in a cooled ultrasonic bath for 18 ± 1 hours.

7.1.7 After 18 hours, remove all the vials and let them stand for approximately 15 minutes to settle.

7.1.8 Filter a portion of the extract through a 0.45 µm PTFE acrodisc and place the filtrate in an autosampler vial.

7.1.9 Retain the remainder of the extract in a Teflon capped vial.

7.2 Calibration

7.2.1 Prepare working standards at a minimum of five concentration levels, ranging from 0.25 – 7.5 µg/mL, by diluting the 1000 µg/mL 8330 Calibration/Spike Stock and the 1000 µg/mL Surrogate Stock with acetonitrile. A typical calibration curve would be:

Level	From Level 5 µL/10 mL	Calibration Stocks µL/10 mL	Final Concentration µg/mL
L1	500	-----	0.25
L2	1000	-----	0.50
L3	-----	10	1.0
L4	-----	25	2.5
L5	-----	50	5.0
L6	-----	75	7.5

7.2.2 The average response factor should be calculated for each analyte. The percent relative standard deviation (%RSD) should be less than 20% for each analyte. If the RSD for any analyte is greater than 20%, review the results (area counts, response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the calibration standards.

7.2.3 If the problem appears to be associated with a single standard, reprep and/or reanalyze that standard and calculate the RSD again.

7.2.4 If the %RSD is still greater than 20%, construct calibration curves of area versus concentration using a first order or linear fit. Correlation coefficients should be 0.990 or greater.

7.2.5 Calibration curve calculations are found in the QA Manual.

7.2.6 Reporting limit verification (RLV) is checked with each calibration curve by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or flagging data.

7.2.7 All extracts should be stored in the refrigerator in the dark.

7.3 Analysis

7.3.1 HPLC Conditions

7.3.1.1 Waters Acquity UPLC BEH C18 column, 100 mm x 2.1 mm x 1.7 µm

7.3.1.2 HPLC Mobile Phase – 69:31 H_20:MeOH

7.3.1.3 Column temperature 50 °C

7.3.1.4 PDA Wavelength 254 nm

7.3.1.5 Flow Rate 0.5 mL/min, run time 7.25 min, isocratic

7.3.1.6 Injection volume 1 µL

7.3.2 Sample peak identification will be confirmed by peak retention time and peak spectrum as compared to standards.

7.3.3 If the response for a peak exceeds the working range of the system or the highest standard, dilute the extract with acetonitrile and re-analyze.

7.3.4 The CCAL is analyzed at the beginning of the run in triplicate (if an initial calibration curve was not analyzed), after every ten samples (singly), and at the end of the run (singly). Recoveries for the triplicates should be ± 15% of the initial curve or corrective action should be taken. Recoveries for the continuing and end CCAL should be ± 15% of the triplicate average.

7.3.5 Corrective action may include reanalyzing the CCAL and/or flagging the data in the daily file.

7.4 Calculation

7.4.1 Computer software calculates the concentration of the sample based on the response. The calculation yields the final result in µg/g, which is equal to mg/kg.

$$Explosives\ (mg/kg) = \frac{(CC)(V)}{(g)(D)}$$

CC = concentration on column (µg/mL)

V = final volume (mL)
g = amount of soil (g)
D = % dry weight of sample/100, or 1 for wet weight basis

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, when there is a change in the test method that may affect how the test is performed or when there is a major change in instrumentation. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the reporting limit or the sample batch is re-analyzed, if possible. If it is not possible to re-analyze, the data will be flagged where appropriate. Do not subtract analytes in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Accuracy control limits are set at 70.0-130% for LCS, MS and surrogates.

9.3.2 Precision control limits are set at 20.0% RPD (relative percent difference) for LCS/LCSD and MS/MSD.

9.3.3 QC calculations are found in the QA Manual.

9.3.4 LCS, MS and surrogates are reviewed.

9.3.5 If the LCS data are outside the limits, the sample batch is re-extracted and/or re-analyzed if possible. If the batch cannot be re-analyzed, the information is placed in the daily and project files, and a case narrative is written for all client reports within the batch.

9.3.6 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS may be flagged in the case narrative of the report.

9.3.7 If a sample surrogate is outside the limits, the sample is re-extracted and/or reanalyzed if possible. If the sample cannot be re-extracted and/or reanalyzed, the data is flagged next to the actual result in the report.

10. REPORTING

10.1 Soil samples results are reported in mg/kg on a dry weight basis.

10.2 The reported result is rounded to two significant figures.

10.3 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 EPA Method 8000B

12.2 EPA Method 8330

Appendix A

Initial Demonstration of Capability (IDC)
Determination of Nitroaromatics and Nitroamines

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards in Ottowa sand, and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: LCS limits (70.0-130%)
   Precision: LCS limits (≤ 20% RPD)

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Nitroaromatics and Nitroamines – EPA Method 8330(M)

Parameter	Soil MDL (mg/kg)	Soil RL (mg/kg)
1,3,5-Trinitrobenzene	0.043	1.0
1,3-Dinitrobenzene	0.047	1.0
2,4,6-Trinitrotoluene	0.13	1.0
2,4-Dinitrotoluene	0.039	1.0
2,6-Dinitrotoluene	0.089	1.0
2-Amino-4,6-dinitrotoluene	0.072	1.0
2-Nitrotoluene	0.16	1.0
3-Nitrotoluene	0.24	1.0
4-Amino-2,6-dinitrotoluene	0.075	1.0
4-Nitrotoluene	0.23	1.0
HMX	0.11	1.0
Nitrobenzene	0.066	1.0
RDX	0.078	1.0
Tetryl	0.17	1.0

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-036.7
REVIEWER:	Scott Creekmur
DATE:	05/03/11

SECTION	CHANGE	RATIONALE
All	Major Revision	New Instrumentation

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**MERCURY SAMPLE PREPARATION FOR COLD VAPOR GENERATION**
SOP NO.:	**LABENV-037.9**

Original Information		
Prepared by:	Lisa Bloomgren	Date: 01/18/02
Technical Review:		Date:
QA/QC Coordinator:	Terri Olson	Date: 02/26/02
Authorized by:	Cheryl Sykora	Date: 02/26/02

Revision Information		
Supersedes:	LABENV-037.8	Date: 04/27/09
Revised by:	Dan Brezina	Date: 11/22/10
Signature:	_____	Date: _____
Technical Review:	William Dahl	Date: 11/22/10
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 11/22/10
Signature:	_____	Date: _____

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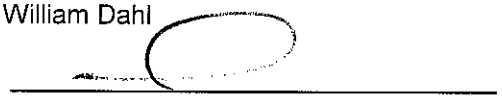
LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**MERCURY SAMPLE PREPARATION FOR COLD VAPOR GENERATION**
SOP NO.:	**LABENV-037.9**

Original Information		
Prepared by:	Lisa Bloomgren	Date: 01/18/02
Technical Review:		Date:
QA/QC Coordinator:	Terri Olson	Date: 02/26/02
Authorized by:	Cheryl Sykora	Date: 02/26/02

Revision Information		
Supersedes:	LABENV-037.8	Date: 04/27/09
Revised by:	Dan Brezina	Date:
Signature:		Date: 11-22-10
Technical Review:	William Dahl	Date:
Signature:		Date: 11/22/10
Authorized by:	Cheryl Sykora	Date:
Signature:		Date: 11/22/10

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SOP TITLE: MERCURY SAMPLE PREPARATION FOR COLD VAPOR GENERATION

1. PURPOSE

1.1 This document defines the procedure to be followed for preparing samples to be analyzed for mercury by the cold vapor atomic absorption technique. The SOP is applicable to mercury samples typically analyzed by EPA 245.1, EPA 7470A, and EPA 7471A.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the SOP.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 Potassium permanganate is added to eliminate possible interferences from sulfide.

3.2 If the laboratory will be filtering the sample for dissolved mercury analysis, do not perform nitric acid preservation.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 Safety glasses and gloves should be worn when handling samples and reagents.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in a polyethylene or glass container and preserved with 1:1 nitric acid to a pH < 2.

5.4 If a water sample is received with pH > 2, it is preserved in the laboratory and allowed to sit for a minimum of 24 hours at which time the pH is re-tested. The addition of the acid is noted on the appropriate chain-of-custody. If pH < 2, proceed. If not, repeat until pH is < 2, if possible. Highly alkaline samples may need to be digested at a reduced volume with excess acid, rather than dilute the original sample in an attempt to lower the pH.

5.5 Document the final pH of all samples on the bench sheet under "Comments."

5.6 The recommended holding time for water samples is 28 days.

5.7 Solid samples should be collected in polyethylene or glass containers and stored at 4 ± 2 °C.

5.8 The recommended holding time for solid samples is 28 days.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Top loading balance

6.2 Block digester

6.3 Digestion vessels

6.4 Plunge filters

6.5 Disposable pipets

6.6 Deionized (DI) water (>16.3 MΩ)

6.7 Nitric Acid (HNO_3), concentrated, trace metal grade

6.8 Hydrochloric acid (HCl), concentrated, trace metal grade

6.9 Aqua regia (3:1 HCl:HNO_3 solution) – prepare immediately before use

6.10 5% potassium permanganate solution

6.11 Hydroxylamine hydrochloride, reagent grade

6.12 Certified Calibration Stock Standard – 1000 ppm (two different lots numbers are used)

6.13 Intermediate Stock Standard 1 (ISS1) – dilute 2.5 mL of the 1000 ppm certified stock standard (first lot number) and 12.5 mL of Aqua Regia with DI water in a 250 mL volumetric flask to produce a 10 ppm solution

6.14 Intermediate Stock Standard 2 (ISS2) – dilute 2.5 mL of the 1000 ppm certified stock standard (second lot number) and 12.5 mL of Aqua Regia with DI water in a 250 mL volumetric flask to produce a 10 ppm solution

6.15 Mercury Working Standard Solution 1 (MWSS1) – dilute 5.0 mL of ISS1 and 25 mL of Aqua Regia with DI water in a 500 mL volumetric flask to produce a 0.10 ppm solution and use this solution for calibration (calibration standards range from 0.50 – 10 ppb), LCS, LCSD, MS, and MSD – the spike concentration for the LCS, LCSD, MS, and MSD is 2.0 ppb

6.16 Mercury Working Standard Solution 2 (MWSS2) – dilute 5.0 mL of ISS2 and 25 mL of Aqua Regia with DI water in a 500 mL volumetric flask to produce a 0.10 ppm solution and use this solution for the QC standard or second source – the concentration for the second source standard is 5.0 ppb

7. PROCEDURE

7.1 Confirm sample ID and requested analyte information with the chain-of-custody (c-o-c).

7.2 For Quality Control (QC), indicate the prep batch # under the project # column for the Blank, Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD). The LCS is also referred to as the Laboratory Fortified Blank (LFB) or Blank Spike (BS). The prep batch number is obtained from the LIMS system. For Matrix Spike (MS) and Matrix Spike Duplicate (MSD), indicate the project and sample number in the appropriate columns. MS is also referred to as the Laboratory Fortified Matrix (LFM). A batch consists of no more than 20 samples. If less than 20 samples are in the batch, "Z" out the remaining spaces on that page, initial, and date.

7.3 Label digestion vessels with sample IDs. Label digestion vessels for the Blank, LCS, and LCSD including the batch number. Along with the sample ID, label MS for sample matrix spike and MSD for sample matrix spike duplicate.

7.4 Preparation of Water Samples

7.4.1 Shake all samples prior to transferring into digestion vessels.

7.4.2 Add 1.0 mL of MWSS1 to the digestion vessels labeled LCS, LCSD, MS, and MSD (obtaining a concentration of 2.0 ppb).

7.4.3 Dilute, with DI water, the blank, LCS, and LCSD to the 50 mL graduation on the digestion vessel. Dilute, with sample, the MS and MSD to the 50 mL graduation on the digestion vessel.

7.4.4 Measure 50 mL of each sample into its respective digestion vessel, using the graduations on the vessel.

7.4.5 Using a disposable pipet, add 2.5 mL of aqua regia to all the digestion vessels.

7.4.6 Add 5.0 mL of 5% potassium permanganate solution to each vessel. Less sample volume should be used if the potassium permanganate is significantly reduced before placing in block digester. Additional potassium permanganate may be used in conjunction with a lesser sample volume.

7.4.7 Loosely cover each digestion vessel with its cap. Place the samples, spikes, and blank in the block digester for 2 hours at 95 °C. Record the time on the bench sheet.

7.4.8 Remove the samples from the block and allow them to cool. Record the time in the on the bench sheet.

7.4.9 Add approximately 400 mg (~½ inch back on tip of spatula) of hydroxylamine hydrochloride to reduce the potassium permanganate, cap tightly and mix.

7.4.10 If sediment is present, filter the sample using a plunge filter. Also filter the QC at this point, including the Blank, LCS, and LCSD. If samples are not being filtered, the QC does not need to be filtered.

7.5 Preparation of TCLP Samples

7.5.1 Shake all samples prior to transferring into digestion vessels.

7.5.2 Add 1.0 mL of MWSS1 to the digestion vessels labeled LCS, LCSD, MS, and MSD (obtaining a concentration of 2.0 ppb).

7.5.3 Add 10 mL of the TCLP leachate blank to the batch Blank, LCS and LCSD samples.

7.5.4 Add 10 mL of each TCLP sample to the appropriate digestion vessel.

7.5.5 Dilute, with DI water, all samples including the Blank, LCS, and LCSD to the 50 mL graduation on the digestion vessel.

7.5.6 Using a disposable pipet, add 2.5 mL of aqua regia to all the digestion vessels.

7.5.7 Add 5.0 mL of 5% potassium permanganate solution to each vessel. Less sample volume should be used if the potassium permanganate is significantly reduced before placing in block digester. Additional potassium permanganate may be used in conjunction with a lesser sample volume.

7.5.8 Loosely cover each digestion vessel with its cap. Place the samples, spikes, and blank in the block digester for 2 hours at 95 °C. Record the time on the bench sheet.

7.5.9 Remove the samples from the block and allow them to cool. Record the time on the bench sheet.

7.5.10 Add approximately 400 mg (~½ inch back on tip of spatula) of hydroxylamine hydrochloride to reduce the potassium permanganate, cap tightly and mix.

7.5.11 If sediment is present, filter the sample using a plunge filter. Also filter the QC at this point, including the Blank, LCS, and LCSD. If samples are not being filtered, the QC does not need to be filtered.

7.6 Preparation of Solid Samples

7.6.1 Weigh out 0.50 grams of the solid samples and place in their respective digest vessel. For one of the solid samples, weigh out two additional aliquots, one for the MS and one for the MSD. Record the actual weights on the bench sheet and LIMS.

7.6.2 Add 1.0 mL of MWSS1 to the digestion vessels labeled LCS, LCSD, MS, and MSD (obtaining a concentration of 2.0 ppb).

7.6.3 Using a disposable pipet, add 2.5 mL of aqua regia to all the digestion vessels.

7.6.4 Dilute the samples, blank, LCS, LCSD, MS, and MSD with DI water to the 50 mL graduation and then add an extra 2.5 mL of DI water.

7.6.5 Add 5.0 mL of the 5% potassium permanganate solution to each vessel. Each vessel should now have a volume of 57.5 mL.

7.6.6 If the potassium permanganate is significantly reduced before placing in the block digester, the sample should be re-prepped with additional potassium permanganate ensuring final volume is 57.5 mL.

7.6.7 Loosely cover each digestion vessel with its cap. Place the samples, spikes, and blank in the block digester for 30 minutes at 95 °C. Record the time on the bench sheet.

7.6.8 Remove the samples from the block and allow them to cool. Record the time on the bench sheet.

7.6.9 Add approximately 400 mg (~½ inch back on tip of spatula) of hydroxylamine hydrochloride to reduce the potassium permanganate, cap tightly and mix.

7.6.10 Filter samples including the blank, LCS, and LCSD using the plunge filters.

7.7 Calibration, analysis and calculation are not applicable to this SOP but are addressed in the appropriate analytical SOP.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 Follow the QA/QC protocol outlined in the appropriate analytical SOP.

10. REPORTING

Not applicable

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

12. REFERENCES

12.1 EPA Methods 245.1, 7470A, and 7471A

Appendix A

Initial Demonstration of Capability (IDC)
Mercury Sample Preparation for Cold Vapor Generation

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: 245.1 = 85.0-115%, 7470A/7471A – 80.0-120% (245.1/7470A may be combined if tighter limits are used for acceptance)
   Precision: 245.1/7470A/7471A = $\leq$ 20 % RPD

7. The reagent blank must be less than the reporting limit (RL).

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-037.9
REVIEWER:	Dan Brezina
DATE:	11/22/10

SECTION	CHANGE	RATIONALE
Various	Deleted digestion log and added reference to LIMS bench sheet	Current procedure
7.1	Deleted reference to digestion log, printed from LIMS	Current procedure

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: ANALYSIS OF SAMPLES BY AXIAL ICP-AES

SOP NO.: LABENV-039.6

Original Information		
Prepared by:	William Dahl	Date: 05/24/00
Technical Review:	Sharon Dahl	Date: 05/24/00
QA/QC Coordinator:		Date:
Authorized by:	Cheryl Sykora	Date: 09/28/00

Revision Information		
Supersedes:	LABENV-039.5	Date: 04/07/08
Revised by:	William Dahl	Date:
Signature:	_____	Date: _____
Technical Review:	Dan Brezina	Date:
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date:
Signature:	_____	Date: _____

When printed, this is an uncontrolled copy

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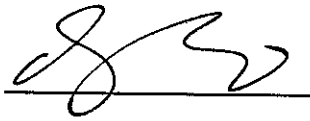
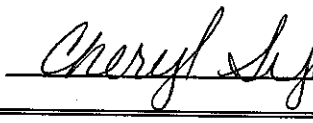
Supersedes:	LABENV-039.5	Date: 04/07/08
Revised by:	William Dahl	Date:
Signature:		Date: 11/23/10
Technical Review:	Dan Brezina	Date:
Signature:		Date: 11-23-10
Authorized by:	Cheryl Sykora	Date:
Signature:		Date: 11/23/10

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SOP TITLE: ANALYSIS OF SAMPLES BY AXIAL ICP-AES

1. PURPOSE

1.1 This document defines the analysis for various metals by axially viewed inductively coupled plasma atomic emission spectroscopy (ICP-AES). The SOP is applicable to samples typically analyzed by EPA 200.7, EPA 6010B, and a modified NIOSH 7303.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the SOP.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is applicable to digestates of various matrices.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 Safety glasses and gloves should be worn when handling samples and reagents.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in polyethylene or glass containers and preserved with 1:1 nitric acid to a pH < 2. Samples to be filtered in the laboratory should be collected in an unpreserved bottle.

5.4 If a water sample is received with pH > 2 then it is preserved in the laboratory and allowed to sit for a minimum of 24 hours at which time the pH is re-tested. If pH < 2, proceed. If not, repeat until pH is < 2, if possible. Highly alkaline samples may need to be digested at reduced volume with excess acid, rather than dilute the original sample in an attempt to lower the pH.

5.5 Document pH and/or lab filtration on raw data batch bench sheet. Document the addition of acid and the timing in the sample comments and work order comments in the work order section of the LIMS.

5.6 The recommended holding time for water samples is six months.

5.7 Solid samples should be collected in polyethylene or glass containers and stored at 4 ± 2 °C or at ambient temperature depending on the metals requested. Sample are immediately stored at 4 ± 2 °C and then moved to ambient if appropriate to do so.

5.8 The recommended holding time for solid samples is six months.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Varian Model VISTA AX™ ICP-AES Instrument, or equivalent

6.2 Varian Model SPS-5™ Autosampler, or equivalent

6.3 Assorted laboratory glassware

6.4 15 mL disposable centrifuge tubes

6.5 Nitric acid (HNO_3) - concentrated, trace metal grade

6.6 Hydrochloric acid (HCl) - concentrated, trace metal grade

6.7 De-ionized (DI) water (>16.3 MΩ)

6.8 Appropriate purchased traceable single element stock standards

6.9 Calibration Stock Standards - various concentration levels, Inorganic Ventures #LTS-STOCK-1A, #LTS-STOCK-2A, and #LTS-STOCK-3A, or equivalent

6.10 Second Source Stock Standards - various concentration levels, use lot number that is separate than that of calibration standards.

6.11 Interference standards – ICSA (IFA) (Fe = 300 mg/L and Al, Ca, Mg = 200 mg/L each)

6.12 Low level check standards – prepare at reporting levels.

6.13 Record all standards traceability information in the LIMS system.

6.14 Liquid Argon Dewars

7. PROCEDURE

7.1 Preparation of Samples

7.1.1 Aqueous samples are prepped for analysis by using the SOP entitled ‘Preparation of Aqueous Samples for Testing by ICP or Flame AA’. This is LABENV42.6.

7.1.2 Soil/solid samples are prepped for analysis by using the SOP entitled ‘Preparation of Solid Samples for Testing by ICP or Flame AA’. This is LABENV43.5.

7.1.3 For AIHA samples (air, paint, wipes, soil for ELPAT) refer to LABIH009.5.

7.2 Calibration

7.2.1 Refer to Equipment SOP entitled 'Inductively Coupled Plasma – Atomic Emission Spectrometer' for instrument set-up.

7.2.2 The calibration consists of one calibration standard prepared in 4% HNO_3 and 5% HCl from the Calibration Stock Standards and a calibration blank. An example of the calibration level would be:

Element	Level (ppm)	Element	Level (ppm)	Element	Level (ppm)
Ag	0.40	Cr	4.0	Pb	4.0
Al	20	Cu	4.0	S	8.0
As	4.0	Fe	20	Sb	4.0
B	4.0	K	20	Se	4.0
Ba	4.0	Li	4.0	SiO2	8
Be	0.40	Mg	40	Sn	4.0
Ca	40	Mn	4.0	Ti	4.0
Cd	4.0	Na	40	Tl	4.0
Sr	4.0	Ni	4.0	V	4.0
Co	4.0	P	4.0	Zn	4.0

7.2.3 Calibration curve calculations are found in the QA Manual.

7.2.4 Reporting limit verification (RLV) is checked with each calibration curve by analyzing a separate standard at or below the reporting limit level. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit. The RLV (CRDL) for AIHA (ELLAP) samples must be ± 20%.

7.3 Analysis

7.3.1 Standards required for each method's analysis are outlined below.

7.3.2 Method EPA 6010B and AIHA (ELLAP)

7.3.2.1 Continuing Calibration Verification (CCV). This is a multi-element, mid-level standard in 4% HNO_3 and 5% HCl prepared from the Calibration Stock Standards. It is analyzed following the calibration, after every 10 samples, and at the end of the run. Elements of interest must recover within ± 10% of the true value. If not, samples bracketed by an out-of-spec CCV must be re-analyzed.

7.3.2.2 Continuing Calibration Blank (CCB). This is the Calibration Blank, a 4% HNO_3/5% HCl solution in DI water. It is analyzed following the calibration, after every 10 samples, and at the end of the run. For elements of interest, the absolute value of the result must be less than the absolute value of the reporting limit. If not, samples bracketed by an out-of-spec CCB must be re-analyzed. This is referred to as the ICB (initial calibration blank) when it is the first one after the calibration.

7.3.2.3 Initial Calibration Verification (ICV). This is a multi-element standard in 4% HNO_3 and 5% HCl prepared from the Second Source Stock Standards. It is analyzed following the calibration and must recover within ± 10% of the true value. If this fails, no data can be reported for analytical lines out of control.

7.3.2.4 Interference Check Standard A. This is called ICSA but referenced at LEGEND as IFA to be consistent with LIMS. This is a multi-element standard in 4% HNO_3 and 5% HCl prepared from the Fe, Al, Ca, and Mg single element stock standards. The IFA is used to verify the inter-element corrections (IEC) of the analytical method and consists of Fe at 300 mg/L, and Al, Ca, and Mg at 200 mg/L. It is analyzed after the calibration and before samples. The absolute value of the analytes of interest (other than Fe, Al, Ca & Mg) must be less than the value of the reporting limit. The IFA is also analyzed at the end of the run, when possible.

7.3.2.5 Iron (Fe) and Aluminum (Al) Spectral Interference Check (SIC) Solutions. These are single element standards (Fe at 300 mg/L and Al at 200 mg/L) in 4% HNO_3 and 5% HCl prepared from the Fe and Al single element stock standards. The Fe and Al SIC Solutions are used to verify and/or correct for interferences from Fe and Al. They are analyzed with every run, as Fe and Al are the most prevalent source of interferences on environmental samples. If an interference is present, IEC factors are re-calculated based on the response from these standards and the run re-processed to reflect these corrections.

7.3.2.6 Spectral Interference Check (SIC). These are single element standards analyzed annually to validate IEC factors for all corrections (not just Fe, Al, Ca, Mg). These are elements known to have potential spectral overlaps with analytes of interest. The following elements are measured: Fe, Al, Ba, Be, Cd, Ce, Co, Cr, Cu, Mn, Ni, Si, Sn, Ti, Tl, V, Zn, Pb, and Mo. If an interference is detected, IEC factors are re-calculated and the method is updated.

7.3.2.7 Linear Dynamic Range (LDR) Determination. The linear dynamic range must be determined annually or when a new method is developed. Standards are measured at successive levels with the criteria being that the standards recover within ± 10% of the true value. Samples with concentrations greater than 90% of the LDR are diluted and re-analyzed.

7.3.3 Method EPA 200.7

7.3.3.1 Instrument Performance Check (IPC): This is identical to the CCV above and is called a CCV at LEGEND. This is a multi-element, mid-level standard in 4% HNO_3 and 5% HCl prepared from the Calibration Stock Standards. It is analyzed following the calibration, after every 10 samples, and at the end of the run. The first IPC (CCV) must recover within ± 5% of the true value. If this fails for any of the elements of interest, the run must be stopped and the instrument recalibrated. Continuing IPC (CCV) determinations must recover within ±10% of true value. If this fails for any of the elements of interest, samples bracketed by an out-of spec IPC (CCV) must be re-analyzed following recalibration of the instrument.

7.3.3.2 Calibration Blank Check. This is identical to the CCB/ICB above. This is the Calibration Blank, a 4% HNO_3/5% HCl solution in DI water. It is analyzed following the calibration, after every 10 samples, and at the end of the run. For elements of interest, the absolute value of the result must be less than the reporting limit. If not, samples bracketed by an out-of-spec Calibration Blank must be re-analyzed.

7.3.3.3 Quality Control Sample (QCS). This is identical to the ICV above. This is a multi-element standard in 4% HNO_3 and 5% HCl prepared from the Second Source Stock Standards. It is analyzed following the calibration and must recover within $\pm$ 5% of the true value. If this fails for any of the elements of interest, the run must be stopped and the instrument re-calibrated.

7.3.3.4 Interference Check Standard A (IFA). This is a multi-element standard in 4% HNO_3 and 5% HCl prepared from the Fe, Al, Ca, and Mg single element stock standards. The IFA is used to verify the inter-element corrections (IEC) of the analytical method and consists of Fe at 300 mg/L, and Al, Ca, and Mg at 200 mg/L. It is analyzed after the calibration and before samples. The absolute value of the analytes of interest (other than Fe, Al, Ca & Mg) must be less than the value of the reporting limit. The IFA is also analyzed at the end of the run, when possible.

7.3.3.5 Iron (Fe) and Aluminum (Al) Spectral Interference Check (SIC) Solutions. These are single element standards (Fe at 300 mg/L and Al at 200 mg/L) in 4% HNO_3 and 5% HCl prepared from the Fe and Al single element stock standards. The Fe and Al SIC Solutions are used to verify and/or correct for interferences from Fe and Al. They are analyzed with every run, as Fe and Al are the most prevalent source of interferences on environmental samples. If an interference is present, IEC factors are re-calculated based on the response from these standards and the run re-processed to reflect these corrections.

7.3.3.6 Spectral Interference Check (SIC). These are single element standards analyzed annually to validate IEC factors for all corrections (not just Fe, Al, Ca, Mg). These are elements known to have potential spectral overlaps with analytes of interest. The following elements are measured: Fe, Al, Ba, Be, Cd, Ce, Co, Cr, Cu, Mn, Ni, Si, Sn, Ti, Tl, V, Zn, Pb, and Mo. If an interference is detected, IEC factors are re-calculated and the method is updated.

7.3.3.7 Linear Dynamic Range (LDR) Determination. The linear dynamic range must be determined annually or when a new method is developed. Standards are measured at successive levels with the criteria being that the standards recover within $\pm$ 10% of the true value. Samples are valid up to 90% of the determined LDR. For example, a 100 mg/L standard is analyzed and reads 95 mg/L. This would validate the LDR to 100 mg/L; however, samples can only be validated to 90 mg/L (90% of the LDR). Samples outside the LDR are diluted and re-analyzed.

7.3.4 Validate IEC corrections for Iron and Aluminum at the end of each instrument run. Reprocess run as necessary.

7.3.5 In the case of AIHA related work, samples with an in-solution concentration greater than the highest calibration level for an analyte of interest must be diluted such that the result falls below the calibration concentration if possible. If not, data must be flagged and qualified.

7.3.6 Create a LIMS sequence prior to analysis. The sequence consists of all standards and samples run on the instrument in order of analysis. Link source standards to the standards in the sequence so that the LIMS may calculate recoveries and provide traceability to stock standards.

7.3.7 Export 'in solution' data for the run and transfer into LIMS by sequence.

7.4 Calculation

7.4.1 The concentration of the analyte in the sample is calculated using the following equations:

$$Water\ Concentration\ (mg/L) = \frac{(C_{in})(FV)(D)}{V}$$

$$Soil\ Concentration\ (mg/kg) = \frac{(C_{in})(FV)(D)}{M}$$

C_{in} = in-solution concentration, mg/L
FV = final volume, mL
D = dilution factor
V = volume of sample, mL
M = mass of sample, g

7.5 Print a copy of the data from the LIMS for each project for internal review.

7.6 Print a copy of the raw data from the ICP including confirmation wavelengths and instrument conditions to be used for internal data review.

7.7 Sign and date the data sheets and the raw data checklist.

7.8 Submit data sheets and raw data to a qualified Chemist for peer review. Peer reviewer signs and dates the data sheets and the raw data checklist after checking data for integrity as well as compliance to protocols.

7.9 Turn data sheet into the Client Manager.

7.10 Archive paper copy of raw data, bench sheets, review checklist, sequence summary, and any other pertinent information, in a file folder organized by sequence number.

7.11 Electronic copies of the raw data are stored on the network.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Retain samples for two months after receipt date. Retain digestates for two months after the preparation date.

8.3 Samples and digestates are disposed of in the acid neutralizing laboratory sinks.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven digested replicate spikes, on an annual basis. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs values can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the absolute value of the reporting limit or the sample batch is redigested if possible. If it is not possible to redigest, the data will be flagged where appropriate. If batch cannot be re-digested and data is subsequently flagged be sure to fill out a Method Non-Conformance (MNC). Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits for laboratory control samples (LCS/LCSD) and matrix spikes (MS/MSD)

9.3.1 Accuracy control limits are set at the following:

9.3.1.1 EPA 200.7 LCS = 85.0-115%.

9.3.1.2 EPA 6010B LCS and NIOSH 7303(M) = 80.0-120%.

9.3.1.3 EPA 200.7 and 6010B MS = 75.0-125%.

9.3.2 Precision control limits are set at $\leq$ 20% RPD.

9.3.3 QC calculations are found in the QA Manual

9.3.4 LCS and MS are reviewed.

9.3.5 If the LCS data are outside the limits, the sample batch is re-digested if possible. If the batch cannot be re-digested, the data is flagged and a Method Non-Confromance (MNC) is filled out.

9.3.6 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the sequence and project files, and the data for that specific MS is flagged.

10. REPORTING

10.1 Solid sample results are reported in mg/kg on a dry weight basis.

10.2 Bulk sample results are reported in mg/kg on an as received basis.

10.3 Water sample results are reported in mg/L.

10.4 The reported result is rounded to two significant figures.

10.5 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A - Initial Demonstration of Capability

11.2 Appendix B - Method Detection Limits and Report Limits

12. REFERENCES

12.1 EPA 200.7 rev 4.4 (May 1994)

12.2 EPA 6010B

12.3 NIOSH 7303.

Appendix A

Initial Demonstration of Capability (IDC)
Axial ICP-AES

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four laboratory control samples of all the parameters and a method blank.

3. Analyze the control samples and method blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual recoveries in concentration and %, the mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: EPA 200.7 = 85.0-115%, EPA 6010B = 80.0-120%
   Precision: $\leq$ 20% RPD

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the QA/QC Coordinator signs the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

10. Note: refer to the AIHA Policy Guide, 2010, for specific instructions on IDCs for the ELPAT and IHPAT programs.

Appendix B

Method Detection Limits and Reporting Limits
Axial ICP-AES

Parameter	Water MDL (mg/L)	Water RL (mg/L)	Soil MDL (mg/kg)	Soil RL (mg/kg)
Aluminum	0.0018	0.020	0.090	1.0
Antimony	0.00078	0.010	0.039	0.50
Arsenic	0.0011	0.010	0.055	0.50
Barium	0.0012	0.020	0.060	1.0
Beryllium	0.000028	0.0050	0.0014	0.25
Boron	0.0083	0.10	0.42	5.0
Cadmium	0.00013	0.0010	0.033	0.25
Calcium	0.030	1.0	1.5	50
Chromium	0.00024	0.010	0.012	0.50
Cobalt	0.00033	0.0050	0.017	0.25
Copper	0.0011	0.020	0.055	1.0
Iron	0.0038	0.050	0.19	2.5
Lead	0.00042	0.0030	0.14	1.0
Magnesium	0.0021	1.0	0.11	50
Manganese	0.00078	0.020	0.011	1.0
Molybdenum	0.00070	0.050	0.035	2.5
Nickel	0.00036	0.0050	0.018	0.25
Phosphorous	0.0035	0.050	0.18	2.5
Potassium	0.0016	1.0	0.080	50
Selenium	0.0019	0.020	0.095	1.0
Silver	0.00031	0.0050	0.016	0.25
Sodium	0.0053	1.0	0.27	50
Thallium	0.00057	0.040	0.029	2.0
Tin	0.00059	0.020	0.030	1.0
Titanium	0.0015	0.020	0.075	1.0
Vanadium	0.00016	0.0050	0.0080	0.25
Zinc	0.0036	0.020	0.18	1.0

DOCUMENT REVIEW

DOCUMENT:	LABENV-039.6 SOP
REVIEWER:	William Dahl
DATE:	11/23/10

SECTION	CHANGE	RATIONALE
5.5	Changed logbook to raw data bath bench sheet	Current Practice
5.7	Changed documentation of acid addition to work order sample comments for lab preservation	Needed a consistent place to document this activity
5.7	Changed storage from 4 ± 2 °C to 4 ± 2 °C or ambient	Ambient is acceptable for some metals
6.10-6.13	Clarified standards information	Clarity
7.1	Added references to preparation SOPs	Clarity
7.2.2	Added Sr to table, changed Si to SiO2	Current Practice
7.2.4	Removed reference to monthly reporting limit check	Current Practice
7.3.2.7	Changed LDR to 90% LDR	Current Practice
various	Added absolute value when discussing specifications related to RL like blanks	Current Practice
various	Changed ICSA to IFA	Clarity
7.3.5	Clarified this applied only to AIHA	Current Practice
7.3.6	Indicated use of Sequence in LIMS	Current Practice
7.4.1	Changed ug/mL to mg/L	Clarity
7.6 and 7.10	Added instruction to print raw data and archive	Current Practice
8.2	Clarified waste disposal	Current Practice
8.2 and 9.3	Added instructions to fill out MNC	Current Practice
12.3	Removed reference to Vista Operating Manual and added refernce to NIOSH 7303	Vista Manual N/A; NIOSH applicable
Appendix A	Made reference to AIHA Policy Guide for IDCs	Current Practice
Appendix B	Updated MDLs	Current MDLs

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF NITROAROMATICS AND NITROAMINES IN WATER BY UPLC

SOP NO.: LABENV-040.7

Original Information		
Prepared by:	Kimberly Dublin	Date: 08/14/00
Technical Review:		Date:
QA/QC Coordinator:	Sharon Dahl	Date: 08/14/00
Authorized by:	Cheryl Sykora	Date: 09/28/00

Revision Information		
Supersedes:	LABENV-040.6	Date: 05/27/09
Revised by:	Scott Creekmur	Date: 05/03/11
Signature:	_____	Date: _____
Technical Review:	Tom Barrett	Date: 05/03/11
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 05/03/11
Signature:	_____	Date: _____

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Prepared by:	Kimberly Dublin	Date: 08/14/00
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Revision Information		
Supersedes:	LABENV-040.6	Date: 05/27/09
Revised by:	Scott Creekmur	Date:
Signature:		Date: 5-3-11
Technical Review:	Tom Barrett	Date:
Signature:		Date: 5/3/11
Authorized by:	Cheryl Sykora	Date:
Signature:		Date: 5/3/11

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SOP TITLE: DETERMINATION OF NITROAROMATICS AND NITROAMINES IN WATER BY UPLC

1. PURPOSE

1.1 This document defines the procedure to be followed for the determination of nitroaromatics and nitroamines in waters by Ultra High Performance Liquid Chromatograph (UPLC) using a photo diode array (PDA) detector. The SOP is applicable to samples typically analyzed by a modified EPA Method 8330.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 An analyst trained by LEGEND Technical Services, Inc. shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 The procedure is limited to water samples.

3.2 Degradation products of tetryl appear as a shoulder on the 2,4,6-Trinitrotoluene peak. Peak heights, rather than peak areas, should be used when tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-Trinitrotoluene.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 When working with organic compounds, wear solvent resistant gloves.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in 1L amber bottles with Teflon lined caps and stored at $\leq$ 6 °C but not freezing. Samples must be protected from light.

5.4 The recommended holding time for water samples is 7 days until extraction, and analysis within 40 days of extraction.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 UPLC with photodiode array detector (PDA)

6.2 Waters Acquity UPLC BEH C18 column, 100 mm x 2.1 mm x 1.7 µm, or equivalent

6.3 Vacuum pump

6.4 Solid phase manifold

6.5 3M Empore discs SBD-RPS catalog #2241, or equivalent

6.6 Erlenmeyer flasks

6.7 Buchner funnel

6.8 Filter paper – Whatman #42 or equivalent

6.9 Waste collection container

6.10 Amber serum vials, 10 mL with PTFE septa

6.11 Amber autosampler vials

6.12 Acrodisc (0.45 µm) – PTFE, or equivalent

6.13 Graduated cylinder – 500 mL

6.14 25 µL, 50 µL, 250 µL, 500 µL and 1.0 mL syringes

6.15 5 mL Graduated disposable pipettes equivalent

6.16 Deionized (DI) Water (>16.3 MΩ)

6.17 Acetonitrile (ACN), HPLC grade

6.18 Methanol (MeOH), HPLC grade

6.19 Acetone, HPLC Grade

6.20 Isopropyl Alcohol (IPA), HPLC Grade

6.21 8330 Calibration/Spike Stock – 1,000 µg/mL EPA 8330 Kit, Ultra #NAIM-833E, or equivalent

6.22 Surrogate Stock – 1,000 µg/mL 1,2-dinitrobenzene, Ultra #IST-600, or equivalent

6.23 Second Source 8330 Stock – 1,000 µg/mL, different vendor or different lot number than stock used for calibration

6.24 Second Source PETN Stock – 1,000 µg/mL, different vendor or different lot number than stock used for calibration

6.25 Second Source Surrogate Stock – 1,000 µg/mL, different vendor or different lot number than stock used for calibration

6.26 8330 Continuing Calibration (CCAL) Solution – dilute 125 µL each of the 1,000 µg/mL Second Source 8330 Stock and 1,000 µg/mL Second Source Surrogate Stock to 50 mL with acetonitrile to produce a 2.5 µg/mL 8330 Continuing Calibration (CCAL) Solution

6.27 8330 Spike Working Solution – dilute 0.5 mL each of the 1,000 µg/mL 8330 Calibration/Spike Stock to 25 mL with acetonitrile to produce a 20 µg/mL 8330 Spike Working Solution

6.28 Surrogate Working Solution – dilute 0.5 mL of the 1,000 µg/mL Surrogate Stock to 25 mL with acetonitrile to produce a 20 µg/mL Surrogate Working Solution

7. PROCEDURE

7.1 Preparation of Samples

7.1.1 Set up filtering apparatus using vacuum pump, Buchner funnel, and Whatman #42 filter paper.

7.1.2 Measure out 500 mL of sample using a graduated cylinder and filter. Repeat with all samples. In each analytical batch choose a sample and measure out two additional aliquots – one for the Matrix Spike (MS) and one for the Matrix Spike Duplicate (MSD).

7.1.3 Measure out 500 mL of reagent water using a graduated cylinder for the method blank and Laboratory Control Spike (LCS).

7.1.4 Add 0.5 mL of the 20 µg/mL Surrogate Working Solution to each sample filtrate, blank, LCS, and spike sample filtrate. The final concentration will be 20 µg/L.

7.1.5 Add 0.5 mL of the 20 µg/mL 8330 Spike Working Solution to the filtrate of the samples in each analytical batch selected for spiking. A typical batch will have an LCS and MS/MSD (an LCS/LCSD will be substituted if insufficient sample is provided). The final concentration will be 20 µg/L.

7.1.6 Set up the solid phase manifold with one 3M Empore disc per sample.

7.1.7 With the vacuum on, rinse the disc with 10 mL of acetone and allow to dry. Then rinse with 10 mL of IPA and allow to dry.

7.1.8 Add 15 mL MeOH to the disc. Let approximately 5 mL flow through and remove the vacuum. Let stand for approximately 1 minute. Filter remaining MeOH through disc until almost dry (5 mL left). DO NOT LET EMPORE DISC GO DRY.

7.1.9 Add 15 mL of DI water to the disc and draw approximately 5 mL through and remove the vacuum. Let stand for 1 minute. DO NOT LET EMPORE DISC GO DRY.

7.1.10 Pour sample onto Empore disc, apply vacuum, and filter until dry.

7.1.11 Place a 10 mL amber vial in manifold column under an Empore disc. Add 5 mL ACN and let stand for approximately 1 minute. Filter ACN into 10 mL vial.

7.1.12 Bring up to 5 mL final volume in ACN using a graduated 5 mL pipette.

7.1.13 Retain the remainder of the extract in a Teflon capped vial.

7.2 Calibration

7.2.1 Prepare working standards at a minimum of five concentration levels, ranging from 0.25 – 7.5 µg/mL, by diluting the 1,000 ppm 8330 Calibration/Spike Stock and the 1,000 ppm Surrogate Stock with acetonitrile. A typical calibration curve would be:

Level	From Level 5 µL/10 mL	Calibration Stocks µL/10 mL	Final Concentration µg/mL
L1	500	-----	0.25
L2	1000	-----	0.50
L3	-----	10	1.0
L4	-----	25	2.5
L5	-----	50	5.0
L6	-----	75	7.5

7.2.2 The average response factor should be calculated for each analyte. The percent relative standard deviation (%RSD) should be less than 20% for each analyte. If the RSD for any analyte is greater than 20%, review the results (area counts, response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the calibration standards.

7.2.3 If the problem appears to be associated with a single standard, reprep and/or reanalyze that standard and calculate the RSD again.

7.2.4 If the %RSD is still greater than 20%, construct calibration curves of area versus concentration using a first order or linear fit. Correlation coefficients should be 0.990 or greater.

7.2.5 Calibration curve calculations are found in the QA Manual.

7.2.6 Reporting limit verification (RLV) is checked with each calibration curve by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or flagging data.

7.2.7 All extracts should be stored in the refrigerator in the dark.

7.3 Analysis

7.3.1 HPLC Conditions

7.3.1.1 Waters Acquity UPLC BEH C18 column, 100 mm x 2.1 mm x 1.7 µm, or equivalent

7.3.1.2 HPLC Mobile Phase 69:31 H_20:MeOH

7.3.1.3 Column Temperature 50 °C

7.3.1.4 Wavelength 254 nm

7.3.1.5 Flow Rate 0.5 mL/min, Isocratic run for 7.25 minutes

7.3.1.6 Injection volume: 1 µL

7.3.2 Sample peak identification will be confirmed by peak retention time and peak spectrum as compared to standards.

7.3.3 If the response for a peak exceeds the working range of the system or the highest standard, dilute the extract with acetonitrile and re-analyze.

7.3.4 The CCAL is analyzed at the beginning of the run in triplicate (if an initial calibration curve was not analyzed), after every ten samples (singly), and at the end of the run (singly). Recoveries for the triplicates should be ± 15% of the initial curve or corrective action should be taken. Recoveries for the continuing and end CCAL should be ± 15% of the triplicate average.

7.3.5 Corrective action may include reanalyzing the CCAL and/or flagging the data in the daily file.

7.4 Calculation

7.4.1 Computer software calculates the concentration of the sample based on the response.

$$Explosives\ (\mu g/L) = \frac{(CC)(FV)}{V}$$

CC = concentration on column (µg/mL)
FV = final volume (mL)
V = volume of sample (L)

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, when there is a change in the test method that may affect how the test is performed or when there is a major change in instrumentation. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDLs and RLs can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the reporting limit or the sample batch is re-analyzed, if possible. If it is not possible to re-analyze, the data will be flagged where appropriate. Do not subtract analytes in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Accuracy control limits are set at 70.0-130% for LCS, MS and surrogates.

9.3.2 Precision control limits are set at 20.0% RPD (relative percent difference) for LCS/LCSD and MS/MSD.

9.3.3 QC calculations are found in the QA Manual.

9.3.4 LCS, MS and surrogates are reviewed.

9.3.5 If the LCS data are outside the limits, the sample batch is re-extracted and/or re-analyzed, if possible. If the batch cannot be re-extracted and/or re-analyzed, the information is placed in the daily and project files, and a case narrative is written for all client reports within the batch.

9.3.6 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS may be flagged in the case narrative of the report.

9.3.7 If a sample surrogate is outside the limits, the sample is re-extracted and/or reanalyzed, if possible. If the sample cannot be re-extracted and/or reanalyzed, the data is flagged next to the actual result in the report.

10. REPORTING

10.1 Samples results are reported in µg/L.

10.2 The reported result is rounded to two significant figures.

10.3 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 EPA Method 8000B

12.2 EPA Method 8330

Appendix A

Initial Demonstration of Capability (IDC)
Determination of Nitroaromatics and Nitroamines

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards in lab-grade water and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: LCS limits (70.0-130%)
   Precision: LCS limits (≤ 20% RPD)

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Nitroaromatics and Nitroamines – EPA Method 8330(M)

Parameter	Water MDL (µg/L)	Water RL (µg/L)
1,3,5-Trinitrobenzene	0.19	4.0
1,3-Dinitrobenzene	0.38	4.0
2,4,6-Trinitrotoluene	0.39	4.0
2,4-Dinitrotoluene	0.34	4.0
2,6-Dinitrotoluene	0.57	4.0
2-Amino-4,6-dinitrotoluene	0.43	4.0
2-Nitrotoluene	0.81	4.0
3-Nitrotoluene	0.61	4.0
4-Amino-2,6-dinitrotoluene	0.51	4.0
4-Nitrotoluene	1.1	4.0
HMX	0.38	4.0
Nitrobenzene	0.62	4.0
RDX	0.48	4.0
Tetryl	0.62	4.0

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-040.7
REVIEWER:	Scott Creekmur
DATE:	05/03/11

SECTION	CHANGE	RATIONALE
All	Major revision	New Instrumentation

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR TESTING BY ICP OR FLAME AA

SOP NO.: LABENV-042.6

Original Information		
Prepared by:	William R. Dahl	Date: 01/30/02
Technical Review:	Brian Leigh	Date: 02/26/02
QA/QC Coordinator:	Terri Olson	Date: 02/26/02
Authorized by:	Cheryl Sykora	Date: 02/26/02

Revision Information		
Supersedes:	LABENV-042.5	Date: 04/07/08
Revised by:	William Dahl	Date:
Signature:	_____	Date: _____
Technical Review:	Dan Brezina	Date:
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date:
Signature:	_____	Date: _____

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Prepared by:	William R. Dahl	Date: 01/30/02
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Authorized by:	Cheryl Sykora	Date: 02/26/02

Revision Information		
Supersedes:	LABENV-042.5	Date: 04/07/08
Revised by:	William Dahl	Date:
Signature:		Date: 11/23/10
Technical Review:	Dan Brezina	Date:
Signature:		Date: 11-23-10
Authorized by:	Cheryl Sykora	Date:
Signature:		Date: 11/23/10

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SOP TITLE: PREPARATION OF AQUEOUS SAMPLES FOR TESTING BY ICP OR FLAME AA

1. PURPOSE

1.1 This document defines the preparation of samples prior to analysis by ICP or Flame AA. It applies to all aqueous matrices and to terminology referring to "liquids" and "TCLP extracts/leachates." The SOP is applicable to samples typically prepared by EPA Methods 200.7 and EPA 3005A.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the SOP.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is appropriate to aqueous liquids with a density around 1 g/mL. If a sample does not fit this criterion it must be prepared using a solid preparation method and reported on a weight basis.

3.2 If the laboratory will be filtering the sample for dissolved metals analyses, do not perform nitric acid preservation.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 Safety glasses and gloves should be worn when handling samples and reagents.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in polyethylene or glass containers and preserved with 1:1 nitric acid to a pH < 2.

5.4 The recommended holding time for water samples is six months.

5.5 If a sample is received with pH > 2 then it is preserved in the laboratory and allowed to sit for a minimum of 24 hours at which time the pH is re-tested. If pH < 2, proceed. If not, repeat until pH is < 2, if possible. Highly alkaline samples may need to be digested at reduced volume with excess acid, rather than dilute the original sample in an attempt to lower the pH.

5.6 Document the final pH of all samples on the raw bench sheet. For samples requiring preservation at the laboratory, document the addition of acid and the times in the LIMS under sample comments.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Block digester

6.2 Screw cap digestion vessels

6.3 Plastic watch glasses

6.4 Nitric acid (HNO_3), Concentrated - Trace Metals Grade

6.5 Hydrochloric Acid (HCl), Concentrated - Trace Metals Grade

6.6 Bottle top dispensers for acids above

6.7 Plunge filters

6.8 Deionized (DI) water (>16.3 MΩ)

7. PROCEDURE

7.1 Batch samples in LIMS including QC (BLK, LCS, LCSD, MS. MSD). Print out raw data bench sheet.

7.2 Be sure to indicate source sample as well as standards used for spiking and reagent trace numbers on the bench sheet..

7.3 TCLP samples are batched separately from other aqueous samples.

7.4 Label digestion vessels with sample IDs. Label digestion vessels for the Blank, LCS, and LCSD including the batch number. Along with the sample ID, label MS for sample matrix spike and MSD for sample matrix spike duplicate. MS is also referred to as the Laboratory Fortified Matrix (LFM).

7.5 Shake all samples prior to transferring into digestion vessels.

7.6 Measure out 50 mL of DI water for each of the Blank, LCS, and LCSD using graduations on the digestion vessels. If the batch is TCLP samples, measure out 10 mL of the TCLP leachate blank for each of the Blank, LCS and LCSD and dilute to 50 mL with DI water.

7.7 Measure out 50 mL of each sample into its assigned digestion vessel. Measure directly into digestion vessels using the graduations on the side of the vessels. Graduation marks are certified for volume. Records are kept in the QA/QC department. If the batch is TCLP

samples, measure out 10 mL of each TCLP sample into the appropriate digestion vessel and dilute to 50 mL with DI water.

7.8 For one of the water samples, measure out two additional aliquots, one for the MS and one for the MSD.

7.9 Alternate volumes may be used if sample is limited or a dilution is required.

7.10 Record the initial volumes of all samples in the raw data bench sheet printout.

7.11 Measure out up to 20 samples per batch.

7.12 Transfer the samples to a rack. Order the samples in the rack from left to right on the long row as they appear in the raw data bench sheet with the QC in the front. The labels for the LCS, LCSD, MS and MSD are seen in the first row.

7.13 A typical spike list is given in the table below. Add 0.050 mL for each of the three groups listed below to the LCS, LCSD, MS, and MSD (final volume is 50 mL). For a batch of TCLP samples, spike 0.10 mL of each of the three groups listed below. Final concentrations will be double those listed. Record all information in the raw data bench sheet.

Parameter	**Stock Concentration (ppm)**	**Final Concentration (ppm)**
Aluminum	2000	2.0
Barium	400	0.40
Calcium	4000	4.0
Cobalt	400	0.40
Iron	2000	2.0
Magnesium	4000	4.0
Manganese	400	0.40
Potassium	2000	2.0
Sodium	4000	4.0
Vanadium	400	0.40
Zinc	400	0.40
Arsenic	400	0.40
Beryllium	40	0.040
Cadmium	400	0.40
Chromium	400	0.40
Copper	400	0.40
Lead	400	0.40
Nickel	400	0.40
Selenium	400	0.40
Silver	40	0.040
Thallium	400	0.40
Antimony	400	0.40
Boron	400	0.40
Molybdenum	400	0.40
Tin	400	0.40

7.14 Add 2 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl to all the samples (including

Blank, LCS, & LCSD).

7.15 Ensure the block digester temperature meter reads 120. (Note: This is the setting of the block coil; actual sample temperature is approximately 95-97 °C).

7.16 Transfer racks of samples to the block digester and place plastic watch glasses over each digestion vessel. Record the time in the raw data bench sheet.

7.17 Let samples heat for 2 to 3 hours, ensuring sufficient volume loss.

7.18 Remove samples from block digester and record the time in the raw data bench sheet. Allow samples to cool.

7.19 Bring all samples up to 50 mL with DI water using the graduations on the digestion vessels.

7.20 Record the final volumes in the raw data bench sheet.

7.21 If sediment is present, filter only those samples using the plunge filters. It is imperative that you also filter the QC at this point, including the Blank, LCS, and LCSD. If samples are not being filtered, the QC does not need to be filtered.

7.22 Cap all samples.

7.23 Place samples in foam batch holders in the order that they are listed in the raw data bench sheet with QC at the beginning in order of BLK, LCS, LCSD, MS, and MSD.

7.24 Update LIMS with all of the new information recorded in the raw data bench sheet and print out a copy of the updated bench sheet stapling it to the raw data bench sheet. Both are retained in the sequence data packet.

7.25 Calibration, analysis and calculation are not applicable to this SOP but are addressed in the appropriate analytical SOP.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures. Refer to analytical SOP for schedule of disposal.

9. QA/QC

9.1 Follow the QA/QC protocol outlined in the appropriate analytical SOP.

10. REPORTING

Not applicable

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

12. REFERENCES

12.1 EPA Method 200.7

12.2 EPA Methods 3005A and 6010B

Appendix A

Initial Demonstration of Capability (IDC)
Preparation of Aqueous Samples for ICP/Flame AA

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four laboratory control samples of all the parameters in lab-grade water and a method blank.

3. Analyze the laboratory control samples and method blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: 200.7 = 85.0-115%, 6010B– 80.0-120% (200.7/6010B may be combined if tighter limits are used for acceptance)
   Precision: 200.7/6010B = $\leq$ 20 % RPD

7. The method blank must be less than the absolute value of the reporting limit (RL).

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

DOCUMENT REVIEW

DOCUMENT:	LABENV-042.6
REVIEWER:	William Dahl
DATE:	11/22/10

SECTION	CHANGE	RATIONALE
Various	Replaced digestion logbook with raw batch bench sheet	Current practice
5.6	Added documentation of lab preservation in LIMS	Formerly no good way to document
6.4 – 6.5	Changed from 1:1 acid to concentrated acid	Results in same final acid concentration
6.6	Added bottle top dispensers	Clarity
7.14	Changed from 4 mL and 5 mL 1:1 to 2 and 2.5 mL concentrated acids	Results in same final acid concentration and works better logistically with the tubes
7.15	Removed reference to Centigrade	Do not want a misunderstanding that this is a temperature
7.24	Added reference to update and printing of final bench sheet	Current Practice
8.1	Added “refer to analytical SOP for disposal schedule”	Current Practice
Appendix A	Changed reagent blank and mid level standard to method blank and lab control samples	Current Practice
Appendix A	Added absolute value when referring to blank specification	Clarity

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: PREPARATION OF SOLID SAMPLES FOR TESTING BY ICP OR FLAME AA

SOP NO.: LABENV-043.5

Original Information		
Prepared by:	William R. Dahl	Date: 01/30/02
Technical Review:	Brian Leigh	Date: 02/26/02
QA/QC Coordinator:	Terri A. Olson	Date: 02/26/02
Authorized by:	Cheryl Sykora	Date: 02/26/02

Revision Information		
Supersedes:	LABENV-043.4	Date: 04/07/08
Revised by:	William Dahl	Date: 11/23/10
Signature:	_____	Date: _____
Technical Review:	Dan Brezina	Date: 11/23/10
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 11/23/10
Signature:	_____	Date: _____

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LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: PREPARATION OF SOLID SAMPLES FOR TESTING BY ICP OR FLAME AA

SOP NO.: LABENV-043.5

Original Information		
Prepared by:	William R. Dahl	Date: 01/30/02
Technical Review:	Brian Leigh	Date: 02/26/02
QA/QC Coordinator:	Terri A. Olson	Date: 02/26/02
Authorized by:	Cheryl Sykora	Date: 02/26/02

Revision Information		
Supersedes:	LABENV-043.4	Date: 04/07/08
Revised by:	William Dahl	Date:
Signature:		Date: 11/23/10
Technical Review:	Dan Brezina	Date:
Signature:		Date: 11-23-10
Authorized by:	Cheryl Sykora	Date:
Signature:	Cheryl Sykora	Date: 11/23/10

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SOP TITLE: PREPARATION OF SOLID SAMPLES FOR TESTING BY ICP OR FLAME AA

1. PURPOSE

1.1 This document defines the preparation of samples prior to analysis by ICP and/or Flame AA. The SOP is applicable to samples typically analyzed prepared by EPA Methods 3050B.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is applicable to all soil matrices, and will also apply to terminology referring to solids and sludges.

3.2 There are many materials that require specific method development to achieve desired objectives. These are handled on a case-by-case basis and may not be covered under this procedure.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 Safety glasses and gloves should be worn when handling samples and reagents.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Solid samples should be collected in polyethylene or glass containers and stored at 4 ± 2 ºC or at ambient temperature depending on the metals requested. Samples are initially stored at 4 ± 2 ºC and then moved to ambient if appropriate.

5.4 The recommended holding time for soil samples is six months.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Block digester

6.2 Screw cap digestion vessels

6.3 Plastic watch glasses

6.4 Nitric acid (HNO_3) 1:1 (trace metal grade:DI)

6.5 Hydrochloric Acid (HCl) 1:1 (trace metal grade:DI)

6.6 Plunge filters

6.7 Deionized (DI) water (>16.3 MΩ)

6.8 Analytical Balance capable of reading 0.0001 g

7. PROCEDURE

7.1 Batch samples in LIMS including QC (BLK, LCS, LCSD, MS. MSD). Print out raw data bench sheet.

7.2 Be sure to indicate source sample as well as standards used for spiking and reagent trace numbers..

7.3 Label digestion vessels with sample IDs. Label digestion vessels for the Blank, LCS, and LCSD including the batch number. Along with the sample ID, label MS for sample matrix spike and MSD for sample matrix spike duplicate. MS is also referred to as the Laboratory Fortified Matrix (LFM).

7.4 Weigh out between 1.00 and 1.04 g aliquot of soil and transfer to a pre-labeled digestion vessel. It may occasionally be necessary to alter the weight of a sample due to limited availability of sample or the need to achieve lower detection levels. For one of the solid samples, weigh out two additional aliquots, one for the MS and one for the MSD.

7.5 Record the weights of all samples in the raw data bench sheet.

7.6 Weigh out up to 20 samples per batch.

7.7 Place the samples in a rack. Arrange them from left to right, on the long row, as they appear in the raw data bench sheet, thus placing the LCS, LCSD, MS and MSD in the first row for easy spiking.

7.8 A typical spike list is given in the table below. Add 0.10 mL for each of the three groups listed below to the LCS, LCSD, MS, and MSD (final volume is 50 mL). Record all information in the Digestion Log Book and LIMS.

Parameter	Stock Concentration (ppm)	Final Concentration (ppm)
Aluminum	2000	4.0
Barium	400	0.80
Calcium	4000	8.0
Cobalt	400	0.80
Iron	2000	4.0
Magnesium	4000	8.0
Manganese	400	0.80
Potassium	2000	4.0
Sodium	4000	8.0
Vanadium	400	0.80
Zinc	400	0.80

Arsenic	400	0.80
Beryllium	40	0.080
Cadmium	400	0.80
Chromium	400	0.80
Copper	400	0.80
Lead	400	0.80
Nickel	400	0.80
Selenium	400	0.80
Silver	40	0.080
Thallium	400	0.80

Antimony	400	0.80
Boron	400	0.80
Molybdenum	400	0.80
Tin	400	0.80

7.9 Add 4 mL of 1:1 HNO_3 and 5 mL of 1:1 HCl to all samples (including Blank, LCS, & LCSD).

7.10 Ensure the block digester temperature meter reads 120. (Note: This is the setting of the block coil, actual sample temperature is approximately 95-97 ºC).

7.11 Transfer racks of samples to the block digester and place plastic watch glasses over each digestion vessel. Record the time in the Raw data bench sheet.

7.12 Let samples heat for 1 – 2 hours

7.13 Remove racks from the block digester and record the time in the raw data bench sheet.

7.14 Bring all samples up to 50 mL with DI water using the graduations on the digestion vessels.

7.15 Record the final volumes in the raw data bench sheet.

7.16 Samples may be allowed to settle, may be centrifuged, or may be filtered with the plunge filters. If samples are filtered then filter ALL samples (including Blank, LCS, and LCSD) using the plunge filters. Be careful when applying pressure to avoid bursting one of the filters. (Reminder: Be sure that the filter side is toward the bottom. Discard handle after use).

7.17 Cap all samples.

7.18 Place samples in foam batch holders, in the order that they are listed in the raw data bench sheet.

7.19 Calibration, analysis and calculation are not applicable to this SOP but are addressed in the appropriate analytical SOP.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures. Refer to analytical SOP for schedule of disposal.

9. QA/QC

9.1 Follow the QA/QC protocol outlined in the appropriate analytical SOP.

10. REPORTING

10.1 Not applicable

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

12. REFERENCES

12.1 EPA Methods 3050B and 6010B

Appendix A

Initial Demonstration of Capability (IDC)
Preparation of Solid Samples for ICP/Flame AA

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four laboratory control samples and a method blank.

3. Analyze the laboratory control samples and method blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: 80.0-120%
   Precision: $\leq$ 20 % RPD

7. The method blank must be less than the absolute value of the reporting limit (RL).

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

DOCUMENT REVIEW

DOCUMENT:	LABENV-43.5
REVIEWER:	William Dahl
DATE:	11/23/10

SECTION	CHANGE	RATIONALE
5.3	Changed from 4 +/- 2 to 4 +/- 2 or ambient	Some metals are fine stored ambient
6.8	Changed from top loader to analytical balance	Current Practice
7.1 and 7.2	Changed to create LIMS batch and print bench sheet from use of logbook	Current Practice
various	Replaced reference to digestion logbook with raw data bench sheet	Current Practices
7.10	Changed from 125 to 120; removed reference to centigrade and temperature	Clarity, the value is to be used as a setting only and not mis-understood to be a temperature value
7.12	Changed to 1-2 hours	It may be suitable for some samples to cook longer than 1 hour
7.16	Added options for settling particulates	Acceptable flexibility in practices
7.18	Spelled out expectations	Clarity
8.1	Added reference to analytical SOP for disposal	Current Practices
Appendix A	Changed mid level replicates to laboratory control samples and reagent blank to method blank	Current Practices
Appendix A	Changed criteria on method blank to less than absolute value of the reporting limit	Clarity

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**DETERMINATION OF ETHYLENE AND PROPYLENE GLYCOL**
SOP NO.:	**LABENV-047.6**

Original Information		
Prepared by:	Brian Krengel	Date: 04/03/01
Technical Review:		Date:
QA/QC Coordinator:	Sharon Dahl	Date: 04/05/01
Authorized by:	Cheryl Sykora	Date: 10/15/01

Revision Information		
Supersedes:	LABENV-047.5	Date: 12/10/10
Revised by:	Triet Le	Date: 5/03/11
Signature:	_____	Date: _____
Technical Review:	Van Pham	Date: 5/03/11
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 5/03/11
Signature:	_____	Date: _____

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LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**DETERMINATION OF ETHYLENE AND PROPYLENE GLYCOL**
SOP NO.:	**LABENV-047.6**

Original Information		
Prepared by:	Brian Krengel	Date: 04/03/01
Technical Review:		Date:
QA/QC Coordinator:	Sharon Dahl	Date: 04/05/01
Authorized by:	Cheryl Sykora	Date: 10/15/01

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Revised by:	Triet Le	Date:
Signature:	T. Le	Date: 05/03/11
Technical Review:	Van Pham	Date:
Signature:	Van Pham	Date: 5/3/11
Authorized by:	Cheryl Sykora	Date:
Signature:	Cheryl Sykora	Date: 5/3/11

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SOP TITLE: DETERMINATION OF ETHYLENE AND PROPYLENE GLYCOL

1. PURPOSE

1.1 This document defines the procedure used to determine ethylene and propylene glycol in water and soil samples by gas chromatography (GC) using a flame ionization detector (FID). The SOP is applicable to samples typically analyzed by a modified EPA Method 8015B and D.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This method is applicable to soil and aqueous samples.

3.2 The presence of hydrocarbons may give false results.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 A lab coat should be worn.

4.4 When working with organic compounds, wear solvent resistant gloves and safety glasses.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in duplicate, with no headspace, in 40 mL unpreserved VOA vials and stored at $\leq$ 6 °C but not freezing.

5.4 The recommended holding time for water samples is 14 days until analysis.

5.5 Soil samples should be collected with no headspace, in a glass jar with a Teflon lined cover, or equivalent, and stored at ≤ 6 °C but not freezing.

5.6 The recommended holding time for soil samples is 14 days until extraction and analysis within 40 days of extraction.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 GC equipped with a flame ionization detector (FID) and a data processing system

6.2 GC column - DB®-Wax: 30 m x 0.53 mm x 1.0 µm film, or equivalent

6.3 GC liner - Restek's Drilled Uniliner® Cat. # 21055, or equivalent

6.4 Wide mouth jars with Teflon lined caps, 4 oz or equivalent

6.5 VOA vials with Teflon lined caps, 40 mL or equivalent

6.6 Assorted volumetric flasks

6.7 Assorted microliter syringes

6.8 2 mL autosampler vials

6.9 Balance, capable of reading to 0.01 g

6.10 Disposable pipets

6.11 Stainless steel spatula

6.12 Deionized (DI) water (>16.3 MΩ)

6.13 Anhydrous Sodium Sulfate (Na_2SO_4) - muffle at 400 °C for four hours before using

6.14 Ottawa Sand (20-30 mesh) or equivalent – muffle at 400 °C for four hours before using

6.15 Acetone – HPLC grade, or equivalent

6.16 Methanol – pesticide grade, or equivalent

6.17 5% Methanol – dilute 5 mL of methanol with 95 mL of organic free water

6.18 Ethylene Glycol Stock - 99.8%, density 1.113 (Sigma-Aldrich Cat. #32,455-8 or equivalent)

6.19 Propylene Glycol Stock - 99.5%, density 1.036 (Sigma-Aldrich Cat. #39,803-9 or equivalent)

6.20 Intermediate Calibration Solution 1 - dilute 100 µL of each glycol stock to 10 mL with organic free water (aqueous samples) or acetone (soil samples) to produce an 11,130 µg/mL ethylene glycol and 10,360 µg/mL propylene glycol Intermediate Calibration Solution 1. Store soil standard in a freezer. Store water standard in the refrigerator.

6.21 Intermediate Calibration Solution 2 - dilute 100 µL of Intermediate Calibration Solution 1 to 2 mL with water (aqueous samples) or acetone (soil samples) to produce a 556.5 µg/mL ethylene glycol and 518 µg/mL propylene glycol Intermediate Calibration Solution 2. Store soil standard in a freezer. Store water standard in the refrigerator.

6.22 Soil Spike Standard - dilute 100 µL of each glycol stock to 10 mL with acetone to produce an 11,130 µg/mL ethylene glycol and 10,360 µg/mL propylene glycol Soil Spike Standard.

6.23 Water Spike Standard – dilute 20 µL of each glycol stock to 10 mL with organic free water to produce a 2,226 µg/mL ethylene glycol and 2,072 µg/mL propylene glycol Water Spike Standard

7. PROCEDURE

7.1 Preparation of Soil Samples

7.1.1 Prepare a method blank by weighing out 5 grams of Ottawa sand into a 40 mL labeled VOA vial.

7.1.2 Weigh 5 grams of each sample into 40 mL labeled VOA vials.

7.1.3 Prepare the laboratory control spike (LCS) by adding 5 grams of Ottawa sand to a 40 mL labeled VOA vial.

7.1.4 Prepare the MS by adding 5 grams of the sample to be spiked to a 40 mL labeled VOA vial; repeat for the MSD.

7.1.5 Add approximately 2-3 grams of sodium sulfate to every sample; mix well.

7.1.6 For the samples in each analytical batch selected for spiking, add 15 µL of the 11,130/10,360 µg/mL Soil Spike Standard to produce a 33.39/31.08 µg/mL on column concentration (based on a 5.0 mL final volume). A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if enough sample is not provided.

7.1.7 Add 5.0 mL of acetone and shake briefly. If the solvent doesn't completely cover the sample, prepare the sample again and add less sodium sulfate.

7.1.8 Sonicate the samples for at least 5 minutes. Let the sample settle.

7.1.9 Carefully pipet the solvent layer into labeled 2 mL autosampler vials. Store the samples in the freezer until analysis.

7.2 Preparation of Water Samples

7.2.1 Prepare a water (method) blank by pipetting approximately 1 mL of 5% methanol into an autosampler vial.

7.2.2 Transfer approximately 1 mL of each sample into a 2 mL autosampler vial

7.2.3 Prepare the LCS by adding 1.0 mL of 5% methanol into an autosampler vial.

7.2.4 Prepare the MS by adding 1.0 mL of the sample to be spiked into an autosampler vial; repeat for the MSD.

7.2.5 For the samples in each analytical batch selected for spiking, add 15 µL of the 2,226/2,072 µg/mL Water Spike Standard to produce a 33.39/31.08 µg/mL on column concentration (based on a 1.0 mL final volume). A typical batch will have an LCS and MS/MSD. LCS/LCSD will be substituted if the sample is suspected to contain high concentration of ethylene/propylene glycol.

7.3 Calibration

7.3.1 Prepare standards at a minimum of 5 concentration levels, ranging from approximately 7.0-60 µg/mL, by diluting the 556.5/518 µg/mL Intermediate Calibration Solution 2 with 5% methanol (aqueous samples) or acetone (soil samples). A typical calibration curve would be:

Inter. Calib. Solution 2 (µL/mL)	Ethylene Glycol Conc. (µg/mL)	Propylene Glycol Conc. (µg/mL)
15	8.35	7.77
25	13.91	12.95
50	27.83	25.90
75	41.74	38.85
100	55.65	51.80

7.3.2 The average response factor should be calculated for each compound. The percent relative standard deviation (%RSD) should be $\leq$ 20% for each compound. If the %RSD of any compound is $\leq$ 20%, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

7.3.3 If the %RSD of any compound is > 20%, construct calibration curves of area versus concentration using a first order or linear fit. Correlation coefficients should be 0.990 or greater.

7.3.4 Calibration curve calculations are found in the QA Manual.

7.3.5 Reporting limit verification (RLV) is checked with each calibration curve or monthly at a minimum, by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.3.6 A mid-point standard is used for the continuing calibration standard (CCAL).

7.3.7 Standards must be replaced after 6 months, or sooner if analysis of spike samples indicates degradation or loss.

7.4 Analysis

7.4.1 GC Conditions

	Soil	Water
Initial temperature (°C)	75	75
Injection temperature (°C)	250	250
Detector temperature (°C)	250	250
Initial time (min)	0	0
Ramp rate (°C/min)	10	7
Final temperature (°C)	210	225
Final time (min)	0	0
Constant flow (mL/min)	6.0	6.0
Attenuation	0	0
Range	0	0
Injection volume (µL)	1	1

7.4.2 Water samples do not require a preparation step. Transfer samples into 2 mL autosampler vials and directly inject.

7.4.3 For water analysis, it is critical to use Restek's Drilled Uniliner® (Cat. # 21055 or equivalent). The use of this liner will result in consistent injections and better peak shapes.

7.4.4 Soil samples are analyzed from the extract collected in the 2 mL autosampler vials.

7.4.5 Retention time windows are determined by the ICAL or the initial CCAL.

7.4.6 The ICAL must be verified on each working day and after every 20 samples by analyzing a CCAL. The result must be within ± 20% or corrective action must be taken.

7.4.7 If the response for a peak exceeds the working range of the system or the highest standard, dilute the extract with 5% methanol (aqueous samples) or acetone (soil samples) and re-analyze.

7.5 Calculation

7.5.1 Calculate the concentration of the analyte in the sample using one of the following equations:

$$\textit{Water Concentration}\ (mg/L) = (C)(F)$$

$$\textit{Soil Concentration}\ (mg/kg) = \frac{(C)(V_{ex})(F)}{(W_s)(D)}$$

C = on-column concentration, µg/mL
V_{ex} = extract volume, mL
F = dilution factor (diluted volume/extract volume)
V_o = volume of sample extracted, L
W_s = sample weight, g
D = % dry weight of sample/100, or 1 for wet weight basis

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 Reporting Limit (RL)

9.1.1 The reporting limit is fixed to a constant value (Appendix B) to maintain consistency in data reporting even though the actual concentration of the lowest standard may be lower. The lowest standard should not exceed 10 µg/mL.

9.2 Method Blank

9.2.1 A method blank is analyzed with each batch of up to 20 samples prepared at the same time. The method blank must be less than the reporting limit or the sample batch is re-extracted and/or re-analyzed if possible. If it is not possible to re-extract and/or re-analyze, the data will be flagged where appropriate. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Accuracy control limits are generated for LCS and MS. In-house control charts are generated semi-annually, using 20 Percent Recovery points, as follows:

9.3.1.1 Upper and Lower Control Limit = Mean ± 3s

9.3.1.2 Upper and Lower Warning Limit = Mean ± 2s

9.3.1.3 s = Standard deviation

9.3.2 Precision control limits are generated for MS and samples. In-house control charts are generated semi-annually, using 20 RPD points, as follows:

9.3.2.1 Control Limit = Mean + 3s

9.3.2.2 Warning Limit = Mean + 2s

9.3.2.3 s = Standard deviation

9.3.2.4 RPD = Relative Percent Difference

9.3.3 In-house limits are used for compliance, as the method does not list specific limits. The limits will be reviewed for reasonableness before being used within the laboratory. In-house limits that calculate narrower than 80.0-120% are set to 80.0-120% (i.e. in-house limits = 85.8-122%, limits are set at 80.0-122%). In-house limits that calculate wider than 70.0-130% are set to 70.0-130% (i.e. in-house limits = 75.8-135%, limits are set at 75.8-130%).

9.3.4 If in-house limits have not been established, and 20 points are not collected within a year, accuracy limits are set at 70.0-130% and precision limits are set at $\leq$ 20% RPD.

9.3.5 QC calculations are found in the QA Manual.

9.3.6 LCS and MS are reviewed.

9.3.7 If the LCS data are outside the limits, a method nonconformance is filled out and the sample batch is re-extracted and/or re-analyzed if possible. If the batch cannot be re-analyzed, the data are flagged and/or a case narrative is written for all client reports within the batch.

9.3.8 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS is flagged and/or a case narrative is written.

10. REPORTING

10.1 Soil samples results are reported in mg/kg on a dry weight basis.

10.2 Water sample results are reported in mg/L.

10.3 The reported result is rounded to two significant figures.

10.4 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A – Initial Demonstration of Capability

11.2 Appendix B – Method Detection Limits and Reporting Limits

12. REFERENCES

12.1 EPA Methods 8015B and D

Appendix A

Initial Demonstration of Capability (IDC)
Ethylene and Propylene Glycol by Methods 8015B and D

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards in Ottawa sand and/or lab-grade water and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual results are entered. The mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: LCS limits or 70.0-130% (if limits aren't generated)
   Precision: LCS limits or $\leq$ 20% RPD (if limits aren't generated)

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Reporting Limits
Ethylene and Propylene Glycol by Methods 8015B and D

Parameter	Water MDL (mg/L)	Water RL (mg/L)	Soil MDL (mg/kg)	Soil RL (mg/kg)
Ethylene Glycol	2.0	10	0.23	10
Propylene Glycol	1.9	10	0.38	10

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-047.6
REVIEWER:	Triet Le
DATE:	04/22/11

SECTION	CHANGE	RATIONALE
1.1	Added reference to revision D	MDH certification for propylene glycol lists only this method
5.3, 5.5	Changed '4 ± 2 °C' to '≤ 6 °C but above freezing'	40 CFR, Part 136 update
7.4.6	Changed recovery from 30 % to 20 %	Per method 8015D
12	Added reference to revision D	Added for certification
Appendix A and B	Added revision D to headers	Added for certification
Appendix B	Added MDLs	MDLs analyzed

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: ANALYSIS OF FLASHPOINT BY PENSKY-MARTENS CLOSED-CUP APPARATUS

SOP NO.: LABENV-048.6

Original Information		
Prepared by:	Lisa Bloomgren	Date: 03/15/01
Technical Review:		Date:
QA/QC Coordinator:	Sharon Dahl	Date: 04/05/01
Authorized by:	Cheryl Sykora	Date: 04/05/01

Revision Information		
Supersedes:	LABENV-048.5	Date: 05/11/09
Revised by:	Victoria Bolton	Date: 05/02/11
Signature:	_____	Date: _____
Technical Review:	William Dahl	Date: 05/02/11
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 05/03/11
Signature:	_____	Date: _____

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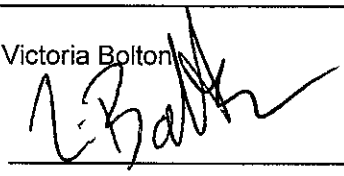
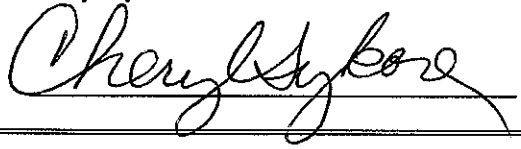
LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: ANALYSIS OF FLASHPOINT BY PENSKY-MARTENS CLOSED-CUP APPARATUS

SOP NO.: LABENV-048.6

Original Information		
Prepared by:	Lisa Bloomgren	Date: 03/15/01
Technical Review:		Date:
QA/QC Coordinator:	Sharon Dahl	Date: 04/05/01
Authorized by:	Cheryl Sykora	Date: 04/05/01

Revision Information		
Supersedes:	LABENV-048.5	Date: 05/11/09
Revised by:	Victoria Bolton	Date: 05/02/11
Signature:		Date: 5/2/11
Technical Review:	William Dahl	Date: 05/02/11
Signature:		Date: 5/2/11
Authorized by:	Cheryl Sykora	Date: 05/02/11
Signature:		Date: 5/3/11

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SOP TITLE: ANALYSIS OF FLASHPOINT BY PENSKY-MARTENS CLOSED-CUP APPARATUS

1. PURPOSE

1.1 This document defines the procedure to be followed when determining the flashpoint of a liquid, solid, or oil sample using the Pensky-Martens closed-cup apparatus. The SOP is based on ASTM D93-02a and EPA SW-846 1010A.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis.

3. PROCEDURE LIMITATIONS

3.1 The Pensky-Martens Closed-Cup method for determining flashpoint was designed for liquids. A modification of the method was developed for the analysis of solids due to client demand.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 Gloves and safety glasses should be worn.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Samples should be collected in non gas permeable containers and stored at < 35 °C or at <6°C but not freezing depending on the data quality objectives.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 Pensky-Martens closed cup apparatus

6.2 Thermometer with a range of at least 70-210 °F, or equivalent

6.3 Propane

6.4 SRM: Xylenes

6.5 Ignition Source (i.e. matches or lighter)

7. PROCEDURE

7.1 Analysis

7.1.1 All samples follow the procedure below except solid samples, which are analyzed without activating the stirring mechanism.

7.1.2 Record sample number, date, initials, and method in the flashpoint logbook. For liquids and oils, the method is recorded as PM. For solids, the method is recorded as PM-Mod.

7.1.3 Thoroughly clean and dry the cup and its accessories. Make sure all solvent is removed from the cup if used in the cleaning process.

7.1.4 For each batch of 20 samples or less analyze an SRM.

7.1.5 Fill the cup with the sample to the level indicated by the filling mark.

7.1.6 Place the lid securely on the cup and insert the thermometer.

7.1.7 Place the cup into the heating block.

7.1.8 Connect the propane source to the apparatus.

7.1.9 Open the valves on the propane tank and on the heater block.

7.1.10 Light the test flame and adjust the size using the valve on the heating block and the screw located on the cup lid.

7.1.11 Insert stir bar stem into stirring mechanism.

7.1.12 Stir sample for approximately 30 seconds.

7.1.13 Discontinue stirring and apply flame to sample for approximately 1 second by twisting the hand knob that controls the shutter.

7.1.14 If the sample does flash, discontinue testing and record flashpoint as less than or equal to the temperature the thermometer reads before heating. It may be necessary to refrigerate a sample prior to analysis depending on the data quality objectives.

7.1.15 If the sample does not flash, continue onto the next step.

7.1.16 Resume stirring.

7.1.17 Turn the dial for the heater on at a setting of 6-7 to achieve the desired temperature rate increase.

7.1.18 Apply the flame to the cup at temperature intervals of approximately 2 °F until either the sample reaches its flashpoint or the temperature rises above 200 °F.

7.1.19 Be sure to discontinue stirring before applying the test flame.

7.1.20 Do not confuse the flashpoint with the bluish halo that may appear close to the actual flashpoint.

7.1.21 Record the flashpoint in the logbook in degrees Fahrenheit to the nearest 0.5 degree. If the sample did not flash below 200 ºF, record the flashpoint as >200 ºF.

7.1.22 Perform a duplicate on any sample that flashes.

7.1.23 Turn the heater block and stirrer off.

7.1.24 Close propane valves and disconnect propane tank.

7.1.25 To cool, place cup with sample into the cup holder located on the back stand.

7.1.26 Correct results for barometric pressure:

7.1.26.1 Go to www.weather.com

7.1.26.2 Type in zip code 55103 to get local weather

7.1.26.3 Go to Current Conditions, expand to details view

7.1.26.4 Record barometric pressure in “inches of mercury”

7.1.26.5 Correct results to nearest 0.5 degrees F using the following equation:

7.1.26.6 Corrected Flash = F + (0.06*(760-(Pin*25.4mm/in))) where F = uncorrected flash in Fahrenheit and Pin = barometric pressure in inches Hg

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Samples that flashed during analysis are disposed of in the appropriate flammable waste containers.

8.3 If the sample did not flash, the aliquot used for analysis can be disposed of down the normal laboratory sink if a liquid or in the normal laboratory garbage if a solid.

8.4 Oils are disposed of in the appropriate organic waste containers.

8.5 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 SRM result must be within 2 degrees F of true value of flashpoint of xylenes solvent used.

9.2 Traceability to the lot of xylenes is included in the laboratory bench sheet and tracked in LIMS.

9.3 Duplicate RPD must be within 5%.

10. REPORTING

10.1 The flashpoint is reported in degrees Fahrenheit.

10.2 Flashpoint is reported to 2 significant figures for results of < 99 °F and to 3 significant figures for results of 100-200 °F.

11. APPENDICES

Not applicable

12. REFERENCES

12.1 ASTM D93-02a

12.2 EPA SW-846 1010A

12.3 Koehler Pensky-Martens Closed Cup Instruction Manual

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-048.6
REVIEWER:	Victoria Bolton
DATE:	05/02/11

SECTION	CHANGE	RATIONALE
5.3	Added optional storage at <6°C but not freezing depending on data quality objectives	Current Process
6.4	Added SRM	Current process
7.1.4	Frequency of SRM	Current process
7.1.18	Changed from 5 degree interval to 2 degree interval	Current process
7.1.26	Added correction for barometric pressure	Method requirement
12.2	Added reference to EPA SW-846 1010A	Clarification

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**MERCURY ANALYSIS BY COLD VAPOR GENERATION**
SOP NO.:	**LABENV-053.8**

Original Information		
Prepared by:	Lisa Bloomgren	Date: 01/15/02
Technical Review:		Date:
QA/QC Coordinator:	Terri A. Olson	Date: 04/26/02
Authorized by:	Cheryl Sykora	Date: 05/22/02

Revision Information		
Supersedes:	LABENV-053.7	Date: 07/24/09
Revised by:	Dan Brezina	Date: 11/23/10
Signature:	_____	Date: _____
Technical Review:	William Dahl	Date: 11/23/10
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 11/23/10
Signature:	_____	Date: _____

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LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**MERCURY ANALYSIS BY COLD VAPOR GENERATION**
SOP NO.:	**LABENV-053.8**

Original Information		
Prepared by:	Lisa Bloomgren	Date: 01/15/02
Technical Review:		Date:
QA/QC Coordinator:	Terri A. Olson	Date: 04/26/02
Authorized by:	Cheryl Sykora	Date: 05/22/02

Revision Information		
Supersedes:	LABENV-053.7	Date: 07/24/09
Revised by:	Dan Brezina	Date:
Signature:		Date: 11-23-10
Technical Review:	William Dahl	Date:
Signature:		Date: 11/23/10
Authorized by:	Cheryl Sykora	Date:
Signature:	Cheryl Sykora	Date: 11/23/10

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SOP TITLE: MERCURY ANALYSIS BY COLD VAPOR GENERATION

1. PURPOSE

1.1 This document defines the procedure to be followed for analyzing samples for mercury by cold vapor technique using an automated atomic absorption spectrophotometer. The SOP is applicable to samples analyzed by EPA 245.1, EPA 7470A, and EPA 7471A.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the SOP.

2.3 An analyst trained by Legend Technical Services, Inc. (LEGEND) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 Samples may need to be run at a dilution if they exceed the calibration or their matrices interfere with analysis.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 Safety glasses and gloves should be worn when handling samples and reagents.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Water samples should be collected in polyethylene or glass containers, preserved with 1:1 nitric acid to a pH < 2.

5.4 If a water sample is received with pH > 2, it is preserved in the laboratory and allowed to sit for a minimum of 24 hours at which time the pH is re-tested. The addition of the acid is noted on the appropriate chain-of-custody. If pH < 2, proceed. If not, repeat until pH is < 2, if possible. Highly alkaline samples may need to be digested at a reduced volume with excess acid, rather than dilute the original sample in an attempt to lower the pH.

5.5 Document the final pH of all samples on the bench sheet under "Comments."

5.6 The recommended holding time for water samples is 28 days.

5.7 Solid samples should be collected in polyethylene or glass containers and stored at 4 ± 2 °C.

5.8 The recommended holding time for solid samples is 28 days.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 CETAC M6000 Mercury Analyzer equipped with naphion cartridge, or equivalent

6.2 ASX-500 Model 510 autosampler, or equivalent

6.3 Top loading balance

6.4 Digestion vessels

6.5 Hydrochloric acid (HCl), concentrated, trace metal grade

6.6 Stannous chloride, reagent grade

6.7 Stannous chloride reducing solvent (10%) in 7% HCl - add 25 g of stannous chloride to a 250 mL volumetric flask containing 100 mL of DI water and 17.5 mL of HCl, dilute to volume with DI water, and invert repeatedly until stannous chloride is in solution (uncap periodically to vent gas)

6.8 Nitric acid (HNO_3), concentrated, trace metal grade

6.9 Aqua regia (3:1 HCl:HNO_3 solution) – prepare immediately before use

6.10 Potassium permanganate solution (5%)

6.11 Hydroxylamine hydrochloride, reagent grade

6.12 Certified Calibration Stock Standard – 1000 ppm (two different lots numbers are used)

6.13 Intermediate Stock Standard 1 (ISS1) – dilute 2.5 mL of the 1000 ppm certified stock standard (first lot number) and 12.5 mL of Aqua Regia with DI water in a 250 mL volumetric flask to produce a 10 ppm solution

6.14 Intermediate Stock Standard 2 (ISS2) – dilute 2.5 mL of the 1000 ppm certified stock standard (second lot number) and 12.5 mL of Aqua Regia with DI water in a 250 mL volumetric flask to produce a 10 ppm solution

6.15 Mercury Working Standard Solution 1 (MWSS1) – dilute 5.0 mL of ISS1 and 25 mL of Aqua Regia with DI water in a 500 mL volumetric flask to produce a 0.10 ppm solution and use this solution for calibration, LCS, LCSD, MS, and MSD – the spike concentration for the LCS, LCSD, MS, and MSD is 2.0 ppb

6.16 Mercury Working Standard Solution 2 (MWSS2) – dilute 5.0 mL of ISS2 and 25 mL of Aqua Regia with DI water in a 500 mL volumetric flask to produce a 0.10 ppm solution and use this solution for the QC standard or second source – the concentration for the second source standard is 5.0 ppb

7. PROCEDURE

7.1 Calibration

7.1.1 Prepare calibration standards in digestion vessels at a minimum of 3 concentration levels, ranging from 0.20 – 10 ppb, by diluting MWSS1 with DI water to the 50 mL graduation. A typical calibration curve would be:

MWSS1 mL/50 mL	Conc. (ppb)
0.0	0.0
0.10	0.20
0.25	0.50
1.0	2.0
2.5	5.0
5.0	10

7.1.2 Prepare the QC standard, or second source, in a digestion vessel by diluting 2.5 mL of MWSS2, with DI water, to the 50 mL graduation.

7.1.3 Using a disposable pipet, add 2.5 mL of aqua regia to all the digestion vessels.

7.1.4 Add 5.0 mL of 5% potassium permanganate solution to each vessel.

7.1.5 Add approximately 400 mg (~½ inch back on tip of spatula) of hydroxylamine hydrochloride to reduce the potassium permanganate, cap tightly and mix.

7.1.6 The calibration curve is Concentration vs. Response. Correlation Coefficients should be 0.995 or greater.

7.1.7 Calibration curve calculations are found in the QA Manual.

7.1.8 Reporting limit verification (RLV) is checked with each calibration curve or monthly at a minimum, by either reprocessing the corresponding calibration level or analyzing a separate standard at or below the reporting limit level. If samples are analyzed less frequently than monthly, the RLV will be checked with the next analysis. The RLV must be ± 40% or corrective action should be taken. Corrective action may include reanalysis, preparing a new standard, performing maintenance, and/or raising the reporting limit.

7.2 Analysis

7.2.1 Fill out the Mercury Log Book.

7.2.2 The QC standard, or second source, is used as the Initial Calibration Verification (ICV) and the Continuing Calibration Verification (CCV).

7.2.3 The QC standard, followed by a QC blank, should be run at the beginning of the run (ICV), after every ten samples (CCV) and at the end of the run.

7.2.4 For Methods EPA 7470A and EPA 7471A, the ICV and CCV (QC standards) recovery range is 90.0-110%. For Method EPA 245.1, the ICV recovery range is 95.0-105% and the CCV recovery range is 90.0-110%. If the CCV standard fails, the samples bracketed with that standard must be reanalyzed.

7.2.5 The absolute value of the QC blank must be less than the Reporting Limit (RL). If the QC blank fails, the samples bracketed with that blank have to be reanalyzed.

7.2.6 Set up the auto sampler as follows:

7.2.6.1 Place calibration blank, standards, and second source in the standards rack.

7.2.6.2 Place samples, spikes, and batch blank in autosampler racks.

7.2.7 Check instrument waste container level and dispose appropriately if full.

7.2.8 Make sure tubing is fitted correctly for sample and reagent lines. Replace if needed.

7.2.9 Check the lamp current and replace the lamp when the current reaches 13-14 mA.

7.2.10 Check the gas-liquid separator, inlet and drain tube for deposits. If deposits are found, clean with a 50% nitric acid solution and rinse thoroughly with DI.

7.2.11 Analyze calibration standards and samples.

7.2.12 Upon completion of the automated run, complete filling out the mercury logbook.

7.3 Calculation

7.3.1 Compute the concentration of the analyte in the sample using the following equation:

$$Water\ Concentration\ (mg/L) = \frac{(C_{in})(FV)(D)}{V(1000)}$$

$$Soil\ Concentration\ (mg/kg) = \frac{(C_{in})(FV)(D)}{M(1000)}$$

C_{in} = in-solution concentration, µg/L
FV = final volume, mL
D = dilution factor
V = volume of sample, mL
M = mass of sample, g

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

8.2 Acidic waste must be neutralized prior to disposal.

8.3 Highly contaminated samples are returned to the client for disposal.

9. QA/QC

9.1 MDL, PQL, RL

9.1.1 Method detection limits (MDLs) and practical quantitation limits (PQLs) are updated, using at least seven replicate spikes, on an annual basis. Reporting limits (RLs) are based on a combination of MDL/PQL studies, interference studies, client requirements, and analyst experience. Data are not always reproducible at statistical MDL and PQL levels; therefore, judgment is used to determine actual RL. The RL used must be greater than or equal to the calculated PQL. Current MDL and RL values can be found in Appendix B. Project specific RLs may override those listed.

9.2 Method Blank

9.2.1 A method blank is analyzed with each group of up to 20 samples prepared at the same time. The absolute value of the method blank must be less than the reporting limit or the sample batch is re-digested, if possible. If it is not possible to re-digest, the data will be flagged where appropriate. Do not subtract compounds in the blank from sample results. Report all blank results with the samples.

9.3 Control Limits

9.3.1 Method limits are used for compliance (see Appendix C).

9.3.2 QC calculations are found in the QA Manual.

9.3.3 LCS and MS are reviewed.

9.3.4 If the LCS data are outside the limits, a method nonconformance is filled out and the sample batch is re-digested if possible. If the batch cannot be re-digested, the data are flagged and/or a case narrative is written for all client reports within the batch.

9.3.5 If the MS data are outside the limits (the LCS has all ready been reviewed and is acceptable), the information is placed in the daily and project files, and the data for that specific MS is flagged and/or a case narrative is written.

10. REPORTING

10.1 Solid sample results are reported in mg/kg on a dry weight basis.

10.2 Bulk sample results are reported in mg/kg on an as received basis.

10.3 Water sample results are reported in mg/L.

10.4 The reported result is rounded to two significant figures.

10.5 The results are placed in the client file and a final report is sent to the client.

11. APPENDICES

11.1 Appendix A - Initial Demonstration of Capability

11.2 Appendix B - Method Detection Limits and Report Limits

11.3 Appendix C - Method Limits

12. REFERENCES

12.1 EPA Methods 245.1, 7470A and 7471A

12.2 M-6000A Mercury Analyzer Operator's Manual, CETAC Technologies, Inc., 480035, Version 1.2, March, 1997

12.3 M-6000A Mercury Analyzer Software Manual, CETAC Technologies, Inc., 480034, Version 1.1, May, 1997

12.4 ASX-500 Model 510 Auto Sampler Operator's Manual, CETAC Technologies, Inc., 480049, Version 1.0, March, 1997

Appendix A

Initial Demonstration of Capability (IDC)
Mercury Analysis by Cold Vapor Generation

1. Prior to using any existing published method, and at any time there is a significant change in the published test method, instrument type or personnel, a demonstration of capability must be made.

2. Prepare four mid-level replicate standards and a reagent blank.

3. Analyze the replicates and reagent blank per the SOP.

4. Compile the following information and give to the QA Department.

   - Analyst
   - Test/procedure
   - Matrix
   - Date of testing
   - Results

5. Using LEGEND form 'IDC 4 rep with RPD', the individual recoveries in concentration and %, the mean recovery in concentration and %, and the % RPD of the replicates are calculated.

6. The results must meet the following criteria:

   Accuracy: EPA 245.1 = 85.0-115%, EPA 7470A and 7471A = 80.0-120%
   (245.1/7470A may be combined if tighter limits are used for acceptance)
   Precision: $\leq$ 20.0% RPD

7. The reagent blank must be less than the reporting limit (RL) – see Appendix B.

8. If the IDC is acceptable, the QA/QC Coordinator signs the form and a copy of the form is placed in the employee's personnel folder.

9. If the IDC is not acceptable, it will be reanalyzed.

Appendix B

Method Detection Limits and Reporting Limits
Mercury Analysis by Cold Vapor Generation

Water

Parameter	MDL (mg/L)	RL (mg/L)
Mercury	0.000037	0.00020

Soil

Parameter	MDL (mg/kg)	RL (mg/kg)
Mercury	0.019	0.10

Appendix C

Limits for Mercury Analysis by Cold Vapor Generation

Method	Accuracy Method Limits (%)	Precision Method Limits (%RPD)
EPA 245.1 (LCS)	85.0-115	≤ 20
EPA 245.1 (MS)	75.0-125	≤ 20
7470A (LCS)	80.0-120	≤ 20
7470A (MS)	75.0-125	≤ 20
7471A (LCS)	80.0-120	≤ 20
7471A (MS)	75.0-125	≤ 20

DOCUMENT REVIEW

DOCUMENT:	SOP LABENV-053.8
REVIEWER:	Dan Brezina
DATE:	11/23/10

SECTION	CHANGE	RATIONALE
Various	Deleted digestion log and added reference to LIMS bench sheet	Current procedure
Appendix B	Updated MDLs	Annual update

LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE:	**BULK SAMPLE IDENTIFICATION USING POLARIZED LIGHT MICROSCOPY**
SOP NO.:	**LABIH-003.9**

Original Information		
Prepared by:	Corine Goodrich	Date: 06/11/02
Technical Review:	Corine Goodrich	Date: 06/11/02
QA/QC Coordinator:		Date:
Authorized by:	Cheryl Sykora	Date: 06/15/02

Revision Information		
Supersedes:	LABIH-003.8	Date: 04/29/09
Revised by:	Todd Giorgi	Date: 09/15/10
Signature:	_____	Date: _____
Technical Review:	Keith Giorgi	Date: 09/15/10
Signature:	_____	Date: _____
Authorized by:	Cheryl Sykora	Date: 09/15/10
Signature:	_____	Date: _____

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LEGEND TECHNICAL SERVICES, INC.

STANDARD OPERATING PROCEDURE

TITLE: BULK SAMPLE IDENTIFICATION USING POLARIZED LIGHT MICROSCOPY

SOP NO.: LABIH-003.9

Original Information		
Prepared by:	Corine Goodrich	Date: 06/11/02
Technical Review:	Corine Goodrich	Date: 06/11/02
QA/QC Coordinator:		Date:
Authorized by:	Cheryl Sykora	Date: 06/15/02

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Revised by:	Todd Giorgi	Date:
Signature:		Date: 9/15/10
Technical Review:	Keith Giorgi	Date:
Signature:		Date: 9/15/10
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SOP TITLE: BULK SAMPLE IDENTIFICATION USING POLARIZED LIGHT MICROSCOPY

1. PURPOSE

1.1 This document defines the procedure to be followed for identifying the presence of asbestos and estimating the percent composition by visual estimation and/or point counting in bulk samples. The SOP is applicable to samples typically analyzed by EPA Method 600/R-93/116, 1993.

2. RESPONSIBILITY/PERSONNEL

2.1 It is the responsibility of the designated quality assurance supervisor to ensure this procedure is followed.

2.2 It is the responsibility of the laboratory analyst to perform all quality control steps as defined in the standard operating procedure.

2.3 It is the responsibility of the supervising microscopist to ensure that this procedure is used, and to resolve any discrepancies with bulk sample results.

2.4 It is the responsibility of the individual using this procedure to bring any problems to the attention of the supervising microscopist.

2.5 An analyst trained in polarized light microscopy (PLM) shall perform the analysis. Each new analyst performs an Initial Demonstration of Capability (IDC). The IDC information can be found in Appendix A.

3. PROCEDURE LIMITATIONS

3.1 This SOP is applicable to bulk asbestos samples.

4. HEALTH AND SAFETY

4.1 Read all Material Safety Data Sheets (MSDS) associated with the chemicals used in this procedure.

4.2 Follow standard laboratory safety practices.

4.3 When working with bulk samples, wear solvent resistant nitrile gloves.

4.4 All work with samples, prior to their being placed on slides, should be done in a HEPA hood.

5. SAMPLE COLLECTION/ACCEPTANCE/REJECTION

5.1 The sample shall be accepted if packaged to protect the sample's integrity and clearly labeled for identification.

5.2 The sample shall not be accepted if it is not clearly identified or packaged so as to protect the sample's integrity.

5.3 Clients bringing samples in inappropriate containers (or no containers) may be provided with a plastic sealable bag, or other appropriate container, to transfer the sample into.

5.4 The sample may be rejected if insufficient sample volume is available.

5.5 The sample may be rejected if it is not representative of the material. The field personnel should make the determination.

5.6 The analyst will contact the client if there are any questions regarding the layers to be analyzed. Roofing material samples are considered one layer.

6. EQUIPMENT/MATERIALS/REAGENTS

6.1 A polarized light microscope (PLM) with polarizer, analyzer, port for wave retardation plate, 360º graduated rotating stage, substage condenser, lamp, lamp iris, and dispersion staining objective

6.1.1 Objectives - 10x, 20x, and 40x or near equivalent

6.1.2 Ocular (eyepieces) - 10x minimum

6.1.3 Dispersion staining objective

6.1.4 Compensator plate - Ca 550 nm ± 20 nm

6.2 Microscope with binocular head, mechanical stage and Koehler illumination capabilities

6.3 Stereomicroscope with magnification of 10x to 40x

6.4 Precleaned glass microscope slides: 75 mm x 25 mm, or equivalent

6.5 Glass cover slips - 22 mm x 22 mm, or equivalent

6.6 Forceps

6.7 Scalpel

6.8 Micropipet

6.9 Syringe

6.10 Kimwipes®

6.11 Mortar and pestle, or mini-blender

6.12 Standard thermometer

6.13 Low temperature muffle furnace capable of heating to 500 ºC

6.14 Drying oven capable of heating to 105 ºC

6.15 HEPA ventilated hood with continuous airflow

6.16 Deionized water

6.17 Hydrochloric acid

6.18 Methylene chloride

6.19 Polycarbonate filters - ca. 0.4 µm pore size

6.20 National Institute of Standards and Technology (NIST) asbestos reference samples, SRM #1866 and 1867

6.21 Precision Calibrated Optical Glass M-25 Reference Set; R.P. - Cargille Laboratories, or equivalent

6.22 Cargille refractive index liquids 1.460 - 1.710 (need 1.550, 1.605, 1.670, 1.680 and access to the rest), or equivalent

6.23 Bulk Asbestos Analysis Sheet

6.24 Bulk Asbestos Point Count Sheet

6.25 Hand-held lab counter (optional)

6.26 Eyepiece reticle equipped with cross hair or Chalkley 25 point array (optional)

6.27 Stage micrometer with 0.1 mm subdivisions (optional)

7. PROCEDURE

7.1 Preparation of Samples

7.1.1 Samples that are received wet should be placed in an oven at approximately 100 °C until dry, prior to mounting.

7.1.2 Fibers that are heavily coated with binder should be scraped with a scalpel before being mounted in refractive index liquid or washed with methylene chloride.

7.1.3 Samples with heavily coated fibers can be washed in hydrochloric acid, rinsed with distilled water and then dried. Fibers can then be mounted in refractive index liquid.

7.1.4 Floor tile samples that cannot be broken down should be ignited in a low temperature furnace at 500 ºC for approximately 15-30 minutes, as needed, before mounting in refractive index liquid.

7.1.5 Black mastic samples should be washed with methylene chloride.

7.1.6 Roofing samples should be muffled in a low temperature oven at 500 ºC before analyzing.

7.1.7 Clean all mounting tools with Kimwipes® and water, then place on clean surface inside HEPA hood.

7.1.8 Place a clean laminated board inside the ventilated hood and place two parallel rows of precleaned glass slides on the board. Disposable paper can be used on the boards if needed. Label and designate the upper row for samples using 1.670 refractive index liquid and the bottom row for samples using 1.550 refractive index liquid.

7.1.9 Assign two slides to each sample number and label accordingly. Apply 1-2 drops of 1.550 refractive index liquid on the first slide, and 1-2 drops of 1.670 refractive index liquid on the second.

7.1.10 Stereoscopically examine the sample. Note different fiber types, color, texture and homogeneity of the sample.

7.1.11 Bring the sample under a hood, using the precleaned forceps

7.1.12 Remove a small but representative quantity of the bulk sample and apply to each of the previously prepared slides. A scalpel can be used to slice off thin sections of harder materials. A mortar and pestle can also be used to break down hard materials.

7.1.13 Tease each small quantity apart with clean forceps. The sample material should be distributed throughout the refractive index liquid.

7.1.14 Place a clean cover slip on the mixture of liquid and sample.

7.2 Calibration

7.2.1 Refractive Index Liquid Calibration

7.2.1.1 Select the glass standard with an RI closest to that of the liquid.

7.2.1.2 Mount the standard in the liquid and observe the dispersion staining color (either particle edge or Becke line).

7.2.1.3 Measure the temperature of the liquid (°C).

7.2.1.4 Convert the dispersion color observed into a corresponding wavelength from the dispersion-staining table.

7.2.1.5 Fill out the 'Refractive Index Liquid Calibration Form' with the information above.

7.2.1.6 The form will automatically calculate n^L_D from the following equation:

$$n^L_D = n^S_D - (\Delta^L - \Delta^S) * K$$

Where:

$\Delta^L = (n_f - n_c)$ of liquid
$\Delta^S = (n_f - n_c)$ of solid

$$K = \frac{(wavelength_0 - 200)^{-1} - 0.002571}{0.001304}$$

7.2.1.7 The form will also calculate $n^{25\ °C}_D$ from the following equation:

$$n^{25\ °C}_D = n^L_D + (25 - t) * dn_D/dt$$

Where:

t = ambient temperature (°C)
dn_D/dt = (-)value from label on liquid

7.2.1.8 Record a change in refractive index greater than 0.004 on the appropriate bottle.

7.3 Analysis

7.3.1 Samples that contain two or more discrete layers will be analyzed and reported separately for each individual layer.

7.3.2 Asbestos Identification

7.3.2.1 Use an eraser to rub the top of the cover slip and spread the sample out.

7.3.2.2 When a fiber group is found, center a fiber on the cross hairs, adjust to 100x magnification and focus. While rotating the microscope stage, observe for pleochroism and note color if found.

7.3.2.3 Follow the flow chart in Appendix B (Procedure for Microscopic Identification of Asbestos) for identification. If amosite fibers have a pale color in the 1.670, use 1.680 for more accurate central stop color.

7.3.2.4 The optical properties of asbestos are defined in Table 1-1 of EPA-600/M4-82-020, 1982, “Interim Method for the Determination of Asbestos in Bulk Insulation Sample” and on pages 19 through 21 of EPA-600/R-93/116, 1993, "Method for the Determination of Asbestos in Bulk Building Materials".

7.3.2.5 Record at least one definitive optical property for non-asbestos fibers, which distinguishes them from asbestos fibers.

7.3.2.6 Record all information compiled from the flow chart analysis on the Bulk Asbestos Analysis Sheet.

7.3.2.7 Repeat until all fiber groups have been identified.

7.3.2.8 For precision and accuracy an asbestos reference sample can be mounted and compared to the sample being analyzed.

7.3.2.9 Vermiculite will be treated as a heterogeneous material. Prior to the above identification, vermiculite will be separated by using the stereoscope. Samples will be analyzed in accordance with Section 9.2 Rapid Screening Analysis to Determine the Weight Percent of Fibrous Amphibole in Vermiculite Attic Insulation of “Research Method for Sampling and Analysis of Fibrous Amphibole in Attic Insulation”.

7.3.2.10 Upon receiving the results, the client will communicate to the analyst whether or not a point count should be performed.

7.3.3 Point Counting

7.3.3.1 If the sample is not homogeneous, a portion must be homogenized by crushing and mixing with a mortar and pestle or mini-blender.

7.3.3.2 From the homogenized sample, take 4-8 random pinch samples and place each on a separate microscope slide.

7.3.3.3 Mount the samples in a refractive index liquid that is suitable (based on the asbestos identification) for the sample and place a cover glass on them.

7.3.3.4 Disperse particles and fibers using a pencil eraser to ensure a non-crowded field of view and to avoid overlapping particles.

7.3.3.5 Place either a cross hairs or point array graticle (e.g. Chalkley 25 point array), in the eyepiece and set the microscope up with the first order wave plate.

7.3.3.6 Analyze the samples using 100x – 200x magnification.

7.3.3.7 Place one of the prepared slides on the stage and move randomly from field to field.

7.3.3.8 Count points (array or cross hair) that fall directly over fiber or binder particles, but do not count unoccupied points. The sample should be well dispersed to allow 25 - 50% empty points. Keep track of the number of occupied points for asbestos separately from the total number of occupied points. If an asbestos point and a binder point occupy a point, the point is counted twice.

7.3.3.9 Count 50-100 occupied points per preparation. Repeat this procedure for all preparations for a total of 400 occupied points.

7.3.3.10 If the point count yields a 'zero' result but asbestos fibers are observed, report as 'trace'.

7.3.3.11 Record all information from the point count on the Bulk Asbestos Point Count Sheet.

7.4 Calculation

7.4.1 Calculate the asbestos percentage as follows:

$$\% \text{ asbestos} = a/t \times 100\%$$

where:

a = total number of asbestos points counted
t = total number of occupied points counted

7.4.2 If a result obtained by point count is different from a result obtained by visual estimation, the point count result will be used.

8. WASTE DISPOSAL

8.1 Dispose of all samples and analysis materials in accordance with current company waste disposal procedures.

9. QA/QC

9.1 The reporting limit (RL) is set at 1% for standard PLM analysis.

9.2 Method Blank

9.2.1 A blank is set up daily using fiberglass as the asbestos-free material. The blank may not contain any amount of asbestos.

9.2.2 If asbestos is found, the blank will be set up a second time for confirmation.

9.2.3 If asbestos is found in the confirmation step, a contamination problem is probable and corrective action is initiated. Corrective actions may include but are not limited to cleaning the glass slides or covers slips, re-cleaning the forceps, HEPA vacuuming the interior of the hood, or replacing the refractive index oils.

9.2.4 If asbestos is not found in the confirmation step, corrective action is not required.

9.2.5 Results are recorded on the 'Daily Checklist Form.'

9.3 If sample is run multiple times, calculate the standard deviation of the results.

9.4 The data should be reviewed by a second person that has sufficient technical expertise. Reviewed data should be initialed and dated by the reviewer.

9.5 Continuing Training

9.5.1 Analysts participate in Round Robin meetings with their peers where asbestos analyses issues are discussed.

9.5.2 Analysts participate in AHERA asbestos training.

9.5.3 Microscopy courses (if applicable).

9.6 For quality control purposes, 10% of the samples will be set up in duplicate. Choose duplicates so approximately 50% are positive for asbestos and 50% are negative for asbestos. Duplicate samples will be analyzed at the end of each week. The results of these samples will be recorded on the 'Bulk Asbestos Chart Table' form. The acceptable ranges are calculated on each appropriate form (1-10% and >10%). If a duplicate is outside the acceptable range, a method nonconformance report is filled out.

9.6.1 If the column containing 'Flag P' is an 'F", the duplicate has failed qualitatively. The sample is reanalyzed, if possible, and a method nonconformance report is filled out.

9.7 If two different analysts read the same slide, the acceptable ranges are:

9.7.1 ≤ 10% asbestos – within 3% units (both analysts must report >1% on sample)

9.7.2 10% to 75% - within 20% units

9.7.3 ≥ 75% asbestos – within 10% units

9.8 Estimation of fiber percentage – reference samples, made by the analyst, are used to estimate percentage until the analyst is proficient. If there is any question of the percentage, a reference slide will be made for comparison.

9.9 If there are discrepancies as to whether the material contains asbestos or not, the supervising microscopist will make the final determination. All samples that appear to have a discrepancy between the bulk sample and the analysis sheet will be re-examined.

9.10 Daily maintenance of polarizing microscopes used in bulk analysis includes:

9.10.1 Checking the alignment of the central stop.

9.10.2 Ensuring that the polarizer and analyzer are orientated at 90 degrees to one another by examining a NIST anthophyllite reference sample.

9.10.3 Centering the objectives.

9.10.4 Checking that the substage condenser and iris diaphragm are centered.

9.10.5 Recording all maintenance requirements on the 'Daily Checklist' form.

9.11 The temperature of the microscope room will be documented daily on the 'Daily Checklist' form.

9.12 When analyzing anthophyllite, actinolite, and tremolite fibers, the polarizer and analyzer will be checked for orientation of 90 degrees to one another, during and after analysis, to ensure that the extinction angles are correct by using the NIST anthophyllite reference standard.

9.13 Point counting will be done once a month on a random sample. The results will be entered on the 'Asbestos Analysis Comparison' form.

9.14 Refractive index liquids will be calibrated with accuracy of ± 0.004 once a month or as needed with Cargille R.I. glass beads and recorded on the 'Refractive Index Liquid Calibration' form.

9.15 Each analyst will participate in the Twin City Asbestos Round Robin (TCARR) with a minimum of four other laboratories on a quarterly basis.

9.16 Each analyst will analyze the National Institute of Standards and Technology's National Voluntary Laboratory Accreditation Program's (NVLAP) proficiency bulk samples. The lead analyst's results are sent to NVLAP.

9.17 If a NVLAP sample was not analyzed within the month, a previously analyzed NVLAP sample will be analyzed. The results of these samples will be recorded on the 'NVLAP Chart Table' form.

9.18 Each month the 'Bulk Asbestos Analysis Monthly QA Summary' will be filled out. The pass/fail percentages for identification and % asbestos will be calculated as the monthly failure rate and should not exceed 1%.

9.19 Records of all bulk QA/QC will be kept with the department's quality control files.

9.20 Requirements

9.20.1 Round Robin participation with at least four other laboratories.

9.20.2 Participation in the National Institute of Standards and Technology's National Voluntary Laboratory Accreditation Program (NVLAP).

9.20.3 All bulk samples will be analyzed by NVLAP authorized personnel at LEGEND or by an analyst at another NVLAP accredited laboratory.

10. REPORTING

10.1 Samples results are reported in %.

10.2 Samples with no detected asbestos are reported as "None Detected".

10.3 Samples with asbestos detected below the reporting limit are reported as <1% or trace.

10.3.1 When the analyst ascertains that although there are asbestos fibers in the sample its concentration is so low that it is far from the level of 1% by calculated visual estimation or point counting, the analyst will report the asbestos concentration as a trace. In this case, the analysis is confident that it is not necessary to make a recommendation to the client that the sample should be analyzed by a more accurate and precise method to verify the sample results.

10.3.2 When reporting the asbestos as <1%, the analyst ascertains that the asbestos concentration is not equal to or higher than 1% by calibrated visual estimation or point counting. When this occurs, the analyst should recommend to the client that verification of the results may be necessary by a more accurate and precise method, such as the bulk TEM methods.

10.4 Samples analyzed using the standard PLM analysis, report results above the reporting limit to the whole number (i.e. 2, 25).

10.5 Samples analyzed using point count, report results at two places past the decimal point (i.e. 0.25, 1.50).

10.6 The results are placed in the client file and a final report is sent to the client indicating the estimated percentage of fibers present and their identification.

11. APPENDICES

11.1 Appendix A - Initial Demonstration of Capability

11.2 Appendix B - Procedure for Microscopic Identification of Asbestos

12. REFERENCES

12.1 EPA-600/R-93/116, 1993, "Method for the Determination of Asbestos in Bulk Building Materials"

12.2 40 CFR Part 763, Volume 52, Number 210, "Asbestos Containing Material in Schools; Final Rule and Notice"

12.3 ASTM D-22 Proposal P236, "Proposed Test Method for Asbestos Containing Material by Polarized Light Microscopy"

12.4 McCrone, Walter C., "Asbestos Identification," McCrone Research Institute, 1987

12.5 Code of Federal Register, Volume 55, Number 224, Nov. 20, 1990; "NESHAP for Asbestos: Point Counting"

12.6 Federal Register, Volume 51, No. 119/Friday, June 20, 1986 under Rules and Regulations for OSHA

12.7 Calibration of Refractive Index Liquids by Using Optical Glass Standards and Dispersion Staining Technique; The Microscope, McCrone Research Institute, Vol. 40, 1992

12.8 Microscope Techniques; R.P. Cargille Laboratories, Inc.; Technical Bulletin RI-T-265

12.9 'Refractive Index Liquid Calibration Using Optical Glass Standards'; Shu-Chun Su, Ph.D; October 1996

12.10 EPA-600/M4-82-020, Dec. 1982, “Interim Method for the Determination of Asbestos in Bulk Insulation Sample

12.11 40 CFR, Part 763, Volume 59, Number 23, February 3, 1994; “Asbestos Model Accreditation Plan; Interim Final Rule

12.12 40 CFR, Part 61, Volume 59, Number 3, January 5, 1994; “Asbestos NESHAP Clarification Regarding Analysis of Multi-layered Systems

12.13 NIST Technical Note 1297, 1994 Edition, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results

12.14 EPA, “Research Method for Sampling and Analysis of Fibrous Amphibole in Vermiculite Attic Insulation” (commonly referred to as the Chatfield/Cincinnati Method)

Appendix A

Initial Demonstration of Capability

1. Each analyst will be trained at the McCrone Research Institute for asbestos identification, when a class is available.

2. Analyze 25 IDC samples over time and record the information below

   A. Analyst name
   B. Date analyzed
   C. Fiber type
   D. % of each fiber type found

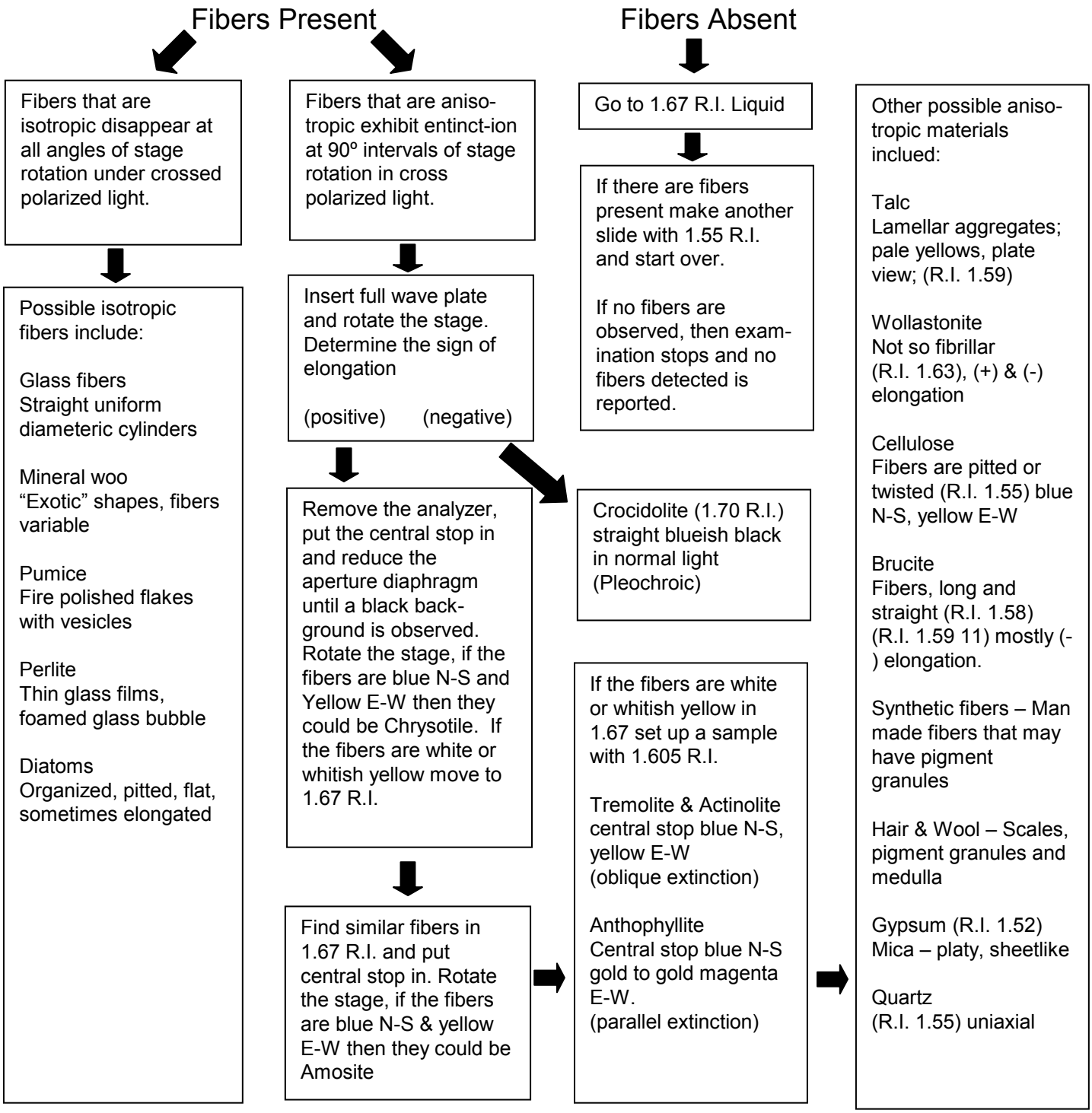
3. 100% of the samples must pass fiber type identification and 99% of the samples must pass the % asbestos.

4. If the IDC is acceptable, the analyst, supervisor, and QA/QC Coordinator sign the form and a copy of the form is placed in the employee's personnel folder.

5. If the IDC is not acceptable, the samples that failed will be reanalyzed and resubmitted until an acceptable IDC is completed.

Appendix B

Procedure for Microscropic Identification of Asbestos



DOCUMENT REVIEW

DOCUMENT:	SOP LABIH-003.9
REVIEWER:	Todd Giorgi
DATE:	09/15/10

SECTION	CHANGE	RATIONALE
6.2	Capablties to capabilities	Typo
7.3.2.9	Revised vermiculite analysis	NVLAP audit finding
9.2.1	Changed method blank to corn starch versus fiberglass	NVLAP audit finding
9.6	Added sentence to state MNC will be filled out if duplicate is outside acceptable range	NVLAP audit finding
12.14	Added reference to vermiculite research method	Referenced in 7.3.2.9

Title: Preparation and Analysis of Nitrocellulose in Aqueous and Soil/Sediment Samples by Colorimetric AutoAnalyzer

Approvals (Signature/Date):

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Technical Manager

Robert Hrabak — 11/12/10 — Date
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1. SCOPE AND APPLICATION

1.1. This method determines nitrocellulose (NC, CAS Number 9004-70-0) by calculation from detected nitrate and nitrite concentrations in aqueous and soil/sediment samples.

1.2. The analytical range for this method is 0.050 to 2.0 mg/L for nitrate plus nitrite. Samples that are over the linear range are diluted and reanalyzed.

1.3. The QuanTIMs method code is WA; cross-reference NCEL_ [A, S].

1.4. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Nitrocellulose is extracted by filtering an aqueous sample through a membrane filter. The membrane filter is extracted with acetone, which is collected in a 50 mL tube.

2.2. Soil/sediment samples for nitrocellulose are washed two times with 1:1 methanol-water. Following the rinse, the soil residue is extracted with acetone, which is decanted into a 50 mL tube.

2.3. The acetone extracts are mixed with sodium hydroxide, and reduced under nitrogen until the acetone has evaporated. The basic solution is heated to hydrolyze the NC to nitrate/nitrite, and then brought up to volume with water. After pH adjustment, an aliquot is filtered and analyzed colorimetrically for nitrate plus nitrite by method MCAWW 353.2.

2.4. Extracts are analyzed on an automated colorimetry instrument fitted with a cadmium reduction coil. A filtered sample is passed through a reduction column to reduce nitrate to nitrite. The sample is then treated to form a highly colored azo dye that is measured colorimetrically. The absorbance is directly proportional to the concentration of nitrate plus nitrite as N.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Nitroaromatics and nitroamines may be removed from the soil/sediment by washing with 1:1 methanol-water before extraction.

4.1.1. Build up of suspended matter in the column restricts sample flow. Particulates are removed by filtering the samples using 0.45 µm filters.

4.2. Iron, copper, and other metals may interfere with the accurate analysis of nitrate and nitrite by binding with the nitrate and/or nitrite in the sample, thus blocking the color formation reaction. EDTA is used in the buffer solution to eliminate the interference.

4.3. Oil and grease will coat the cadmium surface and cause low recoveries. This interference can be removed from the samples by pre-extraction with an organic solvent such as chloroform, methylene chloride, or another suitable solvent.

4.4. Samples preserved with mercuric chloride or sodium thiosulfate will degrade the cadmium reduction column. DO NOT analyze such samples by this method.

4.5. Physical interferences such as color and turbidity in the samples will cause high results and can be minimized by filtration or dilution.

4.6. Samples that discolor when exposed to air for an extended period of time must be analyzed immediately. For this type of sample, it is recommended to pour the solution into the sample cup immediately prior to sample injection to minimize exposure to air.

4.7. Contaminants in solvents, reagents, glassware, and other processing hardware can cause interferences that lead to discrete artifacts and an elevated baseline in the analysis. These materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks and/or method blanks.

4.8. Reagent blanks and/or method blanks are analyzed with each batch to demonstrate that the samples are free from method interferences and artifacts.

4.9. The standards and reagents used should be the highest grade possible to minimize interference problems, minimally reagent grade.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents

are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Handle nitrocellulose (NC) with care, especially when the nitrocellulose contains a high percentage of nitrogen (10% to 14% N). NC is unstable and explosive when dried.

5.1.2. Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.1.3. The use of vacuum systems during Anodisc membrane filtering presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl, or nitrile gloves may be used. Latex and vinyl should not be used when handling methylene chloride or other organic solvents, as they provide no significant protection against these solvent.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in**

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid (1)	Corrosive Oxidizer Dehydra-dator	1 mg/m3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Sodium Hydroxide	Corrosive	2 mg/m3 - Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
EDTA buffer (disodium ethylenediamine tetraacetate	Irritant	None listed	Inhalation may cause respiratory tract irritation. Contact may cause skin or eye irritation.
Phosphoric Acid (1)	Corrosive	1 mg/m3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
N-1-Naphthylethylene diamine Dihydrochloride	Irritant	None Listed	Inhalation, skin contact or eye contact may all cause irritation.
Hydrochloric Acid (1)	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Copper (Cupric) Sulfate	Irritant	1 mg/m3 PEL	Skin contact may cause irritation and itching. Eye contact may cause conjunctivitis, ulceration or clouding of cornea. Inhalation may cause irritation to respiratory tract, and may cause perforation or ulceration.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

Note: *The following apparati are suggested items used in the laboratory. Alternative items may be substituted.*

6.1. Electronic analytical top-loading balance, readable to 0.0001 grams for the preparation of standards.

6.2. Nuclepore Track-Etch membrane filters, 0.2-µm, 47 mm, Whatman International Ltd., catalog number 111106. Handle with care, as membranes are delicate.

6.3. Balance, electronic top-loading, readable to 0.1 grams and a capacity up to approximately 3500 grams for preparation of samples and reagents.

6.4. Beakers, 250 mL, glass, rinsed with acetone and dried before use.

6.5. Bottle; appropriate size, amber glass, with Teflon-lined cap.

6.6. Centrifuge, IEC model Centra-4B.

6.7. Centrifuge tubes, polypropylene, 50 mL capacity with screw cap; Fisher catalog number 05-539-9, or equivalent.

6.8. Filtering apparatus for 47-mm membrane filter, receiving flask, and vacuum pump/trap. Rinse the receiving flask with acetone and dry before use.

6.9. Filter assembly, 0.45 µm, 25 mm PTFE, GD/X; Whatman catalog number 6874-2504, or equivalent, and PTFE, Millipore Millex-LCR, catalog number SLCR025 NB, or equivalent.

6.10. Flasks, volumetric, glass with stopper, appropriate sizes for preparation of standards.

6.11. Alpkem Flow Solution IV automated flow analyzer, consisting of the following:

6.11.1. Automatic sampler.

6.11.2. Proportioning pump.

6.11.3. Injection module equipped with microloop.

6.11.3. Colorimeter with 540 nm filter and flow cell.

6.11.4. Reaction cartridges (#A002670); with an open tubular cadmium reactor (OTCR) (#AOW897).

6.11.5. WinFlow software system (Version 4.0 or equivalent).

6.11.6. Pillow assembly filled with nitrogen gas for segmented flow analysis (SFA).

6.12. Disposable autosampler vials or culture tubes, 12 x 75 mm for samples and standards on the Alpkem autoanalyzer.

6.13. Micropipet, 100-200 µL Wiretrol.

6.14. Nitrogen gas manifold, N-Evaporator, Organomation Analytical Associates model 112.

6.15. Pipet, various sizes from 0.5 mL to 10 mL, gravimetric (disposable).

6.16. Pipet, Pasteur type, glass, 5¾" or 9" long.

6.17. Pipet, volumetric, glass, various sizes.

6.18. pH meter.

6.19. Spatula, micro, stainless steel; for weighing of analytical standards.

6.20. Syringe, 10-cc (disposable), B-D product # 9604 or equivalent.

6.21. Test tube, glass, 16 x 100 mm (16 mL), with Teflon lined screw cap.

7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. When available, pre-made, commercially prepared reagents are purchased.

7.2. Ammonium Hydroxide, concentrated.

7.3. Acetone, pesticide quality or equivalent.

7.4. Brij-35, polyoxyethylene 23 lauryl ether, 30% solution, Fisher, reagent grade or equivalent.

7.5. Methanol, pesticide grade or better.

7.6. Water, organic-free, reagent quality or better. Refer to SOP WS-QA-0014, "Monitoring of Reagent-Grade Laboratory Water" for further details.

7.7. Sodium hydroxide, 1N, aqueous solution. Prepare by dissolving 20.0 grams of sodium hydroxide pellets into a 500 mL volumetric flask with reagent water. Mix well. Store the solution in a plastic bottle, label, and cap tightly. The solution is stable for up to 6 months.

7.8. Sodium hydroxide, 15N, aqueous. Prepared by dissolving 150 g of sodium hydroxide pellets into a 250 mL volumetric flask with deionized water; mix well. Store the solution in a plastic bottle, and cap tightly. The solution is stable for up to 1 year.

WARNING: Sodium hydroxide solution will get extremely hot and give off irritating fumes. Dissolve sodium hydroxide pellets into at least 200 mL of water in the volumetric flask, dissolve the pellets, and dilute to volume. Prepare the solution in the hood.

7.9. Sulfuric acid, 2N, aqueous. Prepared by diluting 28 mL of concentrated sulfuric acid in a 500 mL volumetric flask with reagent water; mix well. Store the solution in a glass bottle, and cap tightly. The solution is stable for up to 6 months.

WARNING: Carefully, add sulfuric acid to water in the volumetric flask, mix well, and dilute to volume. A violent reaction can occur when adding water to concentrated acid. Prepare the solution in the hood.

7.10. Ammonium chloride-EDTA stock solution (buffer solution). Prepare by dissolving 85.0 g of ammonium chloride and 1.0 g of disodium ethylenediamine tetraacetic acid dihydrate (EDTA) in approximately 800 mL of deionized water in a 1 L volumetric flask. Adjust the pH to 8.5 by adding concentrated ammonium hydroxide solution dropwise (monitor with a pH meter) and dilute to the 1 liter mark with deionized water. Filter solution prior to use. The solution is stable for up to 1 year.

7.10.1. Ammonium chloride-EDTA working solution (working buffer solution). Working buffer is prepared by adding 1 mL Brij-35 per 500 mL stock buffer solution. Working buffer is good for 1 week.

7.11. Sulfanilamide reagent. Prepare by adding 100 mL of 85% phosphoric acid to a 1 L flask containing at least 600 mL of deionized water, mix gently, and add 40.0 g of sulfanilamide and 2.0 g of N-1-naphylethylenediamine dihydrochloride (NED). Mix well until the reagents dissolve, and bring the volume to 1-liter mark with deionized water. This solution is light sensitive; store in an amber glass bottle. The solution is stable for three months. Discard when severe discoloration (brown) occurs prior to the three month shelf life.

7.12. Hydrochloric Acid, 1 M, aqueous: Prepare by diluting 8 mL of concentrated hydrochloric acid (HCl) into a 100 mL volumetric flask with deionized water. This solution is stable for at least one year.

WARNING: Prepare this solution in a fume hood, as HCl fumes are corrosive. Add about 75 mL water to the flask, then carefully add the concentrated hydrochloric acid to water in the flask. Swirl gently to mix, then dilute to volume.

7.13. Copper sulfate, 2% w/v, aqueous. Prepare by dissolving 20 g of copper sulfate ($CuSO_4 \bullet 5H_20$) in a 1000 mL volumetric flask with deionized water.

7.14. Activating the OTCR.

7.14.1. Using a 10cc syringe, flush the cadmium column with 10 mL of DI water, followed by 10 mL of 1M HCl.

7.14.2. Rinse the column with another 10 mL of DI water.

7.14.3. Slowly push 10mL of 2% $CuSO_4$ though the column, and allow to stand for 20 min., followed by flushing an additional 20 mL of 2% $CuSO_4$ through the column.

7.14.4. Attach the cadmium column to the manifold once the ammonia chloride buffer is flowing though the system.

7.14.5. Store cadmium column filled with deionized water.

WARNING: Waste containing copper sulfate may not be poured down the drain, but must be placed in a suitable waste container for proper disposal.

7.15. Preparation of Nitrate and Nitrite Standards.

7.15.1. Use a commercially available certified 1000 mg/L stock nitrate solution. Expiration date is one year from opening, or the manufacturer's expiration date if earlier.

7.15.2. Use a commercially available certified 1000 mg/L stock nitrite solution. Expiration date is one year from opening, or the manufacturer's expiration date if earlier.

7.15.3. Prepare an Intermediate Standard of nitrate plus nitrite at 100 mg/mL as N, by diluting 0.5 mL of each 1000 mg/mL stock solution to a final volume of 10 mL with deionized water. Prepare fresh monthly using volumetric glassware.

7.15.4. Prepare Working Standards monthly from the 100 mg/mL intermediate standards with deionized water into each volumetric flask as follows:

Standard ID	Aliquot (mL)	Final Volume (mL)	Conc. (mg/L as N)
S1	0.05	100	0.05
S2	0.20	100	0.20
S3	0.40	100	0.40
S4	1.0	100	1.0
S5	2.0	100	2.0
Blank	0		Blank

Prepare the working standards monthly, or sooner if there are signs of degradation.

7.15.5. Date and stamp all working standards to ensure integrity and timely disposal. Record standard tracking identifiers on worksheets and logbooks.

7.16. Preparation of nitrocellulose standards.

7.16.1. Nitrocellulose in neat form may be ordered through Theatre Effects, item number FP-11, as flash cotton. It is shipped 'wet' for safety. The nitrogen assay of nitrocellulose must be available or be analyzed by a reputable testing laboratory, such as Marine Science Institute at Santa Barbara. If the assayed nitrocellulose amount is less than 10%, reject the material.

7.16.2. To prepare nitrocellulose stock solution, the flash cotton must be dried, unless the moisture content is known. Dry a small portion at room temperature in a dessicator containing Drierite (or other drying agent) under vacuum for a minimum of several days. *Do not use heat to dry nitrocellulose.*

7.16.3. Prepare a stock solution of nitrocellulose at 500 µg/mL in acetone. Carefully weigh 50 mg of the dried nitrocellulose on a weighing boat, transfer to a 100-mL volumetric flask, and dissolve the nitrocellulose in acetone. **NOTE:** *It may be required to shake the solution for approximately 4 to 6 hours to dissolve the NC into solution.* Store the stock solution in an amber glass bottle at 2 to 6°C. The stock solution is stable for six months.

7.17. Reference Standards

7.17.1. Prepare a reference standard to verify the concentration and identity of the standards and spike solutions above.

7.17.2. If a secondary source is not available, a separate intermediate stock solution may be made from the same neat by another chemist or from a neat with a different vendor or lot number. A pre-prepared solution from an approved source can also be use as a second verification standard.

7.18. Prepare the reference standard as described in Section 7.16 as applicable.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous or soil samples are collected in glass containers, sealed with Teflon-lined screw caps, and stored at 2-6°C until extraction. Other liners such as aluminum foil are acceptable.

8.2. Aqueous or soil samples should be extracted and analyzed within 28 days from collection. This holdtime is recommended only, based on method 353.2. The nitrocellulose standard has demonstrated stability up to 6 months. Any deviations from this holdtime will be noted and evaluated in conjunction with client requirements. The samples must be analyzed 48 hours after the completion of the hydrolysis step.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability 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.

9.2. Quality Control Batch The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike/matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify particular samples for MS/MSD, the batch may contain multiple MS/MSDs. See policy WS-PQA-003 for further definition of the batch. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC Program document (WS-PQA-003) for further details of the batch definition.

9.3. Control Limits

In-house historical control limits must be determined for matrix spikes and laboratory control samples (LCS). Refer to policy WS-PQA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All LCS and MS recoveries (except for dilutions) must be entered into QuantIMS or other database so that accurate historical control limits can be generated. For tests without a separate extraction, matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (WS-PQA-003) for further details of control limits.

9.4. Method Blanks One method blank must be processed with each preparation batch. The method blank consists of reagent water for aqueous samples. The method blank is carried through the entire analytical procedure. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. 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 10% of the measured concentration of that analyte in the associated samples, whichever is higher. Certain programs, such as USACE, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit.

- Re-preparation and reanalysis of any samples with reportable concentrations of analytes less that 10 times the value 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. This must be documented in the NCM program.

- If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all positive results in associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.4.1. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.4.2. Aqueous method blank reporting limit = 0.50 mg/L.

9.4.3. Solid method blank reporting limit = 5.0 mg/kg.

9.5. Laboratory Control Samples (LCS) For each batch of samples, analyze an LCS. The LCS contains the analytes of interest, and must contain the same analytes as the matrix spike. If any analyte is outside established control limits, the system is out of control and corrective action must occur. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur:

- Check calculations.
- Check instrument performance.
- Reanalyze the LCS, and if still outside of control limits.

- Evaluate the data, and/or
- Re-prepare and reanalyze all samples in the QC batch.

9.5.1. Data may be reported with an anomaly in the event that the LCS recoveries are high and the analyte of concern is not detected in field samples.

9.5.2. The analyst should evaluate the anomalous analyte recovery for possible trends.

9.5.3. 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.

9.5.4. 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.

9.5.5. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.6. Matrix Spikes For each QC batch, analyze a matrix spike and matrix spike duplicate (MS/MSD). Spiking compounds and levels are given in Table 1. Compare the percent recovery and relative percent difference (RPD) to those 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.
- If the recovery for any component is outside QC limits for both the MS/MSD 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 an MS/MSD is not possible due to limited sample, then an LCS duplicate may be analyzed if required by the program or client.
- The MS/MSD must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.6.1. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. File an NCM stating that the 4X rule was applied. This NCM must be included in the final report.

9.7. Insufficient Sample If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if precision data is required by the client. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of an LCS pair in place of an MS/MSD must be documented using Clouseau.

9.8. Initial Calibration Verification (ICV) -- When available, a second source standard at or near the mid-point of the calibration is analyzed with the initial calibration curve. Each component of the second source calibration must be within ± 10% of its expected value. Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the Initial Calibration Standards.

9.9. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA and Department Manager.

9.10. 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.

9.11. QC Program Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions

10. CALIBRATION

10.1. The instrument optimization is part of the instrument set-up and pre-programmed internally.

10.2. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

10.3. Instrument calibration is done daily before any samples are analyzed. The calibration curve consists of five (5) standards and a blank. The correlation coefficient of the curve must be at least 0.995 or greater.

Note: Generally, if correlation coefficient <0.9985, the instrument is not performing optimally and should be evaluated further before proceeding.

10.4. The Initial Calibration Verification Standard (ICV) and Initial Calibration Blank (ICB) must be analyzed immediately following the initial calibration and prior to sample analysis.

10.4.1. The ICV/CCV standard concentration is at or near the mid-point concentration of the calibration curve and must be within 10% of the expected value. If not, correct the problem and re-analyze any affected samples back to the last valid CCV. Perform a new multi-point calibration if the ICV fails upon re-analysis.

10.4.2. The reagent blank includes all the reagents added after extraction and is used as the ICB and CCB. The ICB/CCB values must be less than the reporting limit.

10.5. A Continuing Calibration Verification (CCV) and the CCB are analyzed following every ten or fewer samples and at the end of the run. This standard must be within 10% of the expected value.

10.6. Corrective actions and suggestions for repeated failures of calibration verifications and calibration blanks.

10.6.1. Check for contamination in the reagents and standards.

10.6.2. Be sure all reagents were prepared correctly and have not expired.

10.6.3. Check the system for obvious problems, such as plugs, leaks, and worn or degraded pump tubes. Also check for bubbles in the flow cell by pinching the tubing coming out of the flow cell, and then releasing it.

10.6.4. Identify samples that may have deactivated the reduction column. Samples may need to be diluted and/or re-extracted. Consult with the Department Manager before continuing.

10.6.5. Analyze a 1 mg/L nitrite standard and compare to a 1 mg/L nitrate standard. If the results differ by more than 15%, the column has deteriorated and needs to be reactivated with copper sulfate.

10.6.6. If you are unable to locate and solve the problem, consult with your Department Manager for further corrective actions.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity,

chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. This SOP was written for the Alpkem Flow Solution IV system and must be followed in order to work with the existing instrument software. Any deviations may require modifications to the instrument program. Also, any changes in the program may require alterations in this SOP. Comparable instrumentation may be used if data quality objectives and method performance criteria can be met.

11.3. Aqueous Sample Preparation

11.3.1. Obtain samples from sample receiving. Allow the samples to equilibrate to room temperature.

11.3.2. Prepare a method blank (MB) for the batch by measuring 100 mL of reagent water into a mixing cylinder.

11.3.3. Prepare the LCS (and LCSD, if needed), by spiking a clean mixing cylinder with 1000 µL of the 500 µg/mL nitrocellulose stock solution to yield 2.0 ppm NC (or 0.20 µg/mL N), assuming the assayed NC amount is 10%. Air dry cylinder, then add 100 mL of reagent water. Mix the contents well by shaking vigorously and/or sonicate in a water bath for approximately 5 minutes.

***NOTE**: Assayed nitrocellulose may vary, therefore the preparation concentration will vary.*

11.3.4. Prepare cylinders for the MS and MSD by spiking two clean cylinders with 1000 µL of the 500 µg/mL nitrocellulose stock solution. Allow the cylinders to air dry prior to adding sample aliquots (as in Section 11.4.3, above).

11.3.5. For each field sample, shake the sample well and quickly measure 100 mL into a 100-mL mixing cylinder or graduated cylinder.

11.3.6. Mix the contents of the MS and MSD cylinders well by shaking vigorously and/or sonicating in a water bath for approximately 5 minutes.

11.3.7. Add 50 µL of Brij-35 solution to each sample, MB, LCS/LCSD, and MS/MSD. Mix the contents well.

11.3.8. Set up the filtering apparatus with a Nuclepore membrane filter.

Note: The Nuclepore membrane filter is very delicate. Handle with care when setting up the apparatus. Do not allow the membrane filter to go dry during the filtration.

11.3.8.1. Mix the water sample well and pour it into the filter assembly.

11.3.8.2. Turn the vacuum on low, and allow the sample to filter through the assembly.

11.3.8.3. Rinse the sample container with 15 mL of reagent water. Filter. Repeat two more times.

11.3.8.4. Wash the filter with 100 mL of reagent water, and stop the flow just before drying.

11.3.9. Carefully remove the filter with tweezers and transfer it to a 250 mL glass beaker. ***The Nuclepore membrane filter is very delicate. Handle with care.*** Rinse the inner surface of the filter holder with approximately 20 mL of acetone and add to the beaker.

11.3.10. Cover the beaker with a watch glass. Let the filter stand submerged in acetone for 1 hour, swirl the solution occasionally.

11.3.11. Transfer the acetone extract to a 50 mL centrifuge tube, rinse the filter and beaker with two 15 mL portions of acetone, and transfer each portion to the centrifuge tube. Do not exceed the 50 mL capacity of the tube.

11.3.12. Store the extracts in the refrigerator at 2-6°C until ready for N-Evap and hydrolysis.

11.4. Soil/Solid Sample Preparation

11.4.1. Allow the soil sample to equilibrate to room temperature.

11.4.2. Prepare 50 mL centrifuge tubes for the spiked samples – LCS (/LCSD) and MS/MSD. Add 1.0 mL of the 500 µg/mL solution into an empty tube. Allow the solution to dry under a gentle stream of nitrogen.

11.4.3. Weigh 10 g of soil sample into a 50 mL centrifuge tube.

11.4.3.1. For the MB and LCS/LCSD, use crushed, ground Ottawa sand as the control matrix.

11.4.4. Wash the samples and QC aliquots with two 20 mL aliquots of 1:1 methanol-water solution. Shake the tube vigorously for approximately 10 seconds and on the platform shaker for 10 minutes at approximately 240 rpm. Centrifuge at approximately 3500 rpm for 20 minutes, decant carefully and discard the

washing solution.

Note: If the solids (particularly solids as wipes) are too loose following centrifuging, it may be necessary to add crushed, ground Ottawa sand to aid the packing of the solids, which improves the recovery of nitrocellulose. A 10 g aliquot of sand should

be sufficient.

11.4.5. Extract the washed residue by adding 15 mL of acetone, shake the tube vigorously for 10 seconds, and allow the contents to stand for 1 hour before proceeding.

11.4.6. Shake the samples on the platform shaker for 10 minutes at approximately 240 rpm, centrifuge at approximately 3500 rpm for 10 minutes, and decant the acetone into a 50-mL centrifuge tube.

11.4.7. Extract the soil two more times with 15 mL portions of acetone, shake vigorously for 10 seconds and on the platform shaker for 10 minutes at approximately 240 rpm. Centrifuge at approximately 3500 rpm for 10 minutes, and pool the acetone in the 50 mL centrifuge tube.

11.4.8. Store the extracts in the refrigerator at 2-6°C until ready for N-Evap and hydrolysis.

11.5. N-Evap and Hydrolysis of Nitrocellulose to nitrate-nitrite

Note: The holding time for the extraction and analysis of samples is 28 days from sampling. Once the extracts are hydrolyzed, they must be analyzed within 48 hours. Coordinate with the General Chemistry Department Manager to ensure that samples can be analyzed within the required holding time.

11.5.1. Set the N-Evap water bath at 50 - 55ºC.

11.5.2. Add 2.0 mL of 1N NaOH to the acetone extracts in the 50 mL centrifuge tubes, and mix the contents well. *Do not start this step unless the samples can be analyzed within 48 hours after the completion of the hydrolysis step.*

11.5.3. Evaporate the acetone extracts under a gentle stream of nitrogen on the N-Evap in a water bath at 50 ± 5ºC. When the volume is reduced to 1 or 2 mL, turn off the nitrogen, and continue heating the extracts for an additional 1 hour.

Note: The chemical reaction step is now completed. Document the time of completion on the benchsheet. Analyze within 48 hours.

11.5.4. Bring each sample up to approximately 10 mL with reagent water. Mix the solution well, and adjust the pH to between 6 and 8 with 2N sulfuric acid

and/or 1N sodium hydroxide. Monitor the pH adjustment using a meter.

11.5.5. Adjust the final volume to 40 mL with reagent water, and mix well. The preparation factor (V_f/W_x) is 40 mL/100 mL for aqueous samples, and 40 mL/10 g for solid samples. (V_f = final extract volume, W_x = initial sample weight or volume)

11.5.6. Filter an aliquot of solid extract through a Whatman GD/X syringe filter on a Millipore Millex-LCR filter assembly into a 16-cc test tube, and submit the filtered extracts for analysis. Filter an aliquot of aqueous extract through a Millipore Millex-LCR filter assembly.

11.5.6.1. The excess solution in the centrifuge tubes may be saved until the analysis of nitrate plus nitrite is complete. Many times, solid extracts are very difficult to filter through Millipore Millex-LCR filter without the Whatman GD/X.

11.6. The extracts are stored at 2-6°C until ready for analysis of nitrate/nitrite. Notify the Department Manager and/or the analyst the NC samples are ready for analysis. Submit the extraction paperwork to the analyst.

11.7. Instrument Start-up

11.7.1. The instrument is to be set up and operated in accordance to the manufacturer's instructions. Install the nitrite and nitrate/nitrite manifolds according to manufacturer's instructions. Nitrite may be analyzed alone if the reduction column is bypassed. The carrier will be deionized water for both preserved and unpreserved samples.

11.7.2. Inspect the manifold for proper connections. Make sure that there is sufficient volume for all reagents for the entire run. All reagents and standards must be within the expiration dates.

11.7.3. Prepare working standards as stated in Section 7.15.

11.7.4. Turn on the power to the system unit, pump, autosampler, and the software system.

11.7.5. Connect all lines to the rinse container containing deionized water and pump through the system for approximately 5 minutes. This step is recommended to ensure that the system does not retain any contaminants prior to starting the analysis.

11.7.6. Connect the reagent lines to their proper containers, and fasten down the pump tube clamps. Make sure the reagent tubes are in good working

condition.

11.7.7. Pump reagents through all lines until all air bubbles have been flushed from the system. No air bubbles should be present after the debubbler.

11.7.8. Create a sample table, and save under the proper test folder. Add a start sequence command to initiate within 30 seconds of machine startup to insure that the autosampler is flushed with rinseate.

11.7.9. Bring the conditioned cadmium column (Section 7.14) on line by turning off the pump and connecting the column into the manifold. See manufacture notes for exact location on the manifold.

11.7.10. Load the NITRATE+NITRITE method into the computer, and press the play button. You will see the NO_2+NO_3 and NO_2 backgrounds on the computer screen.

11.7.10.1. Use the NO_2 method in the computer if NO_2 singly or NO_3 singly is to be determined.

11.7.11. If the flow system is working properly, the average baselines should be stable in about two minutes.

11.8. Analysis

11.8.1. Fill the sample tubes with the working standards and samples in accordance with the sample table.

11.8.2. Start the analysis by pressing the "fast forward" button. The instrument will analyze the standards and proceed with the samples. Monitor the run to insure that the correlation coefficient is > 0.995. If the calibration curve is not acceptable, stop the run, locate and correct the problem, and then recalibrate. The instrument should be monitored periodically to make sure that ICV/CCVs and cadmium column checks pass NO_2^- and NO_3^- limits of ±15% of the standard level. The calibration statistics and raw data should be printed at the end of the run.

11.8.3. Check the run for off scale samples. Dilute and enter into the end of the run sequence if needed. Remember that all samples and dilutions must be bracketed by passing ICV/CCV and ICB/CCB sets.

11.8.4. If additional samples are to be analyzed, reload the autosampler and enter the new table information. Since the instrument is already calibrated, you may save and load the calibration directly to the analysis of the new samples using different batch identification.

11.9. Shut Down

11.9.1. Take the cadmium column off line by disconnecting the column while the pump is not pumping. If the column is still in acceptable condition, prior to reconnecting the column, forcibly push approximately 10 mL of reagent water through the column using a 10 mL luer-lock plastic syringe. .

Warning: Exercise caution when using syringes. Application of excessive force has, upon occasion, caused a rupture during the process.

11.9.2. Place all reagent lines in deionized water and pump at normal speed for at least 5 minutes. Remove the lines from the water and pump the system dry.

11.9.3. Ensure that all reagent and H_2O lines are free of liquid.

11.9.4. Turn off the units and unfasten the pump tube cassettes from the pump. Release the tension levers.

11.9.5. Clean up the work area and replace any reagents, which have been depleted.

WASTE GENERATED MUST BE COLLECTED IN SPECIAL WASTE STREAM CONTAINERS AND STORED PROPERLY.

12. CALCULATIONS/DATA REDUCTION

12.1. For nitrocellulose (NC) conversion, use only the nitrate/nitrite (NO_3+NO_2) concentration.

12.2. The amount of NC found in the sample is derived from the amount of N (nitrate/nitrite) measured, using the following formula

Formula 1 NC = (N / Fp) x (Vf / Wx)

Where: NC = nitrocellulose concentration in µg/mL or µg/g (ppm). N = nitrate plus nitrite concentration in µg/ml. Vf = final extract volume (mL). Wx = Sample mass (grams for soil) or volume (mL for aqueous). Fp = Percent nitrogen factor in nitrocellulose (assay, such as 0.10 for 10% N). Fp may vary depending on the lot.

12.3. If the prep factor Pf is already taken into consideration during the analysis, where:
Formula 2 Pf = Vf / Wx

then:

Formula 3 **NC = N / Fp**

12.4. For % recovery and % RPD calculations, see policy WS-PQA-003.

12.5. The reporting limit for nitrocellulose is dependent on the reporting limit of nitrate/nitrite. The reporting limit for nitrate/nitrite is 0.05 mg/L (ppm). With the final aqueous extract concentration/dilution factor at 100 mL/40 mL, the reporting limit is calculated by using formula 1, assuming the assay of NC is 10% N, and inserting a RL buffer factor of 2.5.

$$\mathbf{NC} = (N / Fp) \times (Vf / Wx) \times 2.5$$

$$NC = ((0.05 \text{ mg/L})/0.100) \times (40 \text{ mL}/100 \text{ mL}) \times 2.5 = 0.50 \text{ µg/mL NC}$$

12.6. With the final soil extract concentration/dilution factor at 10g/40 mL, the reporting limit is calculated using formula 1, assuming the assay of NC is 10% N;

13. METHOD PERFORMANCE

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

13.2. Method Detection Limit: The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration: The 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.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. 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.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Water, hydrochloric acid solution, cupric sulfate solution and EDTA stock buffer solution used to rinse the OTCR. This is collected in a one liter bottle until full or for no more than one year, then transferred to the blue plastic metals acidic waste drum. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Miscellaneous disposable glassware, Anodisc membrane filters, disposable centrifuge tubes, filter discs, chemical resistant gloves, bench paper and similar materials that may or may not be contaminated/hazardous. Place contaminated materials into a contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Extracted soil samples in centrifuge tubes, contaminated with methanol and acetone. These are transferred to the waste disposal area for lab pack and shipment.

15.4. 1:1 water:methanol solution used to wash soil samples prior to extraction. Decant the washing solution into the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.5. Acidic waste from the auto-analyzer. This is collected in a 1-gallon plastic coated carboy. When the carboy is full, or after no more than one year, move the carboy to the waste collection area, where it will be lab-packed into a drum for shipment.

15.6. Unused sample extract in centrifuge tubes, which is retained until after analysis is completed. These are transferred to the waste collection area, where they will be lab-packed into a drum for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. "Quality Control Program", Policy WS-PQA-003.

16.2. "Determination of Nitrocellulose in Soil by Autoanalyzer", Leo O'Shea, Raytheon Laboratories, Boothwyn, PA, August 1997.

16.3. "The Determination of Nitrocellulose in Water by Colorimetric Autoanalyzer", DataChem Laboratories, Version 1.0, January 15, 1991.

16.4. "The Determination of Nitrocellulose in Soil by Colorimetric Autoanalyzer", DataChem Laboratories, Version 1.0, January 8, 1991.

16.5. EPA MCAWW 353.2 Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium reduction).

16.6. Standard Method 4500-NO_3 F. Automated Cadmium Reduction Method, 18th Edition 1992.

16.7. Method source: OI Analytical Document #319437 equivalent to EPA method 353.2.

17. METHOD MODIFICATIONS

17.1. Deviations from reference methods.

17.1.1. The hold time for samples to be analyzed by this method is 28 days, and 48 hours after the hydrolysis step. The O'Shea method suggests the hold time is 40 days and should be analyzed within 24 hours after the chemical reaction (hydrolysis step), whereas, the method suggests that the samples be analyzed within 28 days. The holding time selected by this laboratory is defined by the EPA for nitrate/nitrite, method 353.2.

17.1.2. An Alpkem Flow Solution IV is used in place of a Technicon Autoanalyzer II. Both instruments are compatible and designed for nitrate plus nitrite analysis.

17.1.3. Linear range is designed based on the manufacturer's recommendation of 0.05 to 2.0 mg/L instead of 0.05 to 10 mg/L.

17.1.4. Absorbance is at 540 nm instead of 550 nm.

17.1.5. Sodium hydroxide is used in place of ammonium hydroxide for pH

adjustment. There is no impact on the chemical reaction.

17.1.6. Sulfuric acid is used in place of hydrochloric acid for pH adjustment. There is no impact on the chemical reaction.

17.1.7. Reagent preparations are performed according to the manufacturer's instructions and may differ from the method.

17.1.8. Shelf life for calibration standards is changed to monthly based on the inhouse stability study performed for these analytes.

17.1.9. Preservation with chloroform is omitted. Stock standards are stable for at least 6 months without preserving with chloroform.

17.1.10. This SOP is dedicated for the preparation and analysis of nitrocellulose as nitrate/nitrite. The methods do not clearly define the standard preparation, calibration, and acceptance criteria. The analytical protocols for Nitrate plus Nitrite, method (353.2) are adopted for nitrocellulose.

17.2. Deviations from reference method; O'Shea method.

17.2.1. The nitrocellulose stock solution is valid for up to six months rather than 1 week.

17.2.2. Nanopure water is used instead of deionized water or ASTM Type I water.

17.2.3. When the soil sample is washed and extracted, it is vortexed for 10 seconds and shaken on the platform shaker for 10 minutes, rather than vortexed 15 seconds without any further shaking.

17.2.4. The final extract volume for water and for soil is 40.0 mL instead of 100 mL to achieve a lower reporting limit; 0.50 mg/L NC for aqueous, and 5.0 mg/kg NC for soil.

17.3. Deviations from reference method

17.3.1. To fortify the aqueous samples, the spiking solution is added to a clean mixing cylinder and allowed to dry before adding the 100 mL water sample or the controlled water sample prior to extraction.

17.3.2. Soak the residues on the Anodisc membrane filter in acetone for 1 hour instead of 10 minutes.

17.3.3. A 10 gram soil sample is washed and extracted instead of a 0.5 gram, and washed with methanol instead of water.

17.3.4. After vortexing for 10 seconds, the sample is also shaken on the platform shaker for 10 minutes.

17.3.5. The soil sample is soaked in acetone for an hour or longer rather than 90 minutes.

17.3.6. 2 mL rather than 1 mL of the 1N sodium hydroxide is added to the acetone extracts, and after the acetone dissipates the basic extracts are heated for an additional 1 hour to complete the chemical reaction (hydrolysis of nitrocellulose to nitrate plus nitrite)

17.3.7. The final volume for water is adjusted to 40.0 mL with nanopure water after the pH adjustment to between 6 and 8, instead of transferring to 100-mL volumetric flask, adjusting the volume to mark with ASTM Type I water, and adjusting the pH to between 7 and 9.

18. ATTACHMENTS

18.1. Table 1- Aqueous and Soil LCS and MS/MSD Spike Levels

18.2. Flow Chart 1- Preparation of Samples

18.3. Flow Chart 2- Nitrate Plus Nitrite by Alpkem Flow Solution IV Autoanalyzer

19. REVISION HISTORY

19.1. WS-WC-0050, Revision 3.5, Effective 11/5/2010

19.1.1. Changed Section 11.3.3 spiking level volume from 400 µl to 1000 µl.

19.1.2. Changed Section 11.3.4 spiking level volume from 400 µl to 1000 µl.

19.1.3. Changed Table 1 aqueous LCS and MS/MSD spiking level from 2.0 mg/L to 5.0 mg/L.

19.2. WS-WC-0050, Revision 3.4, Effective 05/03/2010

19.2.1. Editorial revisions – copied applicable Sections from SOP WS-WC-036 to utilize language for identical procedures.

19.2.2. Section 7.15.4 – Updated table with correct standard concentrations.

19.3. WS-WC-0050, Revision 3.3, Effective 11/5/2009

19.3.1. Section 6.11.6, added, "… (Version 4.0 or equivalent).

19.3.2. Added note following Section 7 header: All reagents must be ACS reagent grade or better unless otherwise specified.

19.3.3. Section 7.5, added, "Refer to SOP WS-QA-0014, "Monitoring of Reagent-Grade Laboratory Water" for further details.

19.4. WS-WC-0050, Revision 3.2, Effective 09/04/2009

19.4.1. Added Section 1.4: "When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

19.4.2. Inserted Section 10.2: "For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)"."

19.5. WS-WC-0050, Revision 3.1, Effective 05/01/2009

19.5.1. Section 7.1 - inserted "When available, pre-made, commercially prepared reagents are purchased."

19.5.2. Section 7.13.4 - Deleted 0.025 mg/L standard from table.

19.5.3. Section 12.5 and Section 17.2.4 - inserted buffer factor of 2.5 which changed RL from 0.2 and 2.0 to 0.5 and 5.0 for aqueous and soil samples respectively.

19.6. WS-WC-0050, Revision 3, Effective 2/29/2008

19.6.1. This SOP format has been updated to the new TestAmerica format.

19.7. SAC-WC-0050, Revision 2.2, Effective 4/13/07

19.7.1. Updated to reflect current reporting limit (Section 9.4.3)

19.7.2. Updated to reflect current practice. Connect the nitrate/nitrite buffer line to ammonia chloride buffer. Pump the reagents through all lines until a stable baseline is obtained.

19.8. SAC-WC-0050, Revision 2.1, Effective 1/11/05

19.8.1. Updated equiptment list(Section 6.2, 11.4.8, 11.4.9, 15.2, 17.3.2)

19.9. SAC-WC-0050, Revision 2, Effective 8/12/03

19.9.1. Fixed grammatical errors, improved flow of SOP, updated page formatting.

19.9.2. Updated safety, pollution, and waste disposal sections in accordance with recent EH&S requirements.

19.10. SAC-WC-0050, Revision 1, Revised 6/28/01

TABLE 1

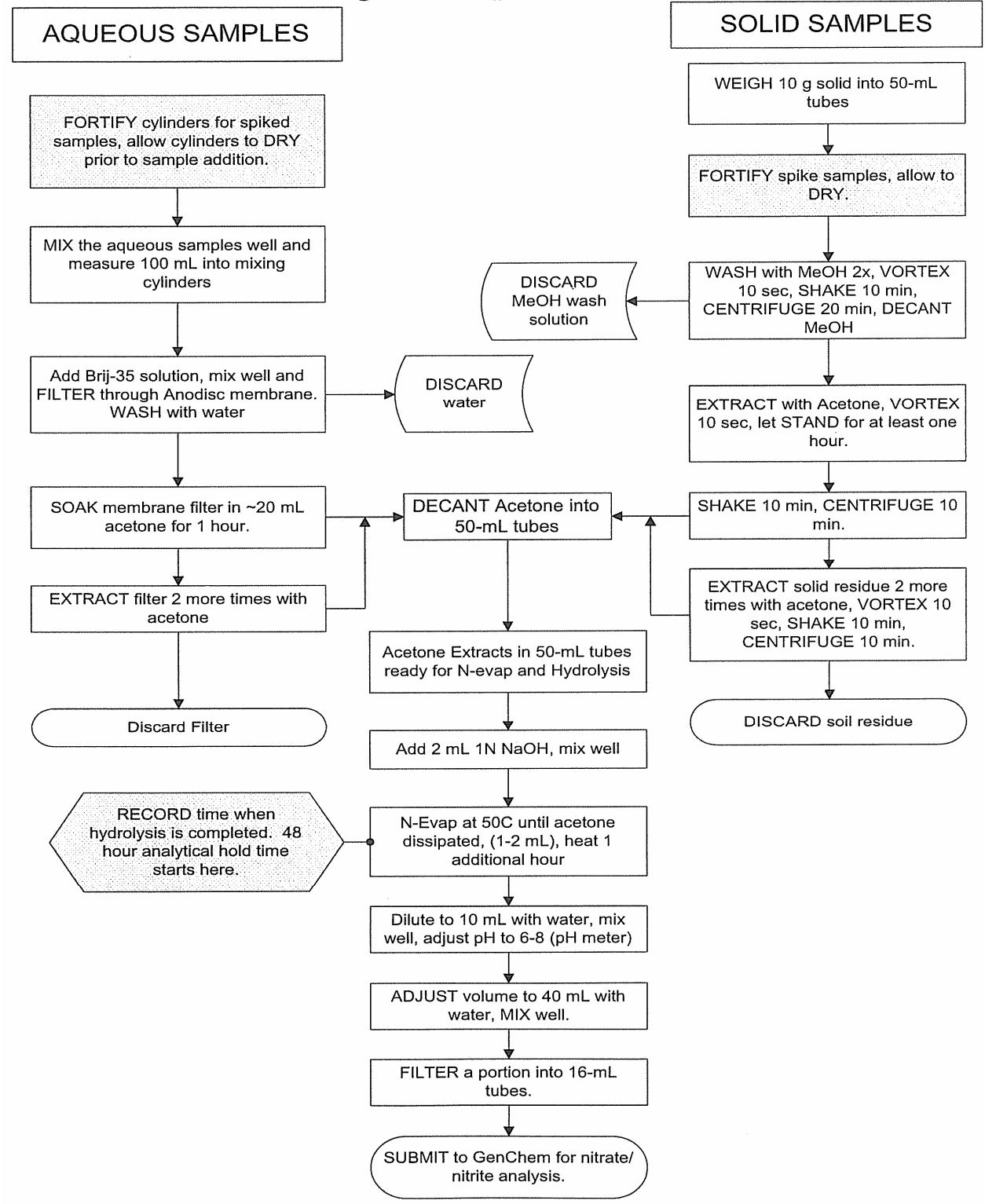
AQUEOUS - LCS AND MS/MSD

Test Components	NC Spike Level, mg/L
Nitrocellulose	5.0

SOIL - LCS AND MS/MSD

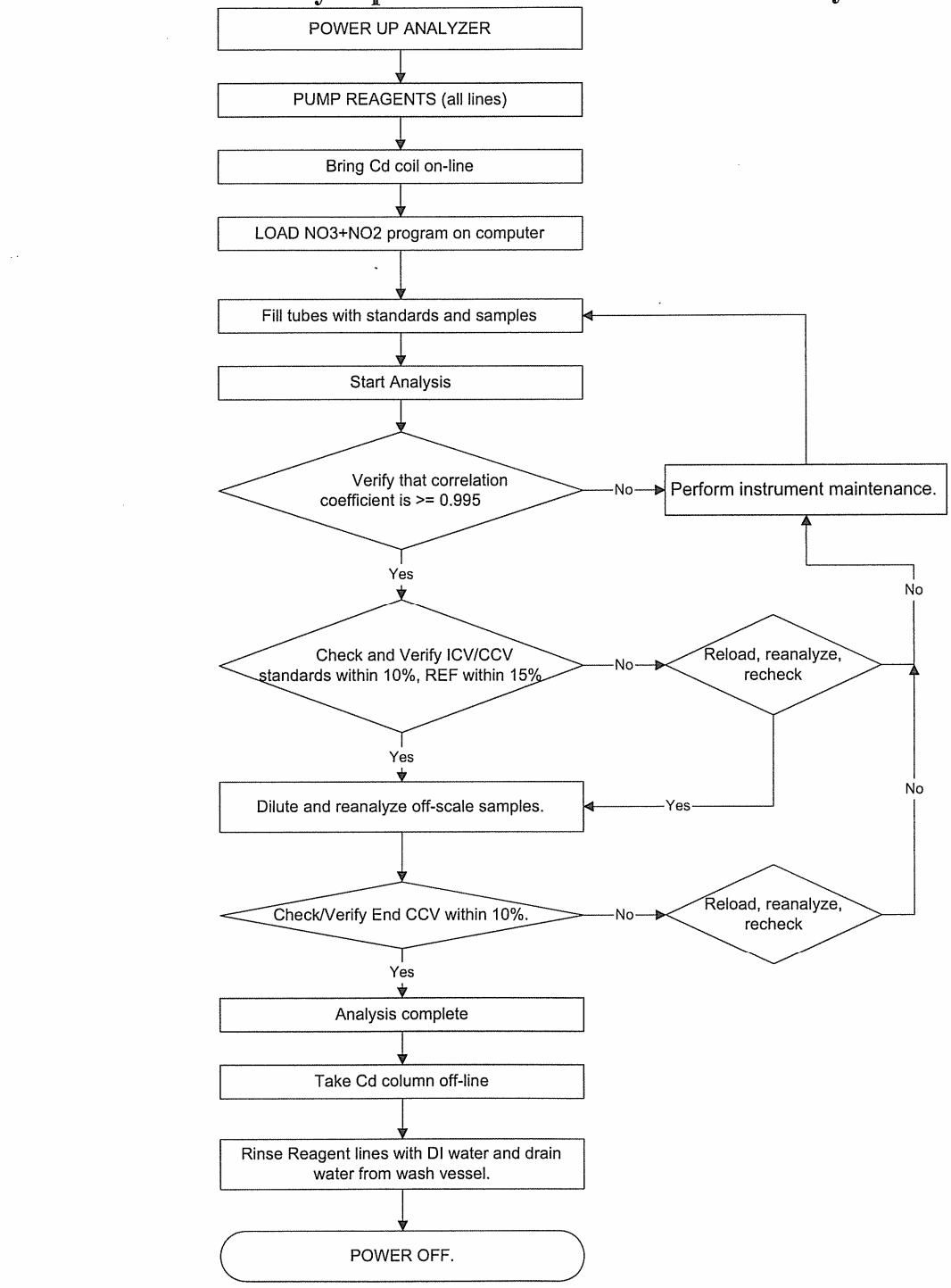
Test Components	NC Spike Level, mg/Kg
Nitrocellulose	50

Flow Chart 1
Flow Diagram- Preparation of Samples



Company Confidential & Proprietary

Flow Chart 2
Nitrate Plus Nitrite By Alpkem Flow Solution IV Autoanalyzer



Company Confidential & Proprietary

Appendix D

Laboratory Certifications

Legend Technical Services, St. Paul, MN
TestAmerica, West Sacramento, CA

Minnesota Department of Health

Environmental Laboratory Accreditation Program

Issues accreditation to

State Laboratory ID: 027-123-295

Legend Technical Services, Inc.

88 Empire Drive

St Paul, MN 55103

for fields of testing listed on the laboratory's accompanying Scope of Certification in accordance with the provisions in Minnesota Laws and Rules.

Continued accreditation is contingent upon successful on-going compliance with Minnesota Statutes 144.97 to 144.98, 2003 NELAC Standard and applicable Minnesota Rules 4740.2010 to 4740.2120. The laboratory's Scope of Certification cites the specific programs, methods, analytes and matrices (i.e. fields of testing) for which MDH issues this accreditation.

This certificate is valid proof of accreditation only when associated with its accompanying Scope of Certification.

The Scope of Certification and reports of on-site inspections are on file at the Minnesota Department of Health, 601 Robert Street North, Saint Paul, Minnesota. Customers may verify the laboratory's accreditation status in Minnesota by contacting MN-ELAP at (651) 201-5200.

Effective Date: 12/17/2010

Expires: 12/31/2011

Susan R. Wyatt

Susan R. Wyatt, MN-ELAP Supervisor

Certificate Number: 173652

UMP013030

Environmental Laboratory Certification Program
Scope of Certification
Certified Minnesota Environmental Laboratories

THIS LISTING OF CERTIFIED FIELDS OF TESTING MUST BE ACCOMPANIED BY CERTIFICATE NUMBER: 173652

State Laboratory ID: 027-123-295 EPA Lab Code: MN00908 Expiration Date: 12/31/2011
Issue Date: 12/17/2010

Legend Technical Services, Inc.
88 Empire Drive
St Paul, MN 55103

Clean Water Program

EPA 200.7

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.7	Aluminum	NPW	MN	
CWP	EPA 200.7	Antimony	NPW	MN	
CWP	EPA 200.7	Arsenic	NPW	MN	
CWP	EPA 200.7	Barium	NPW	MN	
CWP	EPA 200.7	Beryllium	NPW	MN	
CWP	EPA 200.7	Boron	NPW	MN	
CWP	EPA 200.7	Cadmium	NPW	MN	
CWP	EPA 200.7	Calcium	NPW	MN	
CWP	EPA 200.7	Cobalt	NPW	MN	
CWP	EPA 200.7	Copper	NPW	MN	
CWP	EPA 200.7	Iron	NPW	MN	
CWP	EPA 200.7	Lead	NPW	MN	
CWP	EPA 200.7	Magnesium	NPW	MN	
CWP	EPA 200.7	Manganese	NPW	MN	
CWP	EPA 200.7	Molybdenum	NPW	MN	
CWP	EPA 200.7	Nickel	NPW	MN	
CWP	EPA 200.7	Potassium	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.7	Selenium	NPW	MN	
CWP	EPA 200.7	Silica-dissolved	NPW	MN	
CWP	EPA 200.7	Silver	NPW	MN	
CWP	EPA 200.7	Sodium	NPW	MN	
CWP	EPA 200.7	Thallium	NPW	MN	
CWP	EPA 200.7	Tin	NPW	MN	
CWP	EPA 200.7	Total chromium	NPW	MN	
CWP	EPA 200.7	Vanadium	NPW	MN	
CWP	EPA 200.7	Zinc	NPW	MN	

EPA 200.7

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.7	Total hardness as CaCO3	NPW	MN	

EPA 200.8

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.8	Aluminum	NPW	MN	
CWP	EPA 200.8	Antimony	NPW	MN	
CWP	EPA 200.8	Arsenic	NPW	MN	
CWP	EPA 200.8	Barium	NPW	MN	
CWP	EPA 200.8	Beryllium	NPW	MN	
CWP	EPA 200.8	Cadmium	NPW	MN	
CWP	EPA 200.8	Cobalt	NPW	MN	
CWP	EPA 200.8	Copper	NPW	MN	
CWP	EPA 200.8	Lead	NPW	MN	
CWP	EPA 200.8	Manganese	NPW	MN	
CWP	EPA 200.8	Molybdenum	NPW	MN	
CWP	EPA 200.8	Nickel	NPW	MN	
CWP	EPA 200.8	Selenium	NPW	MN	
CWP	EPA 200.8	Silver	NPW	MN	
CWP	EPA 200.8	Thallium	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.8	Total chromium	NPW	MN	
CWP	EPA 200.8	Vanadium	NPW	MN	
CWP	EPA 200.8	Zinc	NPW	MN	

EPA 245.1

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 245.1	Mercury	NPW	MN	

EPA 608

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 608	4,4'-DDD	NPW	MN	
CWP	EPA 608	4,4'-DDE	NPW	MN	
CWP	EPA 608	4,4'-DDT	NPW	MN	
CWP	EPA 608	Aldrin	NPW	MN	
CWP	EPA 608	alpha-BHC (alpha-Hexachlorocyclohexane)	NPW	MN	
CWP	EPA 608	Aroclor-1016 (PCB-1016)	NPW	MN	
CWP	EPA 608	Aroclor-1221 (PCB-1221)	NPW	MN	
CWP	EPA 608	Aroclor-1232 (PCB-1232)	NPW	MN	
CWP	EPA 608	Aroclor-1242 (PCB-1242)	NPW	MN	
CWP	EPA 608	Aroclor-1248 (PCB-1248)	NPW	MN	
CWP	EPA 608	Aroclor-1254 (PCB-1254)	NPW	MN	
CWP	EPA 608	Aroclor-1260 (PCB-1260)	NPW	MN	
CWP	EPA 608	beta-BHC (beta-Hexachlorocyclohexane)	NPW	MN	
CWP	EPA 608	Chlordane (tech.)	NPW	MN	
CWP	EPA 608	delta-BHC	NPW	MN	
CWP	EPA 608	Dieldrin	NPW	MN	
CWP	EPA 608	Endosulfan I	NPW	MN	
CWP	EPA 608	Endosulfan II	NPW	MN	
CWP	EPA 608	Endosulfan sulfate	NPW	MN	
CWP	EPA 608	Endrin	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 608	Endrin aldehyde	NPW	MN	
CWP	EPA 608	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NPW	MN	
CWP	EPA 608	Heptachlor	NPW	MN	
CWP	EPA 608	Heptachlor epoxide	NPW	MN	
CWP	EPA 608	Toxaphene (Chlorinated camphene)	NPW	MN	

EPA 624

Preparation Techniques: Purge and trap;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 624	1,1,1-Trichloroethane	NPW	MN	
CWP	EPA 624	1,1,2,2-Tetrachloroethane	NPW	MN	
CWP	EPA 624	1,1,2-Trichloroethane	NPW	MN	
CWP	EPA 624	1,1-Dichloroethane	NPW	MN	
CWP	EPA 624	1,1-Dichloroethylene	NPW	MN	
CWP	EPA 624	1,2-Dichlorobenzene	NPW	MN	
CWP	EPA 624	1,2-Dichloroethane (Ethylene dichloride)	NPW	MN	
CWP	EPA 624	1,2-Dichloropropane	NPW	MN	
CWP	EPA 624	1,3-Dichlorobenzene	NPW	MN	
CWP	EPA 624	1,4-Dichlorobenzene	NPW	MN	
CWP	EPA 624	Benzene	NPW	MN	
CWP	EPA 624	Bromodichloromethane	NPW	MN	
CWP	EPA 624	Bromoform	NPW	MN	
CWP	EPA 624	Carbon tetrachloride	NPW	MN	
CWP	EPA 624	Chlorobenzene	NPW	MN	
CWP	EPA 624	Chlorodibromomethane	NPW	MN	
CWP	EPA 624	Chloroethane (Ethyl chloride)	NPW	MN	
CWP	EPA 624	Chloroform	NPW	MN	
CWP	EPA 624	cis-1,3-Dichloropropene	NPW	MN	
CWP	EPA 624	Ethylbenzene	NPW	MN	
CWP	EPA 624	Methyl bromide (Bromomethane)	NPW	MN	
CWP	EPA 624	Methyl chloride (Chloromethane)	NPW	MN	
CWP	EPA 624	Methylene chloride (Dichloromethane)	NPW	MN	
CWP	EPA 624	Tetrachloroethylene (Perchloroethylene)	NPW	MN	
CWP	EPA 624	Toluene	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 624	trans-1,2-Dichloroethylene	NPW	MN	
CWP	EPA 624	trans-1,3-Dichloropropylene	NPW	MN	
CWP	EPA 624	Trichloroethene (Trichloroethylene)	NPW	MN	
CWP	EPA 624	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	NPW	MN	
CWP	EPA 624	Vinyl chloride	NPW	MN	

EPA 625

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 625	1,2,4-Trichlorobenzene	NPW	MN	
CWP	EPA 625	2,4,6-Trichlorophenol	NPW	MN	
CWP	EPA 625	2,4-Dichlorophenol	NPW	MN	
CWP	EPA 625	2,4-Dimethylphenol	NPW	MN	
CWP	EPA 625	2,4-Dinitrophenol	NPW	MN	
CWP	EPA 625	2,4-Dinitrotoluene (2,4-DNT)	NPW	MN	
CWP	EPA 625	2,6-Dinitrotoluene (2,6-DNT)	NPW	MN	
CWP	EPA 625	2-Chloronaphthalene	NPW	MN	
CWP	EPA 625	2-Chlorophenol	NPW	MN	
CWP	EPA 625	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NPW	MN	
CWP	EPA 625	2-Nitrophenol	NPW	MN	
CWP	EPA 625	3,3'-Dichlorobenzidine	NPW	MN	
CWP	EPA 625	4-Bromophenyl phenyl ether	NPW	MN	
CWP	EPA 625	4-Chloro-3-methylphenol	NPW	MN	
CWP	EPA 625	4-Chlorophenyl phenylether	NPW	MN	
CWP	EPA 625	4-Nitrophenol	NPW	MN	
CWP	EPA 625	Acenaphthene	NPW	MN	
CWP	EPA 625	Acenaphthylene	NPW	MN	
CWP	EPA 625	Anthracene	NPW	MN	
CWP	EPA 625	Benzidine	NPW	MN	
CWP	EPA 625	Benzo(a)anthracene	NPW	MN	
CWP	EPA 625	Benzo(a)pyrene	NPW	MN	
CWP	EPA 625	Benzo(g,h,i)perylene	NPW	MN	
CWP	EPA 625	Benzo(k)fluoranthene	NPW	MN	
CWP	EPA 625	Benzo[b]fluoranthene	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 625	bis(2-Chloroethoxy)methane	NPW	MN	
CWP	EPA 625	bis(2-Chloroethyl) ether	NPW	MN	
CWP	EPA 625	bis(2-Chloroisopropyl) ether	NPW	MN	
CWP	EPA 625	Butyl benzyl phthalate	NPW	MN	
CWP	EPA 625	Chrysene	NPW	MN	
CWP	EPA 625	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	NPW	MN	
CWP	EPA 625	Di-n-butyl phthalate	NPW	MN	
CWP	EPA 625	Di-n-octyl phthalate	NPW	MN	
CWP	EPA 625	Dibenz(a,h) anthracene	NPW	MN	
CWP	EPA 625	Diethyl phthalate	NPW	MN	
CWP	EPA 625	Dimethyl phthalate	NPW	MN	
CWP	EPA 625	Fluoranthene	NPW	MN	
CWP	EPA 625	Fluorene	NPW	MN	
CWP	EPA 625	Hexachlorobenzene	NPW	MN	
CWP	EPA 625	Hexachlorobutadiene	NPW	MN	
CWP	EPA 625	Hexachlorocyclopentadiene	NPW	MN	
CWP	EPA 625	Hexachloroethane	NPW	MN	
CWP	EPA 625	Indeno(1,2,3-cd) pyrene	NPW	MN	
CWP	EPA 625	Isophorone	NPW	MN	
CWP	EPA 625	n-Nitrosodi-n-propylamine	NPW	MN	
CWP	EPA 625	n-Nitrosodimethylamine	NPW	MN	
CWP	EPA 625	n-Nitrosodiphenylamine	NPW	MN	
CWP	EPA 625	Naphthalene	NPW	MN	
CWP	EPA 625	Nitrobenzene	NPW	MN	
CWP	EPA 625	Pentachlorophenol	NPW	MN	
CWP	EPA 625	Phenanthrene	NPW	MN	
CWP	EPA 625	Phenol	NPW	MN	
CWP	EPA 625	Pyrene	NPW	MN	

SM 2320 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2320 B-97	Alkalinity as CaCO3	NPW	MN	

SM 2340 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2340 B-97	Total hardness as CaCO3	NPW	MN	

SM 2540 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 B-97	Residue-total	NPW	MN	

SM 2540 C-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 C-97	Residue-filterable (TDS)	NPW	MN	

SM 2540 D-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 D-97	Residue-nonfilterable (TSS)	NPW	MN	

SM 3500-Cr B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 3500-Cr B-97	Chromium VI	NPW	MN	

SM 4500-H+ B-96

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-H+ B-96	pH	NPW	MN	

SM 4500-S2¯ D-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-S2¯ D-97	Sulfide	NPW	MN	

SM 5220 D-97

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 5220 D-97	Chemical oxygen demand	NPW	MN	

SM 5310 C-96

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 5310 C-96	Total Organic Carbon	NPW	MN	

Resource Conservation Recovery Program

EPA 600/4-81-045

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 600/4-81-045	PCBs	SCM	MN	

EPA 6010B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010B	Aluminum	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010B	Aluminum	SCM	MN	
RCRP	EPA 6010B	Antimony	SCM	MN	
RCRP	EPA 6010B	Antimony	NPW	MN	
RCRP	EPA 6010B	Arsenic	SCM	MN	
RCRP	EPA 6010B	Arsenic	NPW	MN	
RCRP	EPA 6010B	Barium	NPW	MN	
RCRP	EPA 6010B	Barium	SCM	MN	
RCRP	EPA 6010B	Beryllium	NPW	MN	
RCRP	EPA 6010B	Beryllium	SCM	MN	
RCRP	EPA 6010B	Boron	SCM	MN	
RCRP	EPA 6010B	Boron	NPW	MN	
RCRP	EPA 6010B	Cadmium	NPW	MN	
RCRP	EPA 6010B	Cadmium	SCM	MN	
RCRP	EPA 6010B	Calcium	SCM	MN	
RCRP	EPA 6010B	Calcium	NPW	MN	
RCRP	EPA 6010B	Chromium	SCM	MN	
RCRP	EPA 6010B	Cobalt	SCM	MN	
RCRP	EPA 6010B	Cobalt	NPW	MN	
RCRP	EPA 6010B	Copper	NPW	MN	
RCRP	EPA 6010B	Copper	SCM	MN	
RCRP	EPA 6010B	Iron	NPW	MN	
RCRP	EPA 6010B	Iron	SCM	MN	
RCRP	EPA 6010B	Lead	SCM	MN	
RCRP	EPA 6010B	Lead	NPW	MN	
RCRP	EPA 6010B	Magnesium	NPW	MN	
RCRP	EPA 6010B	Magnesium	SCM	MN	
RCRP	EPA 6010B	Manganese	SCM	MN	
RCRP	EPA 6010B	Manganese	NPW	MN	
RCRP	EPA 6010B	Molybdenum	SCM	MN	
RCRP	EPA 6010B	Molybdenum	NPW	MN	
RCRP	EPA 6010B	Nickel	NPW	MN	
RCRP	EPA 6010B	Nickel	SCM	MN	
RCRP	EPA 6010B	Potassium	SCM	MN	
RCRP	EPA 6010B	Potassium	NPW	MN	
RCRP	EPA 6010B	Selenium	SCM	MN	
RCRP	EPA 6010B	Selenium	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010B	Silica as SiO2	NPW	MN	
RCRP	EPA 6010B	Silver	SCM	MN	
RCRP	EPA 6010B	Silver	NPW	MN	
RCRP	EPA 6010B	Sodium	SCM	MN	
RCRP	EPA 6010B	Sodium	NPW	MN	
RCRP	EPA 6010B	Srontium	SCM	MN	
RCRP	EPA 6010B	Srontium	NPW	MN	
RCRP	EPA 6010B	Thallium	SCM	MN	
RCRP	EPA 6010B	Thallium	NPW	MN	
RCRP	EPA 6010B	Tin	SCM	MN	
RCRP	EPA 6010B	Tin	NPW	MN	
RCRP	EPA 6010B	Total chromium	NPW	MN	
RCRP	EPA 6010B	Vanadium	SCM	MN	
RCRP	EPA 6010B	Vanadium	NPW	MN	
RCRP	EPA 6010B	Zinc	NPW	MN	
RCRP	EPA 6010B	Zinc	SCM	MN	

EPA 6020

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020	Aluminum	NPW	MN	
RCRP	EPA 6020	Antimony	NPW	MN	
RCRP	EPA 6020	Arsenic	NPW	MN	
RCRP	EPA 6020	Barium	NPW	MN	
RCRP	EPA 6020	Beryllium	NPW	MN	
RCRP	EPA 6020	Cadmium	NPW	MN	
RCRP	EPA 6020	Cobalt	NPW	MN	
RCRP	EPA 6020	Copper	NPW	MN	
RCRP	EPA 6020	Lead	NPW	MN	
RCRP	EPA 6020	Manganese	NPW	MN	
RCRP	EPA 6020	Nickel	NPW	MN	
RCRP	EPA 6020	Silver	NPW	MN	
RCRP	EPA 6020	Thallium	NPW	MN	
RCRP	EPA 6020	Total chromium	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020	Zinc	NPW	MN	

EPA 7470A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7470A	Mercury	NPW	MN	

EPA 7471A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7471A	Mercury	SCM	MN	

EPA 8081A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8081A	4,4'-DDD	NPW	MN	
RCRP	EPA 8081A	4,4'-DDD	SCM	MN	
RCRP	EPA 8081A	4,4'-DDE	SCM	MN	
RCRP	EPA 8081A	4,4'-DDE	NPW	MN	
RCRP	EPA 8081A	4,4'-DDT	NPW	MN	
RCRP	EPA 8081A	4,4'-DDT	SCM	MN	
RCRP	EPA 8081A	Aldrin	SCM	MN	
RCRP	EPA 8081A	Aldrin	NPW	MN	
RCRP	EPA 8081A	alpha-BHC (alpha-Hexachlorocyclohexane)	SCM	MN	
RCRP	EPA 8081A	alpha-BHC (alpha-Hexachlorocyclohexane)	NPW	MN	
RCRP	EPA 8081A	alpha-Chlordane	SCM	MN	
RCRP	EPA 8081A	alpha-Chlordane	NPW	MN	
RCRP	EPA 8081A	beta-BHC (beta-Hexachlorocyclohexane)	NPW	MN	
RCRP	EPA 8081A	beta-BHC (beta-Hexachlorocyclohexane)	SCM	MN	
RCRP	EPA 8081A	delta-BHC	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8081A	delta-BHC	NPW	MN	
RCRP	EPA 8081A	Dieldrin	NPW	MN	
RCRP	EPA 8081A	Dieldrin	SCM	MN	
RCRP	EPA 8081A	Endosulfan I	SCM	MN	
RCRP	EPA 8081A	Endosulfan I	NPW	MN	
RCRP	EPA 8081A	Endosulfan II	SCM	MN	
RCRP	EPA 8081A	Endosulfan II	NPW	MN	
RCRP	EPA 8081A	Endosulfan sulfate	NPW	MN	
RCRP	EPA 8081A	Endosulfan sulfate	SCM	MN	
RCRP	EPA 8081A	Endrin	NPW	MN	
RCRP	EPA 8081A	Endrin	SCM	MN	
RCRP	EPA 8081A	Endrin aldehyde	NPW	MN	
RCRP	EPA 8081A	Endrin aldehyde	SCM	MN	
RCRP	EPA 8081A	Endrin ketone	NPW	MN	
RCRP	EPA 8081A	Endrin ketone	SCM	MN	
RCRP	EPA 8081A	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	NPW	MN	
RCRP	EPA 8081A	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	SCM	MN	
RCRP	EPA 8081A	gamma-Chlordane	SCM	MN	
RCRP	EPA 8081A	gamma-Chlordane	NPW	MN	
RCRP	EPA 8081A	Heptachlor	SCM	MN	
RCRP	EPA 8081A	Heptachlor	NPW	MN	
RCRP	EPA 8081A	Heptachlor epoxide	SCM	MN	
RCRP	EPA 8081A	Heptachlor epoxide	NPW	MN	
RCRP	EPA 8081A	Methoxychlor	SCM	MN	
RCRP	EPA 8081A	Methoxychlor	NPW	MN	
RCRP	EPA 8081A	Toxaphene (Chlorinated camphene)	NPW	MN	
RCRP	EPA 8081A	Toxaphene (Chlorinated camphene)	SCM	MN	

EPA 8082

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8082	Aroclor-1016 (PCB-1016)	NPW	MN	
RCRP	EPA 8082	Aroclor-1016 (PCB-1016)	SCM	MN	
RCRP	EPA 8082	Aroclor-1221 (PCB-1221)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8082	Aroclor-1221 (PCB-1221)	NPW	MN	
RCRP	EPA 8082	Aroclor-1232 (PCB-1232)	SCM	MN	
RCRP	EPA 8082	Aroclor-1232 (PCB-1232)	NPW	MN	
RCRP	EPA 8082	Aroclor-1242 (PCB-1242)	SCM	MN	
RCRP	EPA 8082	Aroclor-1242 (PCB-1242)	NPW	MN	
RCRP	EPA 8082	Aroclor-1248 (PCB-1248)	NPW	MN	
RCRP	EPA 8082	Aroclor-1248 (PCB-1248)	SCM	MN	
RCRP	EPA 8082	Aroclor-1254 (PCB-1254)	SCM	MN	
RCRP	EPA 8082	Aroclor-1254 (PCB-1254)	NPW	MN	
RCRP	EPA 8082	Aroclor-1260 (PCB-1260)	SCM	MN	
RCRP	EPA 8082	Aroclor-1260 (PCB-1260)	NPW	MN	
RCRP	EPA 8082	PCBs	SCM	MN	

EPA 8260B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	1,1,1,2-Tetrachloroethane	NPW	MN	
RCRP	EPA 8260B	1,1,1,2-Tetrachloroethane	SCM	MN	
RCRP	EPA 8260B	1,1,1-Trichloroethane	NPW	MN	
RCRP	EPA 8260B	1,1,1-Trichloroethane	SCM	MN	
RCRP	EPA 8260B	1,1,2,2-Tetrachloroethane	NPW	MN	
RCRP	EPA 8260B	1,1,2,2-Tetrachloroethane	SCM	MN	
RCRP	EPA 8260B	1,1,2-Trichloroethane	SCM	MN	
RCRP	EPA 8260B	1,1,2-Trichloroethane	NPW	MN	
RCRP	EPA 8260B	1,1-Dichloroethane	SCM	MN	
RCRP	EPA 8260B	1,1-Dichloroethane	NPW	MN	
RCRP	EPA 8260B	1,1-Dichloroethylene	NPW	MN	
RCRP	EPA 8260B	1,1-Dichloroethylene	SCM	MN	
RCRP	EPA 8260B	1,1-Dichloropropene	NPW	MN	
RCRP	EPA 8260B	1,1-Dichloropropene	SCM	MN	
RCRP	EPA 8260B	1,2,3-Trichlorobenzene	SCM	MN	
RCRP	EPA 8260B	1,2,3-Trichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,2,3-Trichloropropane	SCM	MN	
RCRP	EPA 8260B	1,2,3-Trichloropropane	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	1,2,4-Trichlorobenzene	SCM	MN	
RCRP	EPA 8260B	1,2,4-Trichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,2,4-Trimethylbenzene	SCM	MN	
RCRP	EPA 8260B	1,2,4-Trimethylbenzene	NPW	MN	
RCRP	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)	SCM	MN	
RCRP	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)	NPW	MN	
RCRP	EPA 8260B	1,2-Dibromoethane (EDB, Ethylene dibromide)	SCM	MN	
RCRP	EPA 8260B	1,2-Dibromoethane (EDB, Ethylene dibromide)	NPW	MN	
RCRP	EPA 8260B	1,2-Dichlorobenzene	SCM	MN	
RCRP	EPA 8260B	1,2-Dichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,2-Dichloroethane (Ethylene dichloride)	NPW	MN	
RCRP	EPA 8260B	1,2-Dichloroethane (Ethylene dichloride)	SCM	MN	
RCRP	EPA 8260B	1,2-Dichloropropane	SCM	MN	
RCRP	EPA 8260B	1,2-Dichloropropane	NPW	MN	
RCRP	EPA 8260B	1,3,5-Trimethylbenzene	SCM	MN	
RCRP	EPA 8260B	1,3,5-Trimethylbenzene	NPW	MN	
RCRP	EPA 8260B	1,3-Dichlorobenzene	SCM	MN	
RCRP	EPA 8260B	1,3-Dichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,3-Dichloropropane	SCM	MN	
RCRP	EPA 8260B	1,3-Dichloropropane	NPW	MN	
RCRP	EPA 8260B	1,4-Dichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,4-Dichlorobenzene	SCM	MN	
RCRP	EPA 8260B	2,2-Dichloropropane	NPW	MN	
RCRP	EPA 8260B	2,2-Dichloropropane	SCM	MN	
RCRP	EPA 8260B	2-Butanone (Methyl ethyl ketone, MEK)	SCM	MN	
RCRP	EPA 8260B	2-Butanone (Methyl ethyl ketone, MEK)	NPW	MN	
RCRP	EPA 8260B	2-Chlorotoluene	SCM	MN	
RCRP	EPA 8260B	2-Chlorotoluene	NPW	MN	
RCRP	EPA 8260B	4-Chlorotoluene	NPW	MN	
RCRP	EPA 8260B	4-Chlorotoluene	SCM	MN	
RCRP	EPA 8260B	4-Isopropyltoluene (p-Cymene)	SCM	MN	
RCRP	EPA 8260B	4-Isopropyltoluene (p-Cymene)	NPW	MN	
RCRP	EPA 8260B	4-Methyl-2-pentanone (MIBK)	NPW	MN	
RCRP	EPA 8260B	4-Methyl-2-pentanone (MIBK)	SCM	MN	
RCRP	EPA 8260B	Acetone	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Acetone	SCM	MN	
RCRP	EPA 8260B	Allyl chloride (3-Chloropropene)	NPW	MN	
RCRP	EPA 8260B	Allyl chloride (3-Chloropropene)	SCM	MN	
RCRP	EPA 8260B	Benzene	NPW	MN	
RCRP	EPA 8260B	Benzene	SCM	MN	
RCRP	EPA 8260B	Bromobenzene	NPW	MN	
RCRP	EPA 8260B	Bromobenzene	SCM	MN	
RCRP	EPA 8260B	Bromochloromethane	SCM	MN	
RCRP	EPA 8260B	Bromochloromethane	NPW	MN	
RCRP	EPA 8260B	Bromodichloromethane	NPW	MN	
RCRP	EPA 8260B	Bromodichloromethane	SCM	MN	
RCRP	EPA 8260B	Bromoform	NPW	MN	
RCRP	EPA 8260B	Bromoform	SCM	MN	
RCRP	EPA 8260B	Carbon tetrachloride	SCM	MN	
RCRP	EPA 8260B	Carbon tetrachloride	NPW	MN	
RCRP	EPA 8260B	Chlorobenzene	SCM	MN	
RCRP	EPA 8260B	Chlorobenzene	NPW	MN	
RCRP	EPA 8260B	Chlorodibromomethane	NPW	MN	
RCRP	EPA 8260B	Chlorodibromomethane	SCM	MN	
RCRP	EPA 8260B	Chloroethane (Ethyl chloride)	SCM	MN	
RCRP	EPA 8260B	Chloroethane (Ethyl chloride)	NPW	MN	
RCRP	EPA 8260B	Chloroform	SCM	MN	
RCRP	EPA 8260B	Chloroform	NPW	MN	
RCRP	EPA 8260B	cis-1,2-Dichloroethylene	SCM	MN	
RCRP	EPA 8260B	cis-1,2-Dichloroethylene	NPW	MN	
RCRP	EPA 8260B	cis-1,3-Dichloropropene	SCM	MN	
RCRP	EPA 8260B	cis-1,3-Dichloropropene	NPW	MN	
RCRP	EPA 8260B	Dibromomethane (Methylene bromide)	NPW	MN	
RCRP	EPA 8260B	Dibromomethane (Methylene bromide)	SCM	MN	
RCRP	EPA 8260B	Dichlorodifluoromethane (Freon-12)	NPW	MN	
RCRP	EPA 8260B	Dichlorodifluoromethane (Freon-12)	SCM	MN	
RCRP	EPA 8260B	Diethyl ether	SCM	MN	
RCRP	EPA 8260B	Diethyl ether	NPW	MN	
RCRP	EPA 8260B	Ethylbenzene	SCM	MN	
RCRP	EPA 8260B	Ethylbenzene	NPW	MN	
RCRP	EPA 8260B	Hexachlorobutadiene	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Hexachlorobutadiene	SCM	MN	
RCRP	EPA 8260B	Isopropylbenzene	SCM	MN	
RCRP	EPA 8260B	Isopropylbenzene	NPW	MN	
RCRP	EPA 8260B	m+p-xylene	SCM	MN	
RCRP	EPA 8260B	m+p-xylene	NPW	MN	
RCRP	EPA 8260B	Methyl bromide (Bromomethane)	NPW	MN	
RCRP	EPA 8260B	Methyl bromide (Bromomethane)	SCM	MN	
RCRP	EPA 8260B	Methyl chloride (Chloromethane)	NPW	MN	
RCRP	EPA 8260B	Methyl chloride (Chloromethane)	SCM	MN	
RCRP	EPA 8260B	Methyl tert-butyl ether (MTBE)	NPW	MN	
RCRP	EPA 8260B	Methyl tert-butyl ether (MTBE)	SCM	MN	
RCRP	EPA 8260B	Methylene chloride (Dichloromethane)	SCM	MN	
RCRP	EPA 8260B	Methylene chloride (Dichloromethane)	NPW	MN	
RCRP	EPA 8260B	n-Butylbenzene	NPW	MN	
RCRP	EPA 8260B	n-Butylbenzene	SCM	MN	
RCRP	EPA 8260B	n-Propylbenzene	SCM	MN	
RCRP	EPA 8260B	n-Propylbenzene	NPW	MN	
RCRP	EPA 8260B	Naphthalene	SCM	MN	
RCRP	EPA 8260B	Naphthalene	NPW	MN	
RCRP	EPA 8260B	o-Xylene	NPW	MN	
RCRP	EPA 8260B	o-Xylene	SCM	MN	
RCRP	EPA 8260B	sec-Butylbenzene	NPW	MN	
RCRP	EPA 8260B	sec-Butylbenzene	SCM	MN	
RCRP	EPA 8260B	Styrene	NPW	MN	
RCRP	EPA 8260B	Styrene	SCM	MN	
RCRP	EPA 8260B	tert-Butylbenzene	NPW	MN	
RCRP	EPA 8260B	tert-Butylbenzene	SCM	MN	
RCRP	EPA 8260B	Tetrachloroethylene (Perchloroethylene)	NPW	MN	
RCRP	EPA 8260B	Tetrachloroethylene (Perchloroethylene)	SCM	MN	
RCRP	EPA 8260B	Toluene	NPW	MN	
RCRP	EPA 8260B	Toluene	SCM	MN	
RCRP	EPA 8260B	trans-1,2-Dichloroethylene	SCM	MN	
RCRP	EPA 8260B	trans-1,2-Dichloroethylene	NPW	MN	
RCRP	EPA 8260B	trans-1,3-Dichloropropylene	SCM	MN	
RCRP	EPA 8260B	trans-1,3-Dichloropropylene	NPW	MN	
RCRP	EPA 8260B	Trichloroethene (Trichloroethylene)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Trichloroethene (Trichloroethylene)	NPW	MN	
RCRP	EPA 8260B	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	SCM	MN	
RCRP	EPA 8260B	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	NPW	MN	
RCRP	EPA 8260B	Vinyl chloride	SCM	MN	
RCRP	EPA 8260B	Vinyl chloride	NPW	MN	

EPA 8270C

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	1,2,4-Trichlorobenzene	NPW	MN	
RCRP	EPA 8270C	1,2,4-Trichlorobenzene	SCM	MN	
RCRP	EPA 8270C	1,2-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270C	1,2-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270C	1,2-Diphenylhydrazine	SCM	MN	
RCRP	EPA 8270C	1,2-Diphenylhydrazine	NPW	MN	
RCRP	EPA 8270C	1,3-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270C	1,3-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270C	1,4-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270C	1,4-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270C	2,3,4,6-Tetrachlorophenol	SCM	MN	
RCRP	EPA 8270C	2,3,4,6-Tetrachlorophenol	NPW	MN	
RCRP	EPA 8270C	2,4,5-Trichlorophenol	NPW	MN	
RCRP	EPA 8270C	2,4,5-Trichlorophenol	SCM	MN	
RCRP	EPA 8270C	2,4,6-Trichlorophenol	NPW	MN	
RCRP	EPA 8270C	2,4,6-Trichlorophenol	SCM	MN	
RCRP	EPA 8270C	2,4-Dichlorophenol	NPW	MN	
RCRP	EPA 8270C	2,4-Dichlorophenol	SCM	MN	
RCRP	EPA 8270C	2,4-Dimethylphenol	NPW	MN	
RCRP	EPA 8270C	2,4-Dimethylphenol	SCM	MN	
RCRP	EPA 8270C	2,4-Dinitrophenol	NPW	MN	
RCRP	EPA 8270C	2,4-Dinitrophenol	SCM	MN	
RCRP	EPA 8270C	2,4-Dinitrotoluene (2,4-DNT)	NPW	MN	
RCRP	EPA 8270C	2,4-Dinitrotoluene (2,4-DNT)	SCM	MN	
RCRP	EPA 8270C	2,6-Dichlorophenol	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	2,6-Dichlorophenol	SCM	MN	
RCRP	EPA 8270C	2,6-Dinitrotoluene (2,6-DNT)	NPW	MN	
RCRP	EPA 8270C	2,6-Dinitrotoluene (2,6-DNT)	SCM	MN	
RCRP	EPA 8270C	2-Chloronaphthalene	NPW	MN	
RCRP	EPA 8270C	2-Chloronaphthalene	SCM	MN	
RCRP	EPA 8270C	2-Chlorophenol	SCM	MN	
RCRP	EPA 8270C	2-Chlorophenol	NPW	MN	
RCRP	EPA 8270C	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	SCM	MN	
RCRP	EPA 8270C	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NPW	MN	
RCRP	EPA 8270C	2-Methylnaphthalene	NPW	MN	
RCRP	EPA 8270C	2-Methylnaphthalene	SCM	MN	
RCRP	EPA 8270C	2-Methylphenol (o-Cresol)	NPW	MN	
RCRP	EPA 8270C	2-Methylphenol (o-Cresol)	SCM	MN	
RCRP	EPA 8270C	2-Nitroaniline	SCM	MN	
RCRP	EPA 8270C	2-Nitroaniline	NPW	MN	
RCRP	EPA 8270C	2-Nitrophenol	NPW	MN	
RCRP	EPA 8270C	2-Nitrophenol	SCM	MN	
RCRP	EPA 8270C	3,3'-Dichlorobenzidine	SCM	MN	
RCRP	EPA 8270C	3,3'-Dichlorobenzidine	NPW	MN	
RCRP	EPA 8270C	3-Methylcholanthrene	SCM	MN	
RCRP	EPA 8270C	3-Nitroaniline	NPW	MN	
RCRP	EPA 8270C	3-Nitroaniline	SCM	MN	
RCRP	EPA 8270C	4-Bromophenyl phenyl ether	NPW	MN	
RCRP	EPA 8270C	4-Bromophenyl phenyl ether	SCM	MN	
RCRP	EPA 8270C	4-Chloro-3-methylphenol	SCM	MN	
RCRP	EPA 8270C	4-Chloro-3-methylphenol	NPW	MN	
RCRP	EPA 8270C	4-Chloroaniline	SCM	MN	
RCRP	EPA 8270C	4-Chloroaniline	NPW	MN	
RCRP	EPA 8270C	4-Chlorophenyl phenylether	NPW	MN	
RCRP	EPA 8270C	4-Chlorophenyl phenylether	SCM	MN	
RCRP	EPA 8270C	4-Methylphenol (p-Cresol)	NPW	MN	
RCRP	EPA 8270C	4-Methylphenol (p-Cresol)	SCM	MN	
RCRP	EPA 8270C	4-Nitroaniline	SCM	MN	
RCRP	EPA 8270C	4-Nitroaniline	NPW	MN	
RCRP	EPA 8270C	4-Nitrophenol	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	4-Nitrophenol	SCM	MN	
RCRP	EPA 8270C	7,12-Dimethylbenz(a) anthracene	SCM	MN	
RCRP	EPA 8270C	Acenaphthene	NPW	MN	
RCRP	EPA 8270C	Acenaphthene	SCM	MN	
RCRP	EPA 8270C	Acenaphthylene	SCM	MN	
RCRP	EPA 8270C	Acenaphthylene	NPW	MN	
RCRP	EPA 8270C	Aniline	NPW	MN	
RCRP	EPA 8270C	Aniline	SCM	MN	
RCRP	EPA 8270C	Anthracene	SCM	MN	
RCRP	EPA 8270C	Anthracene	NPW	MN	
RCRP	EPA 8270C	Benzidine	NPW	MN	
RCRP	EPA 8270C	Benzidine	SCM	MN	
RCRP	EPA 8270C	Benzo(a)anthracene	NPW	MN	
RCRP	EPA 8270C	Benzo(a)anthracene	SCM	MN	
RCRP	EPA 8270C	Benzo(a)pyrene	SCM	MN	
RCRP	EPA 8270C	Benzo(a)pyrene	NPW	MN	
RCRP	EPA 8270C	Benzo(g,h,i)perylene	NPW	MN	
RCRP	EPA 8270C	Benzo(g,h,i)perylene	SCM	MN	
RCRP	EPA 8270C	Benzo(k)fluoranthene	SCM	MN	
RCRP	EPA 8270C	Benzo(k)fluoranthene	NPW	MN	
RCRP	EPA 8270C	Benzo[b]fluoranthene	NPW	MN	
RCRP	EPA 8270C	Benzo[b]fluoranthene	SCM	MN	
RCRP	EPA 8270C	Benzoic acid	SCM	MN	
RCRP	EPA 8270C	Benzoic acid	NPW	MN	
RCRP	EPA 8270C	Benzyl alcohol	NPW	MN	
RCRP	EPA 8270C	Benzyl alcohol	SCM	MN	
RCRP	EPA 8270C	bis(2-Chloroethoxy)methane	SCM	MN	
RCRP	EPA 8270C	bis(2-Chloroethoxy)methane	NPW	MN	
RCRP	EPA 8270C	bis(2-Chloroethyl) ether	NPW	MN	
RCRP	EPA 8270C	bis(2-Chloroethyl) ether	SCM	MN	
RCRP	EPA 8270C	bis(2-Chloroisopropyl) ether	SCM	MN	
RCRP	EPA 8270C	bis(2-Chloroisopropyl) ether	NPW	MN	
RCRP	EPA 8270C	Butyl benzyl phthalate	SCM	MN	
RCRP	EPA 8270C	Butyl benzyl phthalate	NPW	MN	
RCRP	EPA 8270C	Chrysene	NPW	MN	
RCRP	EPA 8270C	Chrysene	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	SCM	MN	
RCRP	EPA 8270C	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	NPW	MN	
RCRP	EPA 8270C	Di-n-butyl phthalate	SCM	MN	
RCRP	EPA 8270C	Di-n-butyl phthalate	NPW	MN	
RCRP	EPA 8270C	Di-n-octyl phthalate	SCM	MN	
RCRP	EPA 8270C	Di-n-octyl phthalate	NPW	MN	
RCRP	EPA 8270C	Dibenz(a, j) acridine	SCM	MN	
RCRP	EPA 8270C	Dibenz(a,h) anthracene	SCM	MN	
RCRP	EPA 8270C	Dibenz(a,h) anthracene	NPW	MN	
RCRP	EPA 8270C	Dibenzo(a,e) pyrene	SCM	MN	
RCRP	EPA 8270C	Dibenzofuran	NPW	MN	
RCRP	EPA 8270C	Dibenzofuran	SCM	MN	
RCRP	EPA 8270C	Diethyl phthalate	NPW	MN	
RCRP	EPA 8270C	Diethyl phthalate	SCM	MN	
RCRP	EPA 8270C	Dimethyl phthalate	NPW	MN	
RCRP	EPA 8270C	Dimethyl phthalate	SCM	MN	
RCRP	EPA 8270C	Fluoranthene	NPW	MN	
RCRP	EPA 8270C	Fluoranthene	SCM	MN	
RCRP	EPA 8270C	Fluorene	SCM	MN	
RCRP	EPA 8270C	Fluorene	NPW	MN	
RCRP	EPA 8270C	Hexachlorobenzene	NPW	MN	
RCRP	EPA 8270C	Hexachlorobenzene	SCM	MN	
RCRP	EPA 8270C	Hexachlorobutadiene	SCM	MN	
RCRP	EPA 8270C	Hexachlorobutadiene	NPW	MN	
RCRP	EPA 8270C	Hexachlorocyclopentadiene	NPW	MN	
RCRP	EPA 8270C	Hexachlorocyclopentadiene	SCM	MN	
RCRP	EPA 8270C	Hexachloroethane	SCM	MN	
RCRP	EPA 8270C	Hexachloroethane	NPW	MN	
RCRP	EPA 8270C	Indeno(1,2,3-cd) pyrene	SCM	MN	
RCRP	EPA 8270C	Indeno(1,2,3-cd) pyrene	NPW	MN	
RCRP	EPA 8270C	Isophorone	NPW	MN	
RCRP	EPA 8270C	Isophorone	SCM	MN	
RCRP	EPA 8270C	n-Nitrosodi-n-propylamine	NPW	MN	
RCRP	EPA 8270C	n-Nitrosodi-n-propylamine	SCM	MN	
RCRP	EPA 8270C	n-Nitrosodimethylamine	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	n-Nitrosodimethylamine	SCM	MN	
RCRP	EPA 8270C	n-Nitrosodiphenylamine	NPW	MN	
RCRP	EPA 8270C	n-Nitrosodiphenylamine	SCM	MN	
RCRP	EPA 8270C	Naphthalene	SCM	MN	
RCRP	EPA 8270C	Naphthalene	NPW	MN	
RCRP	EPA 8270C	Nitrobenzene	SCM	MN	
RCRP	EPA 8270C	Nitrobenzene	NPW	MN	
RCRP	EPA 8270C	Pentachlorophenol	NPW	MN	
RCRP	EPA 8270C	Pentachlorophenol	SCM	MN	
RCRP	EPA 8270C	Phenanthrene	NPW	MN	
RCRP	EPA 8270C	Phenanthrene	SCM	MN	
RCRP	EPA 8270C	Phenol	SCM	MN	
RCRP	EPA 8270C	Phenol	NPW	MN	
RCRP	EPA 8270C	Pyrene	NPW	MN	
RCRP	EPA 8270C	Pyrene	SCM	MN	

EPA 8270C SIM

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C SIM	Benzo(a)anthracene	NPW	MN	
RCRP	EPA 8270C SIM	Benzo(a)pyrene	NPW	MN	
RCRP	EPA 8270C SIM	Benzo(j)fluoranthene	NPW	MN	
RCRP	EPA 8270C SIM	Benzo(k)fluoranthene	NPW	MN	
RCRP	EPA 8270C SIM	Benzo[b]fluoranthene	NPW	MN	
RCRP	EPA 8270C SIM	Dibenz(a,h) anthracene	NPW	MN	
RCRP	EPA 8270C SIM	Fluoranthene	NPW	MN	
RCRP	EPA 8270C SIM	Indeno(1,2,3-cd) pyrene	NPW	MN	
RCRP	EPA 8270C SIM	Pyrene	NPW	MN	

Safe Drinking Water Program

EPA 200.7

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 200.7	Aluminum	DW	MN	
SDWP	EPA 200.7	Arsenic	DW	MN	
SDWP	EPA 200.7	Barium	DW	MN	
SDWP	EPA 200.7	Beryllium	DW	MN	
SDWP	EPA 200.7	Cadmium	DW	MN	
SDWP	EPA 200.7	Calcium	DW	MN	
SDWP	EPA 200.7	Chromium	DW	MN	
SDWP	EPA 200.7	Copper	DW	MN	
SDWP	EPA 200.7	Iron	DW	MN	
SDWP	EPA 200.7	Magnesium	DW	MN	
SDWP	EPA 200.7	Manganese	DW	MN	
SDWP	EPA 200.7	Nickel	DW	MN	
SDWP	EPA 200.7	Silica as SiO2	DW	MN	
SDWP	EPA 200.7	Silver	DW	MN	
SDWP	EPA 200.7	Sodium	DW	MN	
SDWP	EPA 200.7	Zinc	DW	MN	

EPA 200.8

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 200.8	Aluminum	DW	MN	
SDWP	EPA 200.8	Antimony	DW	MN	
SDWP	EPA 200.8	Arsenic	DW	MN	
SDWP	EPA 200.8	Barium	DW	MN	
SDWP	EPA 200.8	Beryllium	DW	MN	
SDWP	EPA 200.8	Cadmium	DW	MN	
SDWP	EPA 200.8	Chromium	DW	MN	
SDWP	EPA 200.8	Copper	DW	MN	
SDWP	EPA 200.8	Lead	DW	MN	
SDWP	EPA 200.8	Manganese	DW	MN	
SDWP	EPA 200.8	Mercury	DW	MN	
SDWP	EPA 200.8	Nickel	DW	MN	
SDWP	EPA 200.8	Selenium	DW	MN	
SDWP	EPA 200.8	Silver	DW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 200.8	Thallium	DW	MN	
SDWP	EPA 200.8	Uranium	DW	MN	
SDWP	EPA 200.8	Zinc	DW	MN	

Underground Storage Tank Program

EPA TO-15

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	EPA TO-15	1,1,1-Trichloroethane	AIR	MN	
USTP	EPA TO-15	1,1,2,2-Tetrachloroethane	AIR	MN	
USTP	EPA TO-15	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	AIR	MN	
USTP	EPA TO-15	1,1,2-Trichloroethane	AIR	MN	
USTP	EPA TO-15	1,1-Dichloroethane	AIR	MN	
USTP	EPA TO-15	1,1-Dichloroethylene	AIR	MN	
USTP	EPA TO-15	1,2,4-Trichlorobenzene	AIR	MN	
USTP	EPA TO-15	1,2,4-Trimethylbenzene	AIR	MN	
USTP	EPA TO-15	1,2-Dibromoethane (EDB, Ethylene dibromide)	AIR	MN	
USTP	EPA TO-15	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)	AIR	MN	
USTP	EPA TO-15	1,2-Dichlorobenzene	AIR	MN	
USTP	EPA TO-15	1,2-Dichloroethane (Ethylene dichloride)	AIR	MN	
USTP	EPA TO-15	1,2-Dichloropropane	AIR	MN	
USTP	EPA TO-15	1,3,5-Trimethylbenzene	AIR	MN	
USTP	EPA TO-15	1,3-Butadiene	AIR	MN	
USTP	EPA TO-15	1,3-Dichlorobenzene	AIR	MN	
USTP	EPA TO-15	1,4-Dichlorobenzene	AIR	MN	
USTP	EPA TO-15	1-Propene	AIR	MN	
USTP	EPA TO-15	2-Butanone (Methyl ethyl ketone, MEK)	AIR	MN	
USTP	EPA TO-15	2-Hexanone	AIR	MN	
USTP	EPA TO-15	4-Ethyltoluene	AIR	MN	
USTP	EPA TO-15	4-Methyl-2-pentanone (MIBK)	AIR	MN	
USTP	EPA TO-15	Acetone	AIR	MN	
USTP	EPA TO-15	Benzene	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
USTP	EPA TO-15	Benzyl chloride	AIR	MN	
USTP	EPA TO-15	Bromodichloromethane	AIR	MN	
USTP	EPA TO-15	Bromoform	AIR	MN	
USTP	EPA TO-15	Carbon disulfide	AIR	MN	
USTP	EPA TO-15	Carbon tetrachloride	AIR	MN	
USTP	EPA TO-15	Chlorobenzene	AIR	MN	
USTP	EPA TO-15	Chlorodibromomethane	AIR	MN	
USTP	EPA TO-15	Chloroethane (Ethyl chloride)	AIR	MN	
USTP	EPA TO-15	Chloroform	AIR	MN	
USTP	EPA TO-15	cis-1,2-Dichloroethylene	AIR	MN	
USTP	EPA TO-15	cis-1,3-Dichloropropene	AIR	MN	
USTP	EPA TO-15	Cyclohexane	AIR	MN	
USTP	EPA TO-15	Dichlorodifluoromethane (Freon-12)	AIR	MN	
USTP	EPA TO-15	Ethanol	AIR	MN	
USTP	EPA TO-15	Ethyl acetate	AIR	MN	
USTP	EPA TO-15	Ethylbenzene	AIR	MN	
USTP	EPA TO-15	Hexachlorobutadiene	AIR	MN	
USTP	EPA TO-15	Isopropyl alcohol (2-Propanol, Isopropanol)	AIR	MN	
USTP	EPA TO-15	m+p-xylene	AIR	MN	
USTP	EPA TO-15	Methyl bromide (Bromomethane)	AIR	MN	
USTP	EPA TO-15	Methyl chloride (Chloromethane)	AIR	MN	
USTP	EPA TO-15	Methyl tert-butyl ether (MTBE)	AIR	MN	
USTP	EPA TO-15	Methylene chloride (Dichloromethane)	AIR	MN	
USTP	EPA TO-15	n-Heptane	AIR	MN	
USTP	EPA TO-15	n-Hexane	AIR	MN	
USTP	EPA TO-15	Naphthalene	AIR	MN	
USTP	EPA TO-15	o-Xylene	AIR	MN	
USTP	EPA TO-15	Styrene	AIR	MN	
USTP	EPA TO-15	Tetrachloroethylene (Perchloroethylene)	AIR	MN	
USTP	EPA TO-15	Tetrahydrofuran (THF)	AIR	MN	
USTP	EPA TO-15	Toluene	AIR	MN	
USTP	EPA TO-15	trans-1,2-Dichloroethylene	AIR	MN	
USTP	EPA TO-15	trans-1,3-Dichloropropylene	AIR	MN	
USTP	EPA TO-15	Trichloroethene (Trichloroethylene)	AIR	MN	
USTP	EPA TO-15	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
USTP	EPA TO-15	Vinyl acetate	AIR	MN	
USTP	EPA TO-15	Vinyl chloride	AIR	MN	

WI(95) DRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) DRO	Diesel range organics (DRO)	NPW	MN	
USTP	WI(95) DRO	Diesel range organics (DRO)	SCM	MN	

WI(95) GRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) GRO	Gasoline range organics (GRO)	NPW	MN	
USTP	WI(95) GRO	Gasoline range organics (GRO)	SCM	MN	

WI(95) GRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) GRO	Petroleum Volatile Organic Compounds (PVOC)	SCM	MN	
USTP	WI(95) GRO	Petroleum Volatile Organic Compounds (PVOC)	NPW	MN	

Note: Method beginning with "SM" refer to the approved editions of Standard methods for the Examination of Water and Wastes. Approved methods are listed in the applicable parts of Title 40 of the Code of Federal Regulations (including its subsequent Federal Register updates), MN Statutes and Rules, and state-issued permits.

United States Department of Commerce
National Institute of Standards and Technology

NVLAP®

Certificate of Accreditation to ISO/IEC 17025:2005

NVLAP LAB CODE: 102081-0

Legend Technical Services, Inc.

St. Paul, MN

is accredited by the National Voluntary Laboratory Accreditation Program for specific services, listed on the Scope of Accreditation, for:

BULK ASBESTOS FIBER ANALYSIS

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communique dated January 2009).

2011-04-01 through 2012-03-31

Effective dates

Sally S. Bruce

For the National Institute of Standards and Technology

NVLAP-01C (REV. 2009-01-28)

UMP013056

National Voluntary Laboratory Accreditation Program

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

Legend Technical Services, Inc.
88 Empire Drive
St. Paul, MN 55103
Ms. Cheryl Sykora
Phone: 651-221-4085 Fax: 651-642-1239
E-Mail: cherylsykora@aol.com
URL: http://www.legend-group.com

BULK ASBESTOS FIBER ANALYSIS (PLM) **NVLAP LAB CODE 102081-0**

NVLAP Code	*Designation / Description*
18/A01	EPA-600/M4-82-020: Interim Method for the Determination of Asbestos in Bulk Insulation Samples

2011-04-01 through 2012-03-31
Effective dates

Sally S. Bruce
For the National Institute of Standards and Technology

UMP013057

NELAP - RECOGNIZED

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM BRANCH

CERTIFICATE OF NELAP ACCREDITATION

Is hereby granted to

Testamerica West Sacramento

880 Riverside Parkway

West Sacramento, CA 95605

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

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

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

Certificate No.: **01119CA**

Expiration Date: **1/31/2012**

Effective Date: **2/1/2011**

Richmond, California
subject to forfeiture or revocation

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

State of California—Health and Human Services Agency
California Department of Public Health

MARK B HORTON, MD, MSPH
Director

ARNOLD SCHWARZENEGGER
Governor

December 21, 2010

KARLA S. BUECHLER
TESTAMERICA WEST SACRAMENTO
880 RIVERSIDE PARKWAY
WEST SACRAMENTO, CA 95605

Dear KARLA S. BUECHLER: Certificate No. 01119CA

This is to advise you that the laboratory named above has been accredited under National Environmental Laboratory Accreditation Program (NELAP) as an environmental testing laboratory pursuant to the provisions of the Health and Safety Code (HSC), Division 101, Part 1, Chapter 4, Section 100825, et seq.

The Fields of Accreditation for which this laboratory has been accredited are enclosed. Accreditation shall remain in effect until **January 31, 2012** unless revoked by ELAP or withdrawn at your written request. To maintain accreditation, the laboratory shall comply with the National Environmental Laboratory Accreditation Conference (NELAC) Standards and all associated California Environmental Laboratory Accreditation Program Branch (ELAP) regulations and statutes.

The application for renewal of this certificate must be received before the expiration date of this certificate to remain in force according to the HSC 100845(a).

Please note that your laboratory is required to notify California ELAP of any major changes in key accreditation criteria within 30 calendar days of the change. This written notification includes, but is not limited to, changes in ownership, location, key personnel, and major instrumentation (HSC 100845(b) and (d), and NELAC Standard Section 4.3.2). The certificate must be returned to California ELAP upon loss of accredited status.

Your continued cooperation with the above requirements is essential for maintaining the high quality of the data produced by environmental laboratories accredited by the State of California.

If you have any questions, please contact Jane Jensen at (510) 620-3155.

Sincerely,

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

Enclosure

CALIFORNIA DEPARTMENT OF PUBLIC HEALTH

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM - NELAP RECOGNIZED

NELAP Fields of Accreditation

Testamerica West Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
Phone: (916) 373-5600

Certificate No.: 01119CA
Renew Date: 1/31/2012

102 - Inorganic Chemistry of Drinking Water

102.045	001	EPA 314.0	Perchlorate
102.047	001	EPA 331.0	Perchlorate
102.510	006	SM3120B	Hardness (calc.)
102.520	001	EPA 200.7	Calcium
102.520	002	EPA 200.7	Magnesium
102.520	003	EPA 200.7	Potassium
102.520	004	EPA 200.7	Silica
102.520	005	EPA 200.7	Sodium
102.520	006	EPA 200.7	Hardness (calc.)

103 - Toxic Chemical Elements of Drinking Water

103.130	001	EPA 200.7	Aluminum
103.130	003	EPA 200.7	Barium
103.130	004	EPA 200.7	Beryllium
103.130	005	EPA 200.7	Cadmium
103.130	007	EPA 200.7	Chromium
103.130	008	EPA 200.7	Copper
103.130	009	EPA 200.7	Iron
103.130	011	EPA 200.7	Manganese
103.130	012	EPA 200.7	Nickel
103.130	015	EPA 200.7	Silver
103.130	017	EPA 200.7	Zinc
103.140	001	EPA 200.8	Aluminum
103.140	002	EPA 200.8	Antimony
103.140	003	EPA 200.8	Arsenic
103.140	004	EPA 200.8	Barium
103.140	005	EPA 200.8	Beryllium
103.140	006	EPA 200.8	Cadmium
103.140	007	EPA 200.8	Chromium
103.140	008	EPA 200.8	Copper
103.140	009	EPA 200.8	Lead
103.140	010	EPA 200.8	Manganese
103.140	011	EPA 200.8	Mercury
103.140	012	EPA 200.8	Nickel

As of 12/21/2010, this list supersedes all previous lists for this certificate number.
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103.140	013	EPA 200.8	Selenium
103.140	014	EPA 200.8	Silver
103.140	015	EPA 200.8	Thallium
103.140	016	EPA 200.8	Zinc
103.160	001	EPA 245.1	Mercury

105 - Semi-volatile Organic Chemistry of Drinking Water

105.230	000	EPA 1613	Dioxins
105.230	001	EPA 1613	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)

108 - Inorganic Chemistry of Wastewater

108.020	001	EPA 120.1	Conductivity
108.112	001	EPA 200.7	Boron
108.112	002	EPA 200.7	Calcium
108.112	003	EPA 200.7	Hardness (calc.)
108.112	004	EPA 200.7	Magnesium
108.112	005	EPA 200.7	Potassium
108.112	006	EPA 200.7	Silica
108.112	007	EPA 200.7	Sodium
108.120	001	EPA 300.0	Bromide
108.120	002	EPA 300.0	Chloride
108.120	003	EPA 300.0	Fluoride
108.120	004	EPA 300.0	Nitrate
108.120	005	EPA 300.0	Nitrite
108.120	006	EPA 300.0	Nitrate-nitrite
108.120	008	EPA 300.0	Sulfate
108.141	001	EPA 310.2	Alkalinity
108.183	001	EPA 335.4	Cyanide, Total
108.200	001	EPA 350.1	Ammonia
108.211	001	EPA 351.2	Kjeldahl Nitrogen
108.232	001	EPA 353.2	Nitrate-nitrite
108.232	002	EPA 353.2	Nitrite
108.266	001	EPA 365.4	Phosphorus, Total
108.323	001	EPA 410.4	Chemical Oxygen Demand
108.381	001	EPA 1664A	Oil and Grease
108.410	001	SM2320B	Alkalinity
108.420	001	SM2340B	Hardness (calc.)
108.430	001	SM2510B	Conductivity
108.440	001	SM2540B	Residue, Total
108.441	001	SM2540C	Residue, Filterable
108.442	001	SM2540D	Residue, Non-filterable
108.472	001	SM4500-CN E	Cyanide, Total
108.473	001	SM4500-CN G	Cyanide, amenable

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108.490	001	SM4500-H+ B	pH
108.580	001	SM4500-S= D	Sulfide

109 - Toxic Chemical Elements of Wastewater

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

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109.020	018	EPA 200.8	Zinc
109.020	021	EPA 200.8	Iron
109.020	022	EPA 200.8	Tin
109.020	023	EPA 200.8	Titanium
109.190	001	EPA 245.1	Mercury

111 - Semi-volatile Organic Chemistry of Wastewater

111.111	000	EPA 1613B	Dioxins
111.111	001	EPA 1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
111.111	002	EPA 1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
111.111	003	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	004	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	005	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	006	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
111.111	007	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
111.111	008	EPA 1613B	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
111.111	009	EPA 1613B	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
111.111	010	EPA 1613B	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
111.111	011	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	012	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	013	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
111.111	014	EPA 1613B	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	015	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
111.111	016	EPA 1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
111.111	017	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
111.111	018	EPA 1613B	Total TCDD
111.111	019	EPA 1613B	Total PeCDD
111.111	020	EPA 1613B	Total HxCDD
111.111	021	EPA 1613B	Total HpCDD
111.111	022	EPA 1613B	Total TCDF
111.111	023	EPA 1613B	Total PeCDF
111.111	024	EPA 1613B	Total HxCDF
111.111	025	EPA 1613B	Total HpCDF

114 - Inorganic Chemistry of Hazardous Waste

114.010	001	EPA 6010B	Antimony
114.010	002	EPA 6010B	Arsenic
114.010	003	EPA 6010B	Barium
114.010	004	EPA 6010B	Beryllium
114.010	005	EPA 6010B	Cadmium
114.010	006	EPA 6010B	Chromium
114.010	007	EPA 6010B	Cobalt
114.010	008	EPA 6010B	Copper

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114.010	009	EPA 6010B	Lead	
114.010	010	EPA 6010B	Molybdenum	
114.010	011	EPA 6010B	Nickel	
114.010	012	EPA 6010B	Selenium	
114.010	013	EPA 6010B	Silver	
114.010	014	EPA 6010B	Thallium	
114.010	015	EPA 6010B	Vanadium	
114.010	016	EPA 6010B	Zinc	
114.010	026	EPA 6010B	Silica	Aqueous Only
114.010	027	EPA 6010B	Sodium	
114.020	001	EPA 6020	Antimony	
114.020	002	EPA 6020	Arsenic	
114.020	003	EPA 6020	Barium	
114.020	004	EPA 6020	Beryllium	
114.020	005	EPA 6020	Cadmium	
114.020	006	EPA 6020	Chromium	
114.020	007	EPA 6020	Cobalt	
114.020	008	EPA 6020	Copper	
114.020	009	EPA 6020	Lead	
114.020	010	EPA 6020	Molybdenum	
114.020	011	EPA 6020	Nickel	
114.020	012	EPA 6020	Selenium	
114.020	013	EPA 6020	Silver	
114.020	014	EPA 6020	Thallium	
114.020	015	EPA 6020	Vanadium	
114.020	016	EPA 6020	Zinc	
114.103	001	EPA 7196A	Chromium (VI)	
114.140	001	EPA 7470A	Mercury	
114.141	001	EPA 7471A	Mercury	
114.221	001	EPA 9012A	Cyanide, Total	
114.240	001	EPA 9040B	Corrosivity - pH Determination	
114.241	001	EPA 9045C	Corrosivity - pH Determination	
114.250	001	EPA 9056	Fluoride	

115 - Extraction Test of Hazardous Waste

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

116 - Volatile Organic Chemistry of Hazardous Waste

116.080	000	EPA 8260B	Volatile Organic Compounds
116.080	001	EPA 8260B	Acetone
116.080	003	EPA 8260B	Acrolein

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116.080	004	EPA 8260B	Acrylonitrile
116.080	005	EPA 8260B	Allyl Alcohol
116.080	006	EPA 8260B	Allyl Chloride
116.080	007	EPA 8260B	Benzene
116.080	010	EPA 8260B	Bromochloromethane
116.080	011	EPA 8260B	Bromodichloromethane
116.080	012	EPA 8260B	Bromoform
116.080	013	EPA 8260B	Bromomethane
116.080	015	EPA 8260B	Carbon Disulfide
116.080	016	EPA 8260B	Carbon Tetrachloride
116.080	018	EPA 8260B	Chlorobenzene
116.080	019	EPA 8260B	Chloroethane
116.080	020	EPA 8260B	2-Chloroethyl Vinyl Ether
116.080	021	EPA 8260B	Chloroform
116.080	022	EPA 8260B	Chloromethane
116.080	023	EPA 8260B	Chloroprene
116.080	026	EPA 8260B	Dibromochloromethane
116.080	027	EPA 8260B	Dibromochloropropane
116.080	028	EPA 8260B	1,2-Dibromoethane
116.080	029	EPA 8260B	Dibromofluoromethane
116.080	030	EPA 8260B	Dibromomethane
116.080	031	EPA 8260B	1,2-Dichlorobenzene
116.080	032	EPA 8260B	1,3-Dichlorobenzene
116.080	033	EPA 8260B	1,4-Dichlorobenzene
116.080	035	EPA 8260B	trans-1,4-Dichloro-2-butene
116.080	036	EPA 8260B	Dichlorodifluoromethane
116.080	037	EPA 8260B	1,1-Dichloroethane
116.080	038	EPA 8260B	1,2-Dichloroethane
116.080	039	EPA 8260B	1,1-Dichloroethene
116.080	040	EPA 8260B	trans-1,2-Dichloroethene
116.080	041	EPA 8260B	cis-1,2-Dichloroethene
116.080	042	EPA 8260B	1,2-Dichloropropane
116.080	043	EPA 8260B	1,3-Dichloropropane
116.080	044	EPA 8260B	2,2-Dichloropropane
116.080	045	EPA 8260B	1,1-Dichloropropene
116.080	046	EPA 8260B	cis-1,3-Dichloropropene
116.080	047	EPA 8260B	trans-1,3-Dichloropropene
116.080	050	EPA 8260B	1,4-Dioxane
116.080	053	EPA 8260B	Ethylbenzene
116.080	055	EPA 8260B	Ethyl Methacrylate
116.080	056	EPA 8260B	Hexachlorobutadiene

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116.080	058	EPA 8260B	2-Hexanone (MBK)
116.080	059	EPA 8260B	Iodomethane
116.080	060	EPA 8260B	Isobutyl Alcohol
116.080	062	EPA 8260B	Methacrylonitrile
116.080	064	EPA 8260B	Methyl tert-butyl Ether (MTBE)
116.080	065	EPA 8260B	Methylene Chloride
116.080	066	EPA 8260B	Methyl Ethyl Ketone
116.080	067	EPA 8260B	Methyl Methacrylate
116.080	068	EPA 8260B	4-Methyl-2-pentanone (MIBK)
116.080	069	EPA 8260B	Naphthalene
116.080	078	EPA 8260B	Propionitrile
116.080	081	EPA 8260B	1,1,1,2-Tetrachloroethane
116.080	082	EPA 8260B	1,1,2,2-Tetrachloroethane
116.080	083	EPA 8260B	Tetrachloroethene
116.080	084	EPA 8260B	Toluene
116.080	086	EPA 8260B	1,2,3-Trichlorobenzene
116.080	087	EPA 8260B	1,2,4-Trichlorobenzene
116.080	088	EPA 8260B	1,1,1-Trichloroethane
116.080	089	EPA 8260B	1,1,2-Trichloroethane
116.080	090	EPA 8260B	Trichloroethene
116.080	091	EPA 8260B	Trichlorofluoromethane
116.080	092	EPA 8260B	1,2,3-Trichloropropane
116.080	093	EPA 8260B	Vinyl Acetate
116.080	094	EPA 8260B	Vinyl Chloride
116.080	095	EPA 8260B	Xylenes, Total
116.080	096	EPA 8260B	tert-Amyl Methyl Ether (TAME)
116.080	097	EPA 8260B	tert-Butyl Alcohol (TBA)
116.080	098	EPA 8260B	Ethyl tert-butyl Ether (ETBE)
116.080	099	EPA 8260B	Bromobenzene
116.080	100	EPA 8260B	n-Butylbenzene
116.080	101	EPA 8260B	sec-Butylbenzene
116.080	102	EPA 8260B	tert-Butylbenzene
116.080	103	EPA 8260B	2-Chlorotoluene
116.080	104	EPA 8260B	4-Chlorotoluene
116.080	105	EPA 8260B	Isopropylbenzene
116.080	106	EPA 8260B	N-propylbenzene
116.080	107	EPA 8260B	Styrene
116.080	108	EPA 8260B	1,2,4-Trimethylbenzene
116.080	109	EPA 8260B	1,3,5-Trimethylbenzene
116.080	120	EPA 8260B	Oxygenates
116.100	001	LUFT GC/MS	Total Petroleum Hydrocarbons - Gasoline

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116.100	002	LUFT GC/MS	Benzene
116.100	003	LUFT GC/MS	Toluene
116.100	004	LUFT GC/MS	Xylenes
116.100	005	LUFT GC/MS	Methyl tert-butyl Ether (MTBE)
116.100	010	LUFT GC/MS	BTEX and MTBE

117 - Semi-volatile Organic Chemistry of Hazardous Waste

117.010	001	EPA 8015B	Diesel-range Total Petroleum Hydrocarbons
117.016	001	LUFT	Diesel-range Total Petroleum Hydrocarbons
117.110	000	EPA 8270C	Extractable Organics
117.110	001	EPA 8270C	Acenaphthene
117.110	002	EPA 8270C	Acenaphthylene
117.110	003	EPA 8270C	Acetophenone
117.110	004	EPA 8270C	2-Acetylaminofluorene
117.110	006	EPA 8270C	4-Aminobiphenyl
117.110	007	EPA 8270C	Aniline
117.110	008	EPA 8270C	Anthracene
117.110	009	EPA 8270C	Aramite
117.110	010	EPA 8270C	Benzidine
117.110	011	EPA 8270C	Benz(a)anthracene
117.110	012	EPA 8270C	Benzo(b)fluoranthene
117.110	013	EPA 8270C	Benzo(k)fluoranthene
117.110	014	EPA 8270C	Benzo(g,h,i)perylene
117.110	015	EPA 8270C	Benzo(a)pyrene
117.110	016	EPA 8270C	Benzoic Acid
117.110	018	EPA 8270C	Benzyl Alcohol
117.110	019	EPA 8270C	Benzyl Butyl Phthalate
117.110	020	EPA 8270C	bis(2-chloroethoxy)methane
117.110	021	EPA 8270C	bis(2-chloroethyl) Ether
117.110	022	EPA 8270C	Bis(2-chloroisopropyl) Ether
117.110	023	EPA 8270C	Di(2-ethylhexyl) Phthalate
117.110	024	EPA 8270C	4-Bromophenyl Phenyl Ether
117.110	025	EPA 8270C	Carbazole
117.110	026	EPA 8270C	4-Chloroaniline
117.110	027	EPA 8270C	4-Chloro-3-methylphenol
117.110	028	EPA 8270C	1-Chloronaphthalene
117.110	029	EPA 8270C	2-Chloronaphthalene
117.110	030	EPA 8270C	2-Chlorophenol
117.110	031	EPA 8270C	4-Chlorophenyl Phenyl Ether
117.110	032	EPA 8270C	Chrysene
117.110	035	EPA 8270C	Dibenz(a,j)acridine
117.110	036	EPA 8270C	Dibenz(a,h)anthracene

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

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117.110	088	EPA 8270C	1,4-Naphthoquinone
117.110	089	EPA 8270C	1-Naphthylamine
117.110	090	EPA 8270C	2-Naphthylamine
117.110	092	EPA 8270C	2-Nitroaniline
117.110	093	EPA 8270C	3-Nitroaniline
117.110	094	EPA 8270C	4-Nitroaniline
117.110	095	EPA 8270C	Nitrobenzene
117.110	096	EPA 8270C	2-Nitrophenol
117.110	097	EPA 8270C	4-Nitrophenol
117.110	098	EPA 8270C	N-nitroso-di-n-butylamine
117.110	099	EPA 8270C	N-nitrosodiethylamine
117.110	100	EPA 8270C	N-nitrosodimethylamine
117.110	101	EPA 8270C	N-nitroso-di-n-propylamine
117.110	102	EPA 8270C	N-nitrosodiphenylamine
117.110	103	EPA 8270C	N-nitrosomethylethylamine
117.110	104	EPA 8270C	N-nitrosomorpholine
117.110	105	EPA 8270C	N-nitrosopiperidine
117.110	106	EPA 8270C	N-nitrosopyrrolidine
117.110	107	EPA 8270C	5-Nitro-o-toluidine
117.110	108	EPA 8270C	Pentachlorobenzene
117.110	109	EPA 8270C	Pentachloronitrobenzene
117.110	110	EPA 8270C	Pentachlorophenol
117.110	111	EPA 8270C	Phenacetin
117.110	112	EPA 8270C	Phenanthrene
117.110	113	EPA 8270C	Phenol
117.110	114	EPA 8270C	1,4-Phenylenediamine
117.110	116	EPA 8270C	2-Picoline
117.110	119	EPA 8270C	Pyrene
117.110	120	EPA 8270C	Pyridine
117.110	122	EPA 8270C	Safrole
117.110	124	EPA 8270C	1,2,4,5-Tetrachlorobenzene
117.110	125	EPA 8270C	2,3,4,6-Tetrachlorophenol
117.110	128	EPA 8270C	o-Toluidine
117.110	129	EPA 8270C	1,2,4-Trichlorobenzene
117.110	130	EPA 8270C	2,4,5-Trichlorophenol
117.110	131	EPA 8270C	2,4,6-Trichlorophenol
117.110	132	EPA 8270C	1,3,5-Trinitrobenzene
117.111	015	EPA 8270C	Chlorobenzilate
117.111	021	EPA 8270C	Diallate
117.111	026	EPA 8270C	Dimethoate
117.111	039	EPA 8270C	Isodrin

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117.111	054	EPA 8270C	Parathion Ethyl
117.111	055	EPA 8270C	Parathion Methyl
117.111	056	EPA 8270C	Phorate
117.111	058	EPA 8270C	Sulfotepp
117.111	061	EPA 8270C	O,O,O-triethyl Phosphorothioate
117.111	073	EPA 8270C	Polynuclear Aromatic Hydrocarbons
117.111	074	EPA 8270C	Adipates
117.111	076	EPA 8270C	Phthalates
117.111	076	EPA 8270C	Other Extractables
117.120	000	EPA 8280A	Dioxins and Dibenzofurans
117.120	001	EPA 8280A	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
117.120	002	EPA 8280A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
117.120	003	EPA 8280A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	004	EPA 8280A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	005	EPA 8280A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	006	EPA 8280A	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
117.120	007	EPA 8280A	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
117.120	008	EPA 8280A	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
117.120	009	EPA 8280A	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	010	EPA 8280A	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	011	EPA 8280A	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
117.120	012	EPA 8280A	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	013	EPA 8280A	Total TCDD
117.120	014	EPA 8280A	Total PeCDD
117.120	015	EPA 8280A	Total HxCDD
117.120	016	EPA 8280A	Total TCDF
117.120	017	EPA 8280A	Total PeCDF
117.120	018	EPA 8280A	Total HxCDF
117.120	019	EPA 8280A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
117.120	020	EPA 8280A	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
117.120	021	EPA 8280A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
117.120	022	EPA 8280A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
117.120	023	EPA 8280A	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
117.120	024	EPA 8280A	Total HpCDD
117.120	025	EPA 8280A	Total HpCDF
117.130	000	EPA 8290	Dioxins and Dibenzofurans
117.130	001	EPA 8290	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
117.130	002	EPA 8290	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
117.130	003	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130	004	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130	005	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)

As of 12/21/2010, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

UMP013070

Testamerica West Sacramento

Certificate No.: 01119CA
Renew Date: 1/31/2012

117.130	006	EPA 8290	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
117.130	007	EPA 8290	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
117.130	008	EPA 8290	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
117.130	009	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	010	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	011	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
117.130	012	EPA 8290	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	013	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
117.130	014	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
117.130	015	EPA 8290	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
117.130	016	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
117.130	017	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
117.170	000	EPA 8330	Nitroaromatics and Nitramines
117.170	001	EPA 8330	4-Amino-2,6-dinitrotoluene
117.170	002	EPA 8330	2-Amino-4,6-dinitrotoluene
117.170	003	EPA 8330	1,3-Dinitrobenzene
117.170	004	EPA 8330	2,4-Dinitrotoluene
117.170	005	EPA 8330	2,6-Dinitrotoluene
117.170	006	EPA 8330	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
117.170	007	EPA 8330	Methyl-2,4,6-trinitrophenylnitramine
117.170	008	EPA 8330	Nitrobenzene
117.170	009	EPA 8330	2-Nitrotoluene
117.170	010	EPA 8330	3-Nitrotoluene
117.170	011	EPA 8330	4-Nitrotoluene
117.170	012	EPA 8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
117.170	013	EPA 8330	1,3,5-Trinitrobenzene
117.170	014	EPA 8330	2,4,6-Trinitrotoluene
117.171	000	EPA 8330A	Nitroaromatics and Nitramines
117.171	001	EPA 8330A	4-Amino-2,6-dinitrotoluene
117.171	002	EPA 8330A	2-Amino-4,6-dinitrotoluene
117.171	003	EPA 8330A	1,3-Dinitrobenzene
117.171	004	EPA 8330A	2,4-Dinitrotoluene
117.171	005	EPA 8330A	2,6-Dinitrotoluene
117.171	006	EPA 8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
117.171	007	EPA 8330A	Methyl-2,4,6-trinitrophenylnitramine
117.171	008	EPA 8330A	Nitrobenzene
117.171	009	EPA 8330A	2-Nitrotoluene
117.171	010	EPA 8330A	3-Nitrotoluene
117.171	011	EPA 8330A	4-Nitrotoluene
117.171	012	EPA 8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
117.171	013	EPA 8330A	1,3,5-Trinitrobenzene

Testamerica West Sacramento

Certificate No.: 01119CA
Renew Date: 1/31/2012

117.171	014	EPA 8330A	2,4,6-Trinitrotoluene
117.210	000	EPA 8081A	Organochlorine Pesticides
117.210	001	EPA 8081A	Aldrin
117.210	002	EPA 8081A	a-BHC
117.210	003	EPA 8081A	b-BHC
117.210	004	EPA 8081A	d-BHC
117.210	005	EPA 8081A	g-BHC (Lindane)
117.210	006	EPA 8081A	Captafol
117.210	007	EPA 8081A	a-Chlordane
117.210	008	EPA 8081A	g-Chlordane
117.210	009	EPA 8081A	Chlordane (tech.)
117.210	010	EPA 8081A	Chlorobenzilate
117.210	013	EPA 8081A	4,4'-DDD
117.210	014	EPA 8081A	4,4'-DDE
117.210	015	EPA 8081A	4,4'-DDT
117.210	016	EPA 8081A	Diallate
117.210	020	EPA 8081A	Dieldrin
117.210	021	EPA 8081A	Endosulfan I
117.210	022	EPA 8081A	Endosulfan II
117.210	023	EPA 8081A	Endosulfan Sulfate
117.210	024	EPA 8081A	Endrin
117.210	025	EPA 8081A	Endrin Aldehyde
117.210	026	EPA 8081A	Endrin Ketone
117.210	027	EPA 8081A	Heptachlor
117.210	028	EPA 8081A	Heptachlor Epoxide
117.210	031	EPA 8081A	Isodrin
117.210	033	EPA 8081A	Methoxychlor
117.210	039	EPA 8081A	Toxaphene
117.220	000	EPA 8082	PCBs
117.220	001	EPA 8082	PCB-1016
117.220	002	EPA 8082	PCB-1221
117.220	003	EPA 8082	PCB-1232
117.220	004	EPA 8082	PCB-1242
117.220	005	EPA 8082	PCB-1248
117.220	006	EPA 8082	PCB-1254
117.220	007	EPA 8082	PCB-1260

As of 12/21/2010, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

UMP013072

Appendix E

Barr Field Standard Operating Procedures

STANDARD OPERATING PROCEDURE

Calculation of Purge Volumes for Groundwater Sampling Wells

Revision 3

March 25, 2010

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord		Andrea Nord	3-25-10

Print	Field Technician(s)	Signature	Date
Kim Johannessen		Kim Johannessen	3-25-10

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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	KSJ	Date:	4/4/2011
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Calculation of Purge Volumes for Groundwater Sampling Wells

Purpose

The purpose of this procedure is to describe the methods used in calculating and measuring purge volumes.

Applicability

The procedure applies to the amount of water that is purged out of a well before sampling can occur.

Definition

Purge volume is a specific amount of water removed from a well before sampling.

Equipment

Calculator
Field Logbook

Reference

Groundwater and Surface Water Sampling Procedures by Barr Engineering Co.

Discussion

The procedure will show that a variety of calculations must be carried out before purge volumes are known.

Responsibilities

The sampling technician(s) conducting the purging of the well are responsible for the purge volumes.

Procedure

Calculating and Measuring Purge Volumes

1. Calculate the volume of standing water in the well (using the following equation):

   Note: There is a precalculated chart to determine the volume of standing water (Figure 1).

a. $V = (\pi)(r^2)(h)$

V	=	Volume in cubic feet of standing water
π	=	3.14
r	=	Radius of the well casing or hole (in feet)
h	=	Height of the column of water in the well (in feet)

(h = water level - total well depth)

2. Convert the volume of standing water in the well from cubic feet to gallons using the following equation:

   a. WV = (V)(7.48)

   WV = Well volume in gallons

3. Determine the amount of water to be purged (using this equation):

   a. VP = (WV)(NWV)

   VP = Volume of water pumped
   WV = Well volume in gallons
   NWV = Number of well volumes that monitoring plan requires to be purged

4. Estimate the time it will take for the well to be purged (time pumped).

   a. Determine the flow rate of the well.

   Flow meter— If installed on well, it can be simply read to obtain the flow rate.

   No-flow meter— The rate can be obtained by using a container marked in volumes and calculating the amount of time it takes for the container to fill with purge water.

   Note: See Standard Operating Procedures for Measuring Well Pumping Rates.

   b. Divide the volume of water pumped in the well by the flow rate.

Stabilization Test Measurements

Collection of stabilization test measurements shall begin at the same time as groundwater purging prior to sample collection is initiated. Well stabilization measurements will be collected and recorded at the start of the purging process and once every well volume during the purging process, with a minimum of one measurement collected per well volume removed. A well volume will be measured as the volume of water that occurs in each well from the base of the well to the water level measurement collected prior to initiation of purging. Once three well volumes have been removed, the well may be sampled after three consecutive measurements, collected at the intervals described above, are within the ranges presented below:

Specific Conductance:	±5% of the most recent reading (temperature corrected)
pH	±5% of the most recent reading (in pH units)
Temperature	±5% of the most recent reading (in degrees Celsius)
Oxidation Reduction Potential (Eh)	±20 mV of the most recent reading

Collect samples only after a minimum of three water-column volumes have been purged and stabilization of field water-quality parameters has been demonstrated by meeting the target criteria defined in the preceding paragraph. Field staff shall check operator procedures, equipment functioning, and well construction information for potential problems. In particular, field staff shall re-evaluate whether or not water is being withdrawn from the appropriate depth to effectively evacuate the well.

If all the checks produce no new insight, a decision might be made to collect samples after five or more water-column volumes have been purged even if field measurements have not stabilized. If the well was purged dry, it shall be allowed to recharge and the samples will be collected.

However, if either circumstance applies, the following procedure is required: Before authorizing the laboratory to analyze the samples, the meaningfulness and value of completing laboratory analysis of the sampling suite will be evaluated by reviewing the results of field measurements, well construction data, site hydrology, etc. Where such data is presented, it will be clearly documented that stabilization was not achieved; at a minimum, this fact will be reported on the Field Log Data Sheets and in the Field Sampling Report.

Documentation

The technicians will document flow rate, well volume, time pumped, volume pumped, water level, total well depth and stabilization test measurements on the field log data sheet.

Figures

Figure 1 – Volume of Water in Casing or Hole

Attachments

Attachment 1 – Field Log Data Sheet

Figure 1

Volume of Water in Casing or Hole

Diameter of Casing or Hole (In)	Gallons per Foot of Depth	Cubic Feet per Foot of Depth	Liters per Meter of Depth	Cubic Meters per Meter of Depth
1	0.041	0.0055	0.509	0.509×10^{-3}
1½	0.092	0.0123	1.142	1.142×10^{-3}
2	0.163	0.0218	2.024	2.024×10^{-3}
2½	0.255	0.0341	3.167	3.167×10^{-3}
3	0.367	0.0491	4.558	4.558×10^{-3}
3½	0.500	0.0668	6.209	6.209×10^{-3}
4	0.653	0.0873	8.110	8.110×10^{-3}
4½	0.826	0.1104	10.26	10.26×10^{-3}
5	1.020	0.1364	12.67	12.67×10^{-3}
5½	1.234	0.1650	15.33	15.33×10^{-3}
6	1.469	0.1963	18.24	18.24×10^{-3}
7	2.000	0.2673	24.84	24.84×10^{-3}
8	2.611	0.3491	32.43	32.43×10^{-3}
9	3.305	0.4418	41.04	42.04×10^{-3}
10	4.080	0.5454	50.67	50.67×10^{-3}
11	4.937	0.6600	61.31	61.31×10^{-3}
12	5.875	0.7854	72.96	72.96×10^{-3}
14	8.000	1.069	99.35	99.35×10^{-3}
16	10.44	1.396	129.65	129.65×10^{-3}
18	13.22	1.767	164.18	164.18×10^{-3}
20	16.32	2.182	202.68	202.68×10^{-3}
22	19.75	2.640	245.28	245.28×10^{-3}
24	23.50	3.142	291.85	291.85×10^{-3}
26	27.58	3.687	342.52	342.52×10^{-3}
28	32.00	4.276	397.41	397.41×10^{-3}
30	36.72	4.909	456.02	456.02×10^{-3}
32	41.78	5.585	518.87	518.87×10^{-3}
34	47.16	6.305	585.68	585.68×10^{-3}
36	52.88	7.069	656.72	656.72×10^{-3}

1 gallon = 3.785 liters
1 meter = 3.281 feet
1 gallon water weighs 8.33 lbs. = 3.785 kilograms
1 liter water weighs 1 kilogram = 2.205 lbs.
1 gallon per foot of depth = 12.419 liters per foot of depth
1 gallon per meter of depth = 12.419×10^{-3} cubic meters per meter of depth

Attachment 1
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:	**Monitoring Point:**
Location:	**Date:**
Project #:	**Sample Time:**

GENERAL DATA		STABILIZATION TEST						
Barr lock:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						

Others present:

WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)

CASING & CAP: COLLAR: LOCK: OTHER:

MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:

VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-

oil,grease- bacteria- total metal- filtered metal- methane- filter-

Others:

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

STANDARD OPERATING PROCEDURE

Collection of Quality Control Samples

Revision 4

March 1, 2011

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord			3-1-11

Print	Field Technician(s)	Signature	Date
Marta Nelson			3-1-11

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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for the Collection of Quality Control Samples

Purpose

To describe the procedures used in the collection of quality control samples; equipment blanks, masked duplicate samples (i.e. field duplicate samples), matrix spikes and matrix spike duplicate and trip blank samples.

Applicability

This procedure applies to sample definition, collection and handling techniques used by the technician(s) and the laboratory in regards to quality control samples.

Equipment

Laboratory certified containers appropriate for the required analysis
Nitrile or vinyl gloves
Bailer
Chain of custody
Sample Labels
Sample containers/media
Analyte-free water

Definitions

Equipment Blank ***(also commonly referred to as*** Field blank) samples (or Rinsate Blanks) are prepared on-site. The field technician pours analyte-free water through decontaminated sample collection equipment (bailer or pump, hand-trowl, etc.) and collects the "rinsate" in the appropriate sample container(s). The equipment blank samples will be handled in the same manner as the sample group for which they are intended (i.e. blanks will be stored and transported with the sample group). The purpose of the equipment blank sample is to determine whether the field or sample transporting procedures and environments have contaminated the sample.

Field (or Masked) Duplicates Field duplicate samples are: two identical aliquots of a sample, collected in separate sample bottles at the same time, and placed under identical circumstance using a duel inlet sampler or by splitting a larger aliquot. They are treated exactly the same throughout field and laboratory procedures. Analyses of field duplicate samples give a measure of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Matrix spike and matrix spike duplicates are two identical aliquots of an environmental sample to which known quantities of analytes are added (spiked) in the laboratory. The MS and MSD are prepared and analyzed exactly like their project (native) sample aliquot. Generally, it is required that three separate sample aliquots are collected in the field for each analysis. One aliquot is analyzed to determine the background concentrations in the project sample, a second sample aliquot serves as the MS sample and the third sample aliquot serves as the MSD. The purpose of the MS and MSD is to quantify the bias and precision caused by the sample matrix. The background concentrations of the analytes in the sample matrix must be determined in a

separate aliquot and the measured values in the MS and MSD corrected for background concentrations.

Trip Blank. A trip blank sample is made up of contaminant-free water and is prepared prior to sampling event by the laboratory providing the sampling containers. The purpose of the trip blank sample is to determine if contamination has occurred from any of the following sources; improper sampling container cleaning, contaminated source water, sample contamination during storage and transportation due to exposure to contaminants or any other environmental conditions during sampling. Trip blank samples apply to volatile organic compound (VOC) samples only.

References

Procedures for Ground Water Monitoring, Minnesota Pollution Control Agency Guidelines, December 1986

EPA: Title 40 of the Code of Federal Regulations

Responsibilities

The sampling technician(s) are responsible for the accurate collection of quality control samples. The laboratory is responsible for the accurate set-up and analysis of quality control samples.

Procedure

The ratio of quality control samples are generally one equipment or rinsate blank/field duplicate/MS/MSD per twenty samples, however, specific project requirements may require alternative sampling frequencies for QC samples for each project.

A. Field (masked) duplicate sample:

1. Collect samples by rotating sampling containers from original sample to the field) duplicate sample (using the same exact methods for both).

2. Preserve, store, and transport the field duplicate sample in the same manner as the original sample.

3. Submit the field duplicate sample to the laboratory for the same analyses as the original sample.

B. Trip blank Samples:

1. Trip blank samples are sealed prior to sampling (prepared by the laboratory performing the VOC analysis).

2. Transport trip blank samples to the site in the sample storage cooler containing the VOC vials used for collecting project samples for the sampling event.

3. Trip blank sample containers are not to be opened in the field.

4. Transport trip blank samples back to the laboratory in the sample storage cooler. There must be one set of trip blank samples per sample cooler containing VOC samples from the Site.

5. The trip blanks should be listed on the chain-of-custody along with the other samples and the analysis required. (Trip blanks are only provided for VOCs analyses).

   Note: Labeling of all sample blank containers follow the SOP for the collection of groundwater, soil, or sediment samples.

C. Equipment or rinsate blanks:

1. Obtain the appropriate sampling containers and desired amount (analyte-free) water from the laboratory. (Generally, blanks are taken for each parameter of interest.)

2. Pour analyte-free water through decontaminated sample collection equipment (bailer or pump, hand-trowl, etc.) and collecting the "rinsate" in the appropriate sample containers.

3. Seal the field blank sample containers and store with other samples collected (should be handled in the same manner).

D. Filtered equipment blank:

1. Pour or pump (analyte-free) water into and/or through the groundwater sampling filter.

2. Begin filtering (as described in the standard operating procedure for Filtering Groundwater Samples).

Note: The filtered equipment blank is usually conducted for dissolved metals or dissolved organic carbon samples only.

Sample Storage

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of strapping tape. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Disposal

All waste generated by this process 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 environmental pollution.

Documentation

The technician(s) will document the type and number of quality control samples collected during each field event. All sample information will be documented in the field notebook, field log data sheet and chain-of-custody record.

Attachments

Attachment 1 – Field Log Data Sheet
Attachment 2 – Chain of Custody Form
Attachment 3 – Sample Label – Example
Attachment 4 – Custody Seal – Example

UMP013084

Attachment 1
Field Log Data Sheet – Soil Samples

Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:									Number of Containers/ Analysis																				
Location:																													
Project #:																													
Project Name:																													
Sample Identification	Collection		Matrix			Type																							
	Date	Time	Soil	Sludge		Grab	Comp.	QC	2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:					
1.																													
2.																													
3.																													
4.																													
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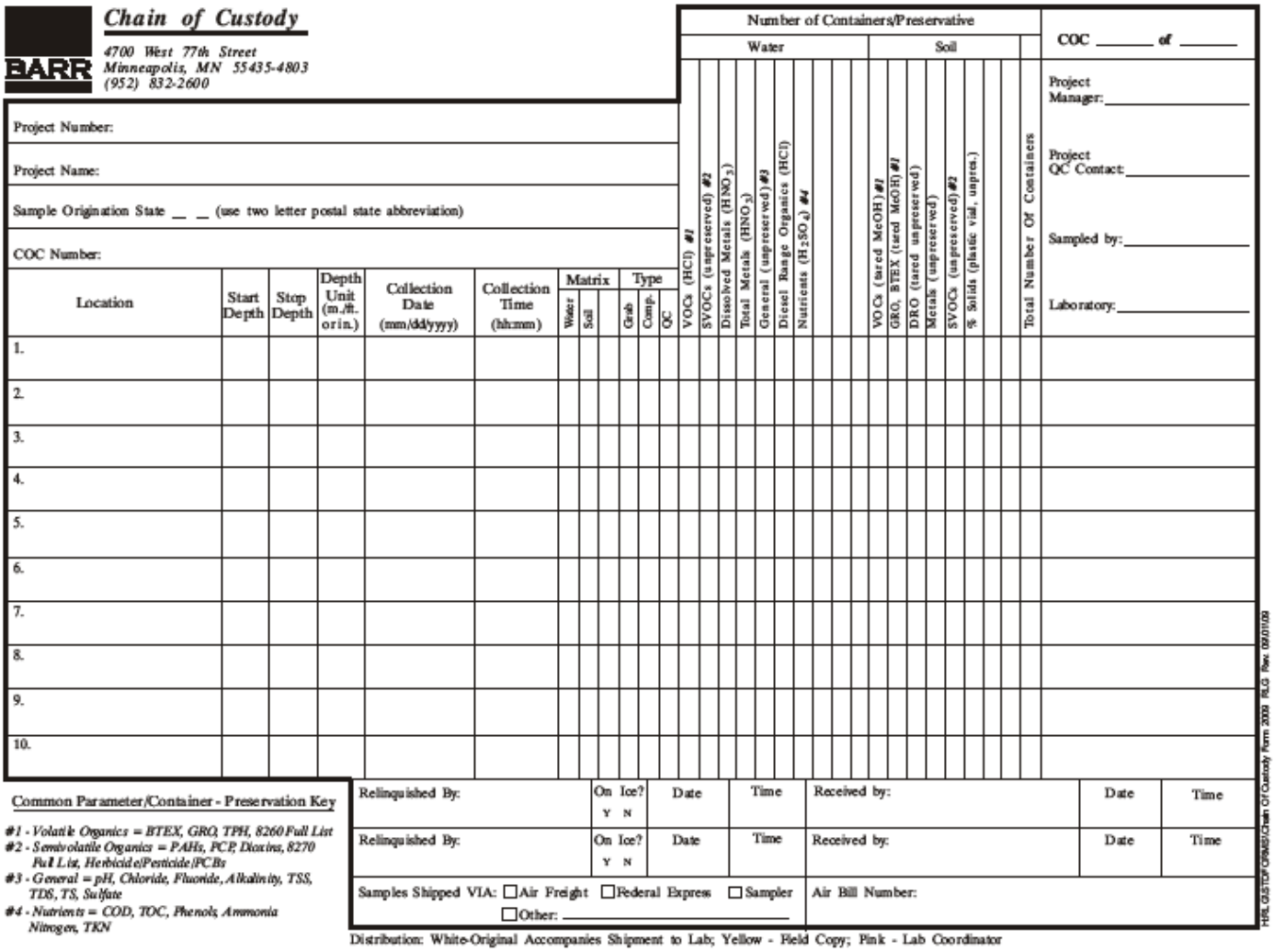
Attachment 2
Chain of Custody Form



Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Number of Containers/Preservative

Water: VOCs (HCl) #1; SVOCs (unpreserved) #2; Dissolved Metals (HNO_3); Total Metals (HNO_3); General (unpreserved) #3; Diesel Range Organics (HCl); Nutrients (H_2SO_4) #4

Soil: VOCs (tared MeOH) #1; GRO, BTEX (tared MeOH) #1; DRO (tared unpreserved); Metals (unpreserved); SVOCs (unpreserved) #2; % Solids (plastic vial, unpres.)

Total Number Of Containers

COC _____ of _____

Project Manager: _____

Project QC Contact: _____

Sampled by: _____

Laboratory: _____

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC
1.										
2.										
3.										
4.										
5.										
6.										
7.										
8.										
9.										
10.										

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Penticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐Air Freight ☐Federal Express ☐Sampler ☐Other: _____ Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

HRL GUSTOFCR65/Chain Of Custody Form 2009 RLG Rev. 09/01/09

Attachment 3
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

Preservative: _____

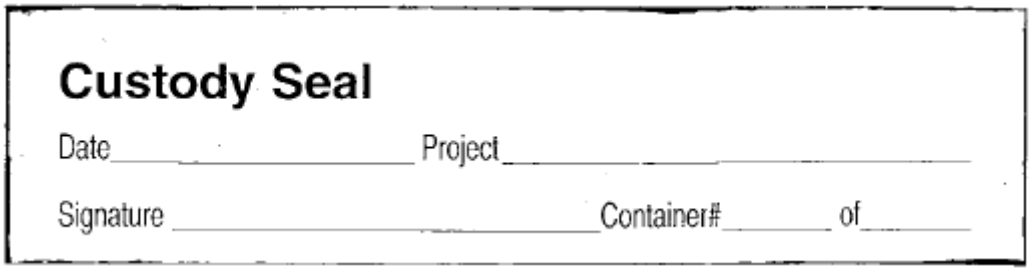
Sampled By: _____

Sample Location: _____

_____

UMP013087

Attachment 4
Custody Seal



STANDARD OPERATING PROCEDURE

Decontamination of Field Sampling Equipment

Revision 3

April 10, 2011

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord			4/10/11

Print	Field Technician(s)	Signature	Date
John W. Juntilla			4/10/11

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Decontamination of Monitoring Well Sampling Equipment

Purpose

The purpose of this procedure is to define the process used for decontaminating all sampling-related equipment including pumps, meters, and materials coming into contact with actual sampling equipment or with sampling personnel. Bailers, protective gear, and filtration devices will be discarded after one use. Stainless steel bailers are used once and returned to an independent laboratory for decontamination.

Applicability

This procedure is applicable to all personnel who are collecting samples and/or decontaminating sampling and field equipment

Equipment

Alconox®
Scrub brush made of inert materials
Distilled or Deionized rinse water
Bucket
Field Log Data Sheets
Field Log Cover Sheets
Field Log Data Reports

Responsibilities

The Equipment Technician is responsible for ensuring all field equipment has been thoroughly decontaminated and prepared for use out in the field. The field technician(s) are responsible for decontamination in the field at each individual sampling point. The field technican(s) are responsible for ensuring adherence to any IDW project-specific requirements as set forth in the QAPP or SAP are met.

Procedure

Decontamination of monitoring well equipment will be performed by the field technician(s) before sampling and after working at each sampling point. All equipment will be handled in a manner that minimizes cross-contamination between points. After cleaning, the equipment will be visibly inspected to detect any residues or other substances that may exist after normal cleaning. If inspection reveals that decontamination was insufficient, the decontamination procedures will be repeated.

Equipment will be decontaminated in the following manner:

1. Equipment that does **not** contact sample water or the inside of the well:

    a. Rinse with clean control water.

b. Inspect for remaining particles or surface film and repeat cleaning and rinse procedures if necessary.

2. Equipment that contacts sample water or the inside of the well:

   a. Clean (inside and outside where possible) with an Alconox®/clean-water solution applied with a scrub brush made of inert materials.

   b. Rinse with clean control water, containerized with other IDW if required by the SAP or QAPP.

   c. Inspect for remaining particles or surface film and repeat cleaning and rinse procedures if necessary.

   d. Shake off remaining water and allow to air dry.

The internal surfaces of pumps and tubing that cannot be adequately cleaned by the above methods alone will also be cleaned by circulating decontamination fluids through them. The fluids will be circulated through this equipment in the order shown above. Special care will be exercised to ensure that the "rinse" fluids will be circulated in sufficient quantities to completely flush out contaminants and detergents.

When transporting or storing equipment after cleaning, the equipment will be protected in a manner that minimizes the potential for contamination.

Disposal

All waste generated by this process 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 environmental pollution.

Documentation

The technician(s) will document the field equipment decontamination procedures on Field Log Data Sheets, Field Log Cover Sheets, and Field Log Data Reports or a project dedicated Field Log book.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Cover Sheet
Attachment 3: Field Log Data Sheet

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

UMP013092

Attachment 2
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

Attachment 3
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample Time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)								
CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

STANDARD OPERATING PROCEDURE

Direct-Push Soil and Groundwater Sample Collection (Geoprobe®)

Revision 4

March 23, 2010

Approved By:

Andrea Nord		3/23/10
Print	QA Manager(s) Signature	Date
KEVIN McGILP		3/23/10
Print	Field Technician(s) Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	JWJ	Date:	4-10-11
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Direct-Push Soil and Groundwater Sample Collection (Geoprobe®)

Purpose

The purpose of this standard operating procedure (SOP) is to describe the procedures for the collection of soil and/or groundwater samples when Geoprobe® field methods are used.

Applicability

This SOP will be utilized wherever direct-push (i.e., Geoprobe®) methods are employed for the retrieval of soil or groundwater from designated sampling locations.

Equipment

Direct-push soil sampling rig
Direct-push sampler liner
Direct-push probe
Extension rods
Screen (four-foot lengths)
Polyethylene tubing
Pump (peristaltic or vacuum)
Pre-cleaned-certified Sampling Containers
Alconox®
Deionized or tap water
Stainless steel spoons, scoops or trowels
Clean pair of surgical gloves
Appropriate personal protective equipment
Field notebook and/or Field Log Data Sheets
Chain of Custody Form
Sample Labels
Coolers
Bagged ice
Tape
Field balance (for soils)
Water-proof ink pen

References

Procedures for Ground Water Monitoring, Minnesota Pollution Control Agency Guidelines, December 1986
EPA: Title 40 of the Code of Federal Regulations

Responsibilities

The environmental technician(s) or geologist is responsible for the proper collection of soil and water samples, sample identification, quality control procedures, and documentation.

UMP013096

Procedure

1. Approximately one week before the sampling event, the appropriate sample containers should be ordered from the laboratory.

2. Before leaving for the site, account for all the containers.

3. When the sample is ready to be collected label the containers with the following information:

   - Project number
   - Location sampled
   - Individual collecting the samples
   - Date and time of sample collection
   - Sample analysis (if required by the laboratory)

   Note: Use an indelible permanent pen to avoid ink bleeding.

4. Put on a new pair of disposable sampling gloves at each sampling location.

Soil Sampling with a Direct-push Soil Boring Rig:

A. Preparation of Soil Sampling Equipment

All soil sampling equipment will be carefully cleaned before use. All sampling tools including stainless steel spoons/scoops/trowels will be cleaned before use and in between sampling locations by cleaning with deionized or tap water and Alconox®, using a brush if necessary to remove particulate matter or films and rinsed thoroughly with deionized water. To prevent sample cross-contamination, the sampler will put on a new pair of disposable sampling gloves at each sampling location. Direct-push sampler liners (soils) are one-time use and disposable.

B. Soil Sample Collection

Soils are generally continuously sampled using the direct push method. This method generally utilizes steel drive rods and a 2-inch outside diameter (O.D.) soil core sampler with a dedicated 1.75-inch inside diameter (I.D) removable acetate liner. The probe rods and sampling unit are driven to the desired sampling depth by the static weight of the carrier vehicle and hydraulic hammer percussion. Two or four-foot sample cores are typically collected. The assembly is brought to the surface and the soil sample is exposed by cutting open the acetate plastic liner. In most investigations, the entire cores are field screened for moisture, odor, oil sheen, discoloration and the presence of organic soil vapors and classified in accordance with ASTM D-2488, Standard Practice for Description and Identification of Soils (Visual/Manual Method.) Soil sample field screening procedures are described in a separate standard operating procedure

1. Collecting Volatile Organic Samples

   It is important to note that there are different jar sizes and sampling media available for collecting a soil sample for volatile organic compounds (VOCs). The table below

describes the sample volumes and preservation techniques for the most common sampling media.

Summary of Typical Sampling Media and Soil Volumes Used for Volatile Organic Compound Determination			
VOC Sample Media	Preservative	Volume of Preservative (mL)	Volume of Sample (g)
2 oz. glass jar with PTFE-lined lid	MeOH, cool 4 °	10	10
	MeOH, cool 4 °	25	25
4 oz. glass jar with PTFE-lined lid	MeOH, cool 4 °	10	10
	MeOH, cool 4 °	25	25
Encore® Sampler			
5 gram device	Freeze or extrude into chemical preservative	Maintain a 1:1 ratio of soil to preservative if chemical preservation is used.	5
25 gram device	Freeze or extrude into chemical preservative	Maintain a 1:1 ratio of soil to preservative if chemical preservation is used.	25
Terracore® Kit			
1 MeOH and 2 water preserved glass vial	MeOH, cool 4 °	5	5
	Water Submersion, cool 4 °	5	5
1 MeOH and 2 sodium bisulfite preserved glass vials	MeOH, cool 4 °	5	5
	Sodium Bisulfite, cool 4 °	5	5

Note: Samples for volatile analysis should be collected prior to any other analysis.

A. Before beginning the collection of VOC soil samples, verify field balance using a 50 gram weight. If the balance is off by ± 5 grams, recalibrate the instrument following the manufacturer's recommendations.

B. Cut open the liner using a knife or similar utensil.

C. Because certain regulations do not allow a weighed sample to be submitted for analysis, it is recommended that the desired weight of soil be weighed using a field balance to gauge the approximate volume of soil (i.e. typically 5, 10 or 25 grams of soil) required to achieve the appropriate weight required for VOC

UMP013098

analysis. Using a stainless-steel spoon/trowel and a field balance, collect the desired grams of soil in a laboratory-provided tared sample container. Once the volume of soil is approximated, the sample aliquot is discarded. Then, collect another equal aliquot of soil for preservation and analysis.

Depending on the laboratory that supplied the container, methanol may be provided in a snap-cap vial that will be opened and poured over the soil in the pre-tared container or the container will been received with the appropriate volume of methanol already added. In this case, avoid splashing the methanol when adding the soil volume. The VOC ration must be 1:1 soil to methanol.

D. Wipe the jar lip and screw threads to remove soil and ensuring a tight seal with the lid of the container.

E. Cool the sample to approximately 4°C immediately after collection.

2. Collecting Semivolatile Organic or Metals Samples (or any other soil sample)

A. Cut open the liner using a knife or similar utensil.

B. Retrieve sample using a clean stainless steel spoon/trowel. Fill sample jar, wipe the jar lip and screw threads to remove soil and ensuring a tight seal with the lid of the container. No preservatives are required for soil samples except VOCs.

C. Cool the sample to approximately 4°C immediately after collection.

Groundwater Sampling with a Direct-push Soil Boring Rig:

Groundwater samples will be collected by advancing the direct-push probe to the desired sampling depth. When the sampling depth is reached, small diameter extension rods will be run through the steel probe rods to push out the expendable drive point. Next, a one-inch screen (four-foot length) is extended into the formation. Following screen placement, polyethylene tubing is placed into the temporary well, and a peristaltic pump (or equivalent) is used to draw water samples to the surface to be placed in appropriate vials or bottles for laboratory analysis.

After each well is constructed, the probe rods are washed in an Alconox®/water mixture and rinsed with water. The polyethylene tubing is discharged after each sample was collected and new tubing used for the collection of the next sample. The temporary well locations will be abandoned following all State regulations.

Container volume, type, and preservative are important considerations in groundwater sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat examinations. The container type or construction varies with the analysis required: (1) septum-sealed 40-ml glass vial is used for volatile organic compounds; (2) semivolatile analyses usually require a glass container (note—amber-tinted glass prevents sunlight from affecting the sample); and (3) polyethylene containers are used for general parameters, metals, and inorganics. The analytical laboratory will preserve the container before shipment. Preservation and shelf life vary; contact the laboratory to determine if an on-hand container is still useful.

A. Groundwater Sample Collection

1. Volatiles—Use caution because concentrated acid may be present. Do not rinse or overfill glass vials. Hold bottle in one hand, the cap right side up in the other. Pour slowly, avoiding air bubbles and overfilling the vial. Cap tightly, invert the bottle, and tap gently. If any air bubbles appear in the vial, discard and collect sample in a new vial. After collecting the required number of vials (usually sets of 2 or 3, depending on the laboratory), label them with the necessary information, insert them in a Ziplock® plastic bag, and place in a cooler with ice.

2. Semivolatiles—Fill container slowly with a minimum headspace and cap tightly. Do not rinse glass containers. Place container directly in a cooler with ice.

3. Filtered Metals—Typically field filtering of groundwater samples collected from a Geoprobe® boring is not advised. Undeveloped temporary borings of this type will likely contain significant solids that would require several attempts to filter adequately. In these cases, the laboratory(ies) can perform this filtering, if necessary. However, this would require an **unpreserved** aliquot of sample for filtration and preservation (of nitric acid) at the laboratory. Should field filtering be required, see the Barr Engineering Co. Standard Operating Procedure for Filtering Groundwater Samples). Pour sample into metals sample container, minimizing headspace and avoiding spillage. Use caution handling metals containers because of nitric acid. Place directly in a cooler with ice.

4. Other Organics or Inorganics—Containers may contain acid(s), use caution when handling. Fill containers appropriately, rinsing any unpreserved containers three times, minimizing splashing and spillage. Place container directly in a cooler with ice.

Quality Control Samples

The effectiveness of the sample handling techniques is monitored by collecting both preserved and unpreserved field blank samples. For additional information, consult the Barr Engineering Co. SOP for the Collection of Quality Control Samples.

Field (or Masked) duplicate samples will be collected to measure relative sampling (and laboratory) precision. The ratio of quality control samples are generally 1 field blank/field duplicate sample per twenty samples; however, specific project requirements may be determined by the QAPP/SAP for the project. These samples are collected at the same time using the same procedures, equipment, and types of containers as the required samples. They are also preserved in the same manner and are either co-located or split and submitted for the same analyses as the native sample(s).

Trip blank samples are only applicable when sampling/analyzing for volatile organics. Their purpose is to determine if contamination has occurred as a result of improper sample container cleaning, contaminated blank source water, sample contamination during storage and transport due to exposure to volatile organics, or other environmental conditions during sampling and analysis. The water will be free of contaminants. The trip blanks are prepared, sealed and labeled appropriately at the lab, and transported to the field in the same containers as the sample vials. The trip blank samples are not opened in the field. They are transferred

to the coolers designated for volatile sample storage and are transported with the project samples to the analytical laboratory.

Field (or rinsate) blank samples are used to evaluate the effects of sampling cross-contamination caused by inadequately decontaminated equipment. Their purpose is to determine if contamination has occurred as a result of improper equipment cleaning. Field blank samples are prepared onsite by pouring analyte-free water through decontaminated sample collection equipment (bailer, pump, tubing, hoses, stainless-steel bowls, trowels, etc.) and collecting the rinsate in the appropriate sample container. The field blank samples will be handled in the same manner as the sample group for which they are intended (i.e., blanks will be stored and transported with the sample group).

The volume of the sample obtained should be sufficient to perform all required analyses with an additional amount collected to satisfy the needs for quality control, split samples, or repeat examinations. The QA Staff should be consulted for any specific volume requirements.

The elapsed time between sample collection and initiation of each laboratory analysis will fall within a prescribed time frame. Holding times for samples required by this project are prescribed by EPA: Title 40 of the Code of Federal Regulations.

Water and Soil Sample Storage

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Samples will be kept cold (approximately 4°C) until receipt at the laboratory, where they are to be stored in a refrigerated area. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of packing tape. All samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

Interferences/Discussion

Volatile and low-level mercury samples must be collected prior to any other analyses and metals must be collected prior to cyanide samples to avoid possible cross-contamination or other potential data quality issues. After collection, all samples should be handled as few times as possible. Samplers should use extreme care to ensure that samples are not contaminated. If samples are placed in a cooler, samplers should ensure that melted ice cannot cause sample containers to become submerged, as this may result in cross-contamination. Plastic bags, such as Ziplock® bags, should be used when small sample containers (e.g., VOC vials) are placed in coolers to prevent cross-contamination.

Some compounds can be detected in the parts per billion and/or parts per trillion range. Extreme care will be taken to prevent cross-contamination of these samples. A clean pair of new, disposable gloves will be worn for each sample location. Sample containers for source samples or samples suspected of containing high concentrations of contaminants are placed in separate plastic bags and coolers immediately after collecting, preserving and tagging.

Sample collection activities will proceed progressively from the least contaminated area to the most contaminated area (when known).

Disposal

All waste generated by this process 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 environmental pollution.

Documentation

The technician(s) will document the type and number of samples collected during each field event. All sample information will be documented in the field notebook, field log data sheet and chain-of-custody record.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Sampling Report
Attachment 5: Field Log Data Sheet

Attachment 1
Chain of Custody Form

Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Number of Containers/Preservative

COC _____ of _____

Project Manager:

Project QC Contact:

Sampled by:

Laboratory:

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil	Matrix:	Type: Grab	Type: Comp.	Type: QC	Water: VOCs (HCl) #1	Water: SVOCs (unpreserved) #2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (unpreserved) #3	Water: Diesel Range Organics (HCl)	Water: Nutrients (H_2SO_4) #4	Water:	Water:	Water:	Soil: VOCs (tared MeOH) #1	Soil: GRO, BTEX (tared MeOH) #1	Soil: DRO (tared unpreserved)	Soil: Metals (unpreserved)	Soil: SVOCs (unpreserved) #2	Soil: % Solids (plastic vial, unpres.)	Soil:	Soil:	Soil:	Total Number Of Containers
1.																														
2.																														
3.																														
4.																														
5.																														
6.																														
7.																														
8.																														
9.																														
10.																														

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐Air Freight ☐Federal Express ☐Sampler ☐Other: _____ Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

HRL GLSTOF CHMS/Chain Of Custody Form 2009 RLG Rev. 09/01/09

UMP013103

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

Preservative: _____

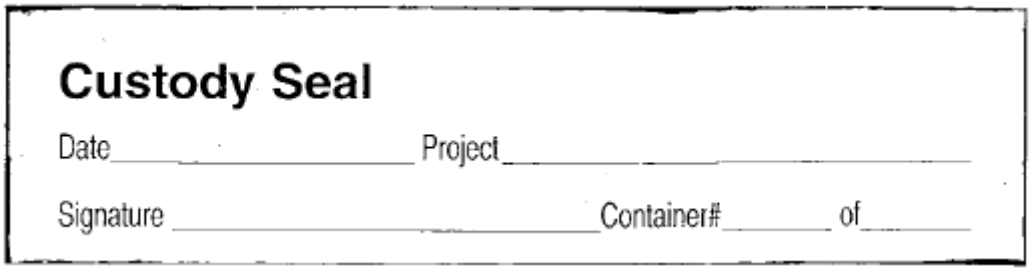
Sampled By: _____

Sample Location: _____

_____

UMP013104

Attachment 3
Custody Seal – if applicable



Attachment 4
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

<Name inserts here>
Environmental Technician

Document1

UMP013106

Attachment 5
Field Log Data Sheet

Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:
Location:
Project #:
Project Name:

Sample Identification	Collection		Matrix			Type			Number of Containers/ Analysis																
	Date	Time	Soil	Sludge		Grab	Comp.	QC	2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:
1.																									
2.																									
3.																									
4.																									
5.																									
6.																									
7.																									
8.																									
9.																									
10.																									
11.																									
12.																									
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14.																									
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18																									
19.																									
20.																									

UMP013107

STANDARD OPERATING PROCEDURE

Documentation on a Chain-of-Custody Form

Revision 3

March 23, 2010

Approved By: Andrea Nord _____ 03/23/10

Print QA Manager(s) Signature Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	AAN	Date:	04/08/11
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for Documentation on a Chain-of-Custody Form

Purpose

The purpose of this procedure is to describe how to properly document information on a Chain-of-Custody (COC) form.

Applicability

These procedures apply to anyone, any time a COC is required.

Definitions

Chain-of-Custody A legally binding document that identifies sample identification, analyses required, and shows traceable possession of samples from the time they are obtained until they are introduced as evidence in legal proceedings.

Equipment

Chain of Custody form
Indelible ink pen

References

Groundwater sampling guidelines and groundwater and surface water sampling procedures by Barr Engineering Company.

Responsibilities

The environmental technician(s)/field technician(s) are responsible for accurate and complete documentation on the COC.

Procedure

The COC is the most important sampling document, it must be filled out accurately and completely every time.

Completing a Chain-of-Custody

1. The COC should be completed prior to leaving the sampling location.

2. Complete one COC or more as needed for each cooler of samples.

3. The COC must contain the following information:

    a. Project number
    b. Project name
    c. Two digit state identification for the state the samples originated from/sampled in
    d. Unique Chain-of-Custody number
    e. Sample location
    f. Sample start depth (if applicable)
    g. Sample stop depth (if applicable)
    h. Depth unit of measurement (meter, feet, inches, etc)
    i. Date and time of sample collection
    j. Sample matrix
    k. Container type and number
    l. Whether the sample is a grab, composite, or blank sample
    m. Project manager
    n. Project Quality Control (QC) contact
    o. Initals of sample technician(s)
    p. Laboratory name
    q. Analyses required
    r. Signature of sampler(s)
    s. Signature of transferee
    t. Date and time of transfer
    u. Method of transport and any shipping numbers
    v. Presence or absence of ice
    w. Method of transport (UPS, FedEx, local courier, Sampler, etc.)
    x. Air Bill number (if applicable)
    y. If sample preservation check conducted in the field indicates:
    1) additional preservation is required for inorganic samples. Note this on the COC or perform a pH adjustment and note the volume, concentration and preservative type on the COC. Or,
    2) that a VOC sample is not properly preserved, note this on the COC, request a 7 day TAT due to the analytical method holding time is 7 days from collection.

4. The COC should always accompany the cooler of samples associated with the COC.

    a. Distribution of the COC pages:

    Pages one and two go to the laboratory, page three goes to the lab coordinator, and the fourth page is the field copy.

Documentation

The Chain-of-Custody form is the documented proof of possession of samples collected. This is documented by field personnel collecting the samples and the laboratory receiving the samples.

Attachments

Attachment 1: Chain of Custody Form

Attachment 1
Chain of Custody Form

Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Number of Containers/Preservative

Water: VOCs (HCl) #1 | SVOCs (unpreserved) #2 | Dissolved Metals (HNO_3) | Total Metals (HNO_3) | General (unpreserved) #3 | Diesel Range Organics (HCl) | Nutrients (H_2SO_4) #4 | | | |

Soil: VOCs (tared MeOH) #1 | GRO, BTEX (tared MeOH) #1 | DRO (tared unpreserved) | Metals (unpreserved) | SVOCs (unpreserved) #2 | % Solids (plastic vial, unpres.) | | | | Total Number Of Containers

COC _____ of _____

Project Manager: _____

Project QC Contact: _____

Sampled by: _____

Laboratory: _____

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil		Type: Grab	Type: Comp.	Type: QC
1.											
2.											
3.											
4.											
5.											
6.											
7.											
8.											
9.											
10.											

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐Air Freight ☐Federal Express ☐Sampler ☐Other: _____

Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:\SL\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09/01/09

UMP013111

STANDARD OPERATING PROCEDURE

Field Screening Soil Samples

Revision 4

March 1, 2011

Approved By:	Andrea Nord	Andrea Nord		4/8/11
	Print	QA Manager(s)	Signature	Date
	John W. Juntilla	John W. Juntilla		3/24/11
	Print	Field Technician(s)	Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedure for Field Screening Soil Samples

Purpose

To describe the procedure for properly screening soil or sediment samples in the field.

Applicability

This procedure applies to all field technicians responsible for field screening soil or sediment samples.

Definitions

PPE Personal protective equipment
PID Photoionization Detector
FID Flame Ionization Detector

Equipment

PPE (gloves, safety glasses)
Project Health and Safety Plan
Quart-sized-self-sealing Polyethylene bag
Photoionization detector (PID)
Flame ionization detector (FID)
Thermometer
Indelible ink pen or pencil
Stainless-steel spoon
Squirt bottle with tap water
Logbook
Alconox®
Brush

Responsibilities

The environmental technician(s) is responsible for the proper sample identification; field screening procedures; field equipment and calibration; quality control procedures and documentation.

Procedure

The field screening techniques for soils are as follows: (1) visual examination; (2) odor; (3) headspace organic vapor screening; and (4) oil sheen. The results of these four screening procedures may be used to screen soil samples for possible contamination.

- **Visual Examination.** A visual examination of the soil sample will include noting any discoloration of the soil or visible oiliness or tar.

- **Odor.** The sampler will note odor only if noticed incidentally while handling the soil sample. Samplers will not unduly expose themselves to sample odors. Odor will be described as light, moderate, or strong, and appropriate description of the type and odor, if evident.

- **Headspace Organic Vapor Screening.** The polyethylene bag headspace method recommended by the Minnesota Pollution Control Agency will be used in the field to screen soils suspected to contain volatile organic compounds. The screening method is intended to be used in conjunction with other "real time" observations.

  The following equipment is required to conduct headspace organic vapor screening: photoionization or flame ionization detector (PID or FID), self-sealing quart-sized polyethylene bag, a log book or record sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan (PHASP). The meter shall be calibrated daily or more frequently if suspect data is obtained.

  The following procedure will be used for checking the calibration of the flame ionization detector:

  FID calibration check is conducted using a two point calibration process with methane gas. Calibrate the instrument by analyzing the calibration gas at 100 ppm and 1,000 ppm. If instrument values exceed ± 5% from true value, then the FID needs to be recalibrated.

  Reference the Standard Operating procedure for the TVA1000B (FID) for further information.

  The following procedure will be used for checking the calibration of the PID:

  PID calibration check is conducted using isobutylene calibration gas at a concentration of 100 ppm. Analyze a sample of the calibration gas, evaluate result, if result exceeds ± 5% from true value, then the PID needs to be recalibrated.

  Reference the Standard Operating procedure for the specific PID model for further information.

The following procedure will be used for conducting headspace organic vapor screening:

1. Soil samples collected from a split-barrel sampler or a direct-push (i.e., Geoprobe®) sample liner will be collected immediately after opening the barrel or liner. If the sample is collected from an excavation wall, soil pile, or backhoe bucket, it will be collected from a freshly exposed surface.

2. Half-fill the bag with the sample to be analyzed using a stainless-steel spoon or a gloved hand and immediately seal it.

3. Agitate the bag for 15 seconds. Manually break up any soil clumps within the bag.

4. Allow headspace development for approximately 10 minutes. The sample should be kept in a shaded area out of direct sunlight. Ambient temperatures during headspace development should be recorded. When ambient temperatures are below 50°F, headspace development should be conducted inside a heated vehicle or building.

5. Agitate the bag for an additional 15 seconds.

6. Quickly puncture the bag with the sampling probe to a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particles.

7. Record the highest meter response as the headspace concentration. The maximum response will likely occur between 0 to 5 seconds.

8. When using a FID, it may be necessary to correct for methane. In this case, take a reading first with carbon filter, then without. This will require two duplicate bag samples. The second reading less the first is the headspace adjusted for methane. Adjusted readings less than zero are considered zero. Methane correction is not necessary if a PID is used.

- **Oil Sheen Test.** The oil sheen or hydrocarbon is a method used to immediately determine the approximate magnitude of coal tar contamination in soil by observation of the sample in the field. The test is useful in soils which do not have a high binding capacity with polyaromatic hydrocarbons (PAHs) (i.e., the PAHs are free on the surface of the soil particles and can be released by a stream of water).

  The equipment required to conduct the oil sheen test includes: a stainless-steel spoon, a squirt bottle filled with tap water, a log book or recording sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan. Decontamination of the spoon between test events will consist of scrubbing the surface of the spoon with a solution of Alconox® in water using a brush and then rinsing the spoon with water.

  The procedure for conducting the oil sheen test consists of obtaining approximately 50 grams (about 30 cc) of representative soil with the spoon and then directing a stream of water onto the soil in the spoon with the squirt bottle until the soil is saturated and water begins to collect around the soil. The amount of oil sheen present on the water is determined by observation and the results of the test are reported as a magnitude of oil sheen observed: none, trace, light, moderate, heavy or rainbow. The test results, sample location, and observations of the sample's appearance and odor are recorded in the log book.

  The specific soil types at the area of investigation should be accounted for when performing the oil sheen test. The best results are obtained in silts, sands, and/or gravels with low organic content. The results obtained from clayey soils may appear deceptively low. Typical descriptions of each test result are given below.

Oil Sheen Test Result	Description
None	No sheen detected.
Trace	Possible or faint oil sheen observed (may not continue to generate sheen as additional water is added).
Light	Obvious sheen that may not cover entire water surface
Moderate	Definite oil sheen that covers entire surface, but "rainbow colors" not distinguishable.
Heavy	Definite oil film or product that does not display rainbow colors.
Rainbow	Definite oil sheen, film or product that displays rainbow colors.

Interferences

Interferences on the test can be caused by any contaminant which will cause an oil sheen on water. The samples will be carefully observed for characteristic appearance or odors which may indicate a possible contaminant other than coal tar or petroleum substances. Sunlight and low temperatures may interfere with headspace development. Water and soil particles may interfere with PID and FID readings.

Documentation

The technician(s) will document the soil sampling events in a project dedicated field logbook or on field log data sheets.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Data Sheet

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

Attachment 2
Field Log Data Sheet

Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:									Number of Containers/ Analysis																	
Location:																										
Project #:																										
Project Name:																										
Sample Identification	Collection		Matrix			Type			2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:		
	Date	Time	Soil	Sludge		Grab	Comp.	QC																		
1.																										
2.																										
3.																										
4.																										
5.																										
6.																										
7.																										
8.																										
9.																										
10.																										
11.																										
12.																										
13.																										
14.																										
15.																										
16.																										
17.																										
18																										
19.																										
20.																										

STANDARD OPERATING PROCEDURE

Filtering of Groundwater and Surface Water Samples

Revision 2

March 23, 2010

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord	Andrea Nord		3-23-10

Print	Field Technician(s)	Signature	Date
Kim Johannessen	Kim Johannessen		3-23-10

Barr Engineering Company
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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	KSJ	Date:	4/4/2011
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Filtering of Groundwater and Surface Water Samples

Purpose

To describe the filtering process for groundwater and surface water samples to remove silt, clay, and particles.

Applicability

These procedures apply to the filtering of groundwater and surface water for laboratory analysis.

Equipment

Ziploc Baggies
Cooler
Bagged Ice
Chain of Custody Form
Sample Label
Talc-free latex or vinyl gloves
0.45 micron pore size filter
0.60 micron pore size filter – required if prefiltering the sample
Peristaltic or vacuum Pump
Tubing
Bubble Wrap

References

Corning Disposable Sterile Filter Information Booklet.

Responsibilities

The environmental technicians are responsible for the filtering of groundwater and surface water samples. The Field Operations/QA Officer or the environmental technician(s) will order the sample containers prior to the sampling event. The environmental technician(s) is responsible for the proper collection of groundwater and surface water samples, sample identification, quality control procedures, and documentation.

Procedure

Vacuum Pump - Filtering Process

1. Collect groundwater or surface water sample in an unpreserved sample container (filtering must be done within 15 minutes of collection).

2. Pour groundwater or surface water sample into 200-ml or 500-ml Corning Disposable Sterile Filter, depending on volume needed.

3. The filters must be 0.45 micron pore size.

UMP013120

Note: Prefiltering may be needed if sample is too turbid. The prefilter will filter particles up to 0.60 micron pore size.

a. Add prefilter to filter by placing it over the filter membrane (extends the life of the filter).
b. Filter membrane must be covered completely by prefilter to work properly.
c. Prefilter must be placed rough side up to be effective.

4. Attach vacuum pump to filter; turn on power.

5. Filter groundwater or surface water sample until amount of sample needed is filtered.

   Note: Additional filters may be needed to get enough sample volume.

6. After filtering is complete, pour contents into the appropriate sample container, dispose of filter. Depending upon groundwater conditions, additional filters may be required.

In-line - Filtering Process

1. Attach 0.45 micron pore size filter to the end of purge tubing, ensuring direction of flow is correct. (filtering must be done within 15 minutes of collection).

2. Place appropriate sample container at the filter outlet

3. Turn on pump and purge a minimum of one filter volume through filter before filling sample container. A new filter must be used for each sampling location. Depending upon groundwater conditions, additional filters may be required.

4. After filtering is complete, pour contents into the appropriate sample container, dispose of filter. Depending upon groundwater conditions, additional filters may be required.

Quality Control Samples

Equipment Blank An aliquot of reagent water that is subjected in the laboratory to all aspects of sample collection and analysis, including contact with all sampling devices and apparatus. The purpose of the equipment blank is to determine if the sampling devices and apparatus for sample collection have been adequately cleaned before they are shipped to the field site. An acceptable equipment blank must be achieved before the sampling devices and Apparatus are used for sample collection. One equipment blank must be collected per site or every 10 samples, whichever is more frequent.

Field Blank An aliquot of water that is placed in a sample container in the laboratory, shipped to the field, and treated as a sample in all respects, including contact with the sampling devices and exposure to sampling site conditions, filtration, storage and preservation, and all analytical procedures. The purpose of the field blank is to determine whether the field or sample transporting procedures and environments have contaminated the sample. One field blank must be collected per site or every 10 samples, whichever is more frequent.

UMP013121

Field Duplicate Two identical aliquots of a sample collected in separate sample bottles at the same time and placed under identical circumstance using a duel inlet sampler or by splitting a larger aliquot and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicate samples give a measure of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures. One set of field duplicates must be collected per site or every 10 samples, whichever is more frequent.

Sample Storage

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of fiberglass tape. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Documentation

The technician(s) will document the water sampling events on field log data sheets, field log cover sheets, and field log data reports. The technicians will document the number of filters and prefilters used for each sample filtered on the field log data sheet. They will also document the type and number of bottles on both the field log data sheet and chain-of-custody record. The analysis for each bottle and the laboratory used will be documented on the chain-of-custody record. The sampling request form will document which sampling containers are used for which water samples.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Sampling Report
Attachment 5: Field Log Cover Sheet
Attachment 6: Field Log Data Sheet

Attachment 1
Chain of Custody Form

Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Number of Containers/Preservative

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Water: VOCs (HCl) #1	Water: SVOCs (unpreserved) #2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (unpreserved) #3	Water: Diesel Range Organics (HCl)	Water: Nutrients (H_2SO_4) #4	Soil: VOCs (tared MeOH) #1	Soil: GRO, BTEX (tared MeOH) #1	Soil: DRO (tared unpreserved)	Soil: Metals (unpreserved)	Soil: SVOCs (unpreserved) #2	Soil: % Solids (plastic vial, unpres.)	Total Number Of Containers
1.																								
2.																								
3.																								
4.																								
5.																								
6.																								
7.																								
8.																								
9.																								
10.																								

COC _____ of _____

Project Manager: _____

Project QC Contact: _____

Sampled by: _____

Laboratory: _____

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other: _____ Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:RLGISTDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

UMP013123

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

Preservative: _____

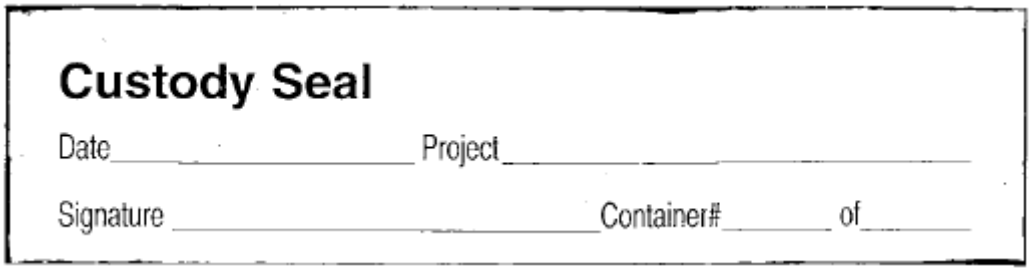
Sampled By: _____

Sample Location: _____

_____

UMP013124

Attachment 3
Custody Seal – if applicable



Attachment 4
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ` 4700 W. 77th Street ` Minneapolis, MN 55435-4803 ` 952/832-2600

Attachment 5
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

Attachment 6
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample Time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)								
CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

STANDARD OPERATING PROCEDURE

Collection of Each Type of Groundwater Sample from Monitoring Wells, Residential Wells and Residential Systems

Revision 3

March 23, 2010

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord			3-23-10

Print	Field Technician(s)	Signature	Date
Kim Johannessen			3-23-10

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	KSJ	Date:	4/4/2011
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Collection of Each Type of Groundwater Sample from Monitoring Wells, Residential Wells and Residential Systems

Purpose

The purpose of this procedure is to describe the collection of water samples for volatiles, semivolatiles, metals, inorganics, bacteria, and dioxin from monitoring wells, residential wells and residential systems.

Applicability

This procedure applies to the stabilization of monitoring wells and subsequent collection of groundwater samples by the sampling technician(s). It identifies each container type (volume, construction, preservative) required for each category of analyses, their corresponding holding times and collection procedures from monitoring wells, residential wells and residential systems.

Definitions

Headspace. The air space between the container top and the water sample level.

Holding Time. Period of time between sample collection and when the sample is analyzed.

Sample Preservation. The stability of analytes depends upon the proper preservation technique and preservation acceptance criteria as defined by EPA Title 40 of the Code of Federal Regulations and corresponding method criteria.

Equipment

Sampler media
Pre-cleaned-certified Sampling Containers
Coolers
Ziploc® Baggy
Ice
Water-proof ink pen or pencil
Bailer (Stainless Steel or Polyethylene)
Nitrile Gloves
Water Quality Meter
Sample label
Chain of Custody Form
Alconox

References

Quality Assurance Manual: Groundwater and Surface Water Sampling Procedures, Barr Engineering Co.; American Water Works Association: Pocket Guide to Water Sampling; Environmental Sampling, A Summary, the Radian Corporation.
Ground Water Sampling Guidelines by MPCA
EPA: Title 40 of the Code of Federal Regulations

Responsibilities

The Field Operations/QA Officer or the environmental technician(s) will order the sample containers prior to the sampling event. The environmental technician(s) is responsible for the proper collection of monitoring wells, residential wells, and residential system groundwater samples; sample identification; quality control procedures; sample filtering and documentation.

Procedure

I. Obtain Sampling Media

Approximately one week before the sampling event, the sample containers should be ordered from the laboratory.

Note: Container volume, type, and preservative are important considerations in sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat examinations. The container type or construction varies with the analysis required. The analytical laboratory will preserve the container before shipment. Preservation and shelf life vary; contact the laboratory to determine if an on-hand container is still useful.

II. Measure Water Level, Well Depth and Purge

Once the water level and well depth measurements have been taken and the well has been purged in accordance to Barr's Calculation of Purge Volumes for Groundwater Sampling Wells SOP and allowed to stabilize, the technician can begin groundwater sampling.

Stabilization Test Measurements

Collection of stabilization test measurements shall begin at the same time as groundwater purging prior to sample collection is initiated. Well stabilization measurements will be collected and recorded at the start of the purging process and once every ten minutes during the purging process, with a minimum of one measurement collected per well volume removed. A well volume will be measured as the volume of water that occurs in each well from the base of the well to the water level measurement collected prior to initiation of purging. Once three well volumes have been removed, the well may be sampled after three consecutive measurements, collected at the intervals described above, are within the ranges presented below:

Specific Conductance:	$\pm$5% of the most recent reading (temperature corrected)
pH	$\pm$5% of the most recent reading (in pH units)
Temperature	$\pm$5% of the most recent reading (in degrees Celsius)
Oxidation Reduction Potential (Eh)	$\pm$20 mV of the most recent reading

Collect samples only after a minimum of three water-column volumes have been purged and stabilization of field water-quality parameters has been demonstrated by meeting the target criteria defined in the preceding paragraph. Field technician will check operator procedures, equipment functioning, and well construction information for potential problems. In particular, field staff will re-evaluate whether or not water is being withdrawn from the appropriate depth to effectively evacuate the well.

If all the checks produce no new insight, a decision might be made to collect samples after five or more water-column volumes have been purged even if field measurements have not stabilized. If the well was purged dry, it shall be allowed to recharge and the samples will be collected.

However, if either circumstance applies, the following procedure is required: Before authorizing the laboratory to analyze the samples, the meaningfulness and value of completing laboratory analysis of the sampling suite will be evaluated by reviewing the results of field measurements, well construction data, site hydrology, etc. Where such data is presented, it will be clearly documented that stabilization was not achieved; at a minimum, this fact will be reported on the field data sheets and in the Field Sampling Report.

III. Groundwater Sampling

1. Monitoring Wells (Permanent or Temporary)

1.a Monitoring wells may either be installed permanently or temporarily. They are constructed for the collection of groundwater samples. These monitoring wells have a wide variety of diameters. Groundwater samples might also be collected out of a pit or a drilled hole.

1. Put on sampling gloves to protect the sample and skin.

   Note: New sampling gloves are needed for each well. Never reuse old gloves.

2. Prepare sampling containers by filling out the label with the following information:

   – Project number
   – Location sampled
   – Individual collecting the samples
   – Date and time of sample collection
   – Sample analysis (if required by the lab)

   Note: Use an inedible permanent pen to avoid ink bleeding.

3. Sampling

   a. Sampling Technique Using a Polyethylene Bailer (1) or Stainless Steel Bailer (2):

      1. Polyethylene bailer and Cord reel and rope— Tie the rope to the bailer and lower the bailer into the well with the cord reel.

      2. Remove foil from the bailer top (stainless steel).

3. Connect the rope to the bailer top.

4. Remove foil from the bailer body (stainless steel) and the check valve (Teflon).

5. Connect these two parts together; screw these pieces into the bailer top.

6. Slowly rotate the cord reel to lower the bailer into the top of the water column.

   Note: Make sure not to stir up the water with the bailer, thus volatizing the samples.

7. Keep the bailer in the top portion of the water column when collecting the sample.

8. When the bailer is filled, slowly rotate the cord reel to retrieve the bailer out of the well.

9. Collect samples by utilizing steps outlined in this SOP.

10. When all samples are collected, place the samples in a sampling cooler with ice.

11. Disassemble the sampling apparatus.

    Step 1: Cut rope several feet above bailer
    Step 2: Dismantle bailer assembly
    Step 3: Place bailer parts into a dirty bailer cooler (cooler is then sent to lab for decontamination of bailers)

12. After sampling is completed, clean sampling apparatus with alconox or equivalent and distilled water.

b. Sampling technique utilizing a peristaltic pump:

   The maximum depth for a peristaltic pump is typically 25 feet, but may be less at higher altitudes.

   This pump is used when the water level is within suction lift, i.e., within about 25 feet of the ground surface. It usually is a low-volume suction pump with low pumping rates suitable for sampling shallow, small-diameter wells.

   1. Cut tubing to desired length.

   2. Connect tubing to pump head, leaving 1 to 2 feet for discharge line.

   3. Lower tubing into the well water (1 to 2 feet below surface).

4. Turn on pump and set speed at the desired rate of flow.

2. Residential Sampling—potable water supply

2.a Residential sampling is sampling conducted on a potable water supply. It is very important that these samples are representative of that water supply. The sampling point must be located ahead of any filtering devices or water conditioners. The highest standard of sampling technique is required for residential well sampling.

1. Put on sampling gloves to prevent contamination of the samples..

2. Purge private wells before sampling (including taking pH, conductivity, and temperature).

Note: Rule of thumb—at least one well and storage tank volume should be removed. A 15-minute purging period is usually sufficient for residential wells.

3. Prepare sampling containers by filling out the label with the following information:
   – Project number
   – Location sampled
   – Individual collecting the samples
   – Date and time of sample collection
   – Sample analysis (if required by the lab)

4. Unscrew sampling container top (do not let the container or container top touch anything).

Note: If applicable, collect the volatile samples first, proceeding to the least volatile.

5. Collect sample from the purge tap.

6. After completing the collection of the samples, place samples in a cooler with ice.

7. Turn off the tap; clean up any mess made by sampling.

3. Residential Systems (water supply system)

3.a Residential systems is sampling done on a water supply system. It must be representative of the water quality of that system. Preferably, a sampling tap will be ahead of the storage tank and close to the well head. Sample collection from this tap in the system must be from a steady stream of water.

1. Select a tap that is free from exterior contamination (remove anything attached to the faucet).

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- If bacterial samples are to be collected, flame the end of the tap with a lighter or match to sterilize the tap.

2. Put on sampling gloves to prevent contamination of the samples

3. Turn on water tap; make sure the water is a steady stream out of the tap.

   Note: If water is not a steady stream, find a new tap. Also, make sure the tap is not leaking by the valve handle.

4. The water tap should be run steadily for two to three minutes or a sufficient time to permit clearing of the service line. Take pH, conductivity, and temperature.

5. Prepare sampling containers by filling out the label with the following information:

   - Project number
   - Location sampled
   - Individual collecting the samples
   - Date and time of sample collection
   - Sample analysis (if required by the lab)

6. Without changing water flow, the sample(s) can be collected.

   Note: Make sure there is no water splash up into the sampling container or cap. If applicable, collect the volatile samples first, proceeding to the least volatile.

7. Place sampling containers in the appropriate cooler with bagged ice.

8. Clean up any mess made by the sampling event.

3.b Collecting Field Samples

To ensure sample integrity, collect volatile samples first, then proceed to the least volatile method required for the site.

1. Volatiles and WI Gasoline range organics (WIGRO)– Samples to be analyzed for volatile organics will be collected in two or three 40-ml vials with Teflon®-lined septum caps. Use caution because concentrated acid may be present. Do not rinse glass vials. Hold bottle in one hand, the cap right side up in the other. Allow a slow stream of water to run into the 40-ml vial. The vial should be held at an angle while filling to prevent water from falling directly to the bottom of the container and becoming overly disturbed. While holding the vial vertically, add the water sample until a small meniscus forms on the top of the sample container. Avoid air bubbles and overfilling the vial. Cap tightly, invert the bottle, and tap gently. If any air bubbles appear in the vial, discard and collect sample in a new vial. These samples will be cooled to approximately 4°C. After collecting the required number of vials, insert them in a zip-lock plastic bag and place in a cooler with ice.

If prescribed by site-specific situations a duplicate volatile sample may be collected and field checked with a pH indicator strip to assess the pH of the sample. If the pH is greater than 2, the laboratory will be instructed to reduce the holding time of that day's samples to the 7-day holding period used for unpreserved samples.

2. Semivolatiles (includes: Pesticides, PCB, Herbicides, BNAs, Dioxin and Furans)– Samples to be analyzed for semivolatile organics will be collected in a 1-liter amber glass jar with a Teflon-lined septum cap for each fraction. Fill container slowly with a minimum headspace and cap tightly. Do not rinse glass containers. Place container directly in a cooler with ice. These samples will be cooled to approximately 4°C.

   Note: For Dioxin and furan analysis, the bottles must be preserved with 80 mg. sodium thiosulate if they are being collected from a chlorinated source.

3. WI Diesel Range Organics (WIDRO) – Samples to be analyzed for WIDRO are to be collected in a 1-liter amber glass jar with a Teflon-lined septum cap and preserved with 1:1 HCl to a pH or less than 2. Fill container slowly with a minimum of headspace and cap tightly. Do not rinse glass containers. Place container directly into a cooler with ice. These samples will be cooled to approximately 4°C.

4. Other Organics – Containers may contain acid, use caution when handling. Fill containers completely minimizing headspace and avoiding spillage. Place container directly in a cooler with ice.

5. Metals

   - Total Metals – Samples to be analyzed for metals will be collected in a 500-mL or 1-liter polyethylene jar with a polyethylene-lined closure. These samples will be preserved in by the lab with a 1:1 (50%) solution of Nitric Acid to reduce the pH of the sample to less than 2.

   - Filtered Metals – Select the appropriate Corning filter size, either 250-ml or 500-ml volume (see Standard Operating Procedures for filtering groundwater samples). Pour filtered sample into metals sample container, minimizing headspace and avoiding spillage. Use caution handling metals containers because of nitric acid. Place directly in a cooler with ice.

6. Phenolics – Samples to be analyzed for phenol will be collected in a 1-liter glass jar. These samples will be preserved in the field with sulfuric acid to reduce the pH of the sample to less than 2 and cooled to approximately 4°C.

7. Oil and Grease by hexane extraction – Samples to be analyzed for Oil and Grease will be collected in a 1-liter glass jar with a Teflon-lined septum cap preserved to a pH or less than 2 with either 1:1 hydrochloric acid or 1:1 sulfuric acid. These samples will be cooled to approximately 4°C.

8. Cyanide – Groundwater samples to be analyzed for cyanide will be collected in a 1-liter polyethylene container with a polyethylene cap and preserved with sodium hydroxide to pH greater than 12 and cooled to approximately 4°C.

9. Collecting General Chemistry Samples – Samples to be analyzed for sulfate, chloride, carbonate, and bicarbonate will be collected in 1-liter plastic jars. These samples will be cooled to approximately 4°C.

10. Bacteria – Plastic bottles or glass containers preserved with 10 mg of sodium thiosulfate are used for bacterial sample collection. Care should be taken not to contaminate the container before collecting the sample. Fill the container within 1 inch of the top. This allows the laboratory to shake and mix the contents before analysis. Close and seal the Whirl Pak; grasp the wire ends and flip the pack in a circular motion several times and twist the wires together. Pack the containers carefully in a cooler with ice.

IV. Collecting Quality Control Samples

The effectiveness of the sample handling techniques is monitored by collecting both preserved and unpreserved field blank samples.

Field (or Masked) duplicate samples will be collected to measure relative sampling precision. Five percent of all samples collected are collected in duplicate. These samples are collected at the same time using the same procedures, equipment, and types of containers as the required samples. They are also preserved in the same manner and are either co-located or split and submitted for the same analyses as the required samples.

Trip blanks are only used when sampling for volatile organics. Their purpose is to determine if contamination has occurred as a result of improper sample container cleaning, contaminated blank source water, sample contamination during storage and transport due to exposure to volatile organics, or other environmental conditions during sampling and analysis. Trip blanks are prepared prior to the sampling events by the laboratory providing the sample containers. The water will be free of contaminants. The trip blanks are prepared by the lab, sealed and labeled appropriately at the lab, and transported to the field in the same containers as the sample vials. These blanks are not opened in the field. They are transferred to the coolers designated for volatile sample storage and transport and accompany the samples to the analytical laboratory.

Field blanks (or Rinsate Blanks) are used to evaluate the effects of onsite equipment contaminants. Their purpose is to determine if contamination has occurred as a result of improper equipment cleaning. Field blanks are prepared onsite by pouring analyte-free water through decontaminated sample collection equipment (bailer or pump) and collecting the rinsate in a sample container. The field blanks will be handled in the same manner as the sample group for which they are intended (i.e., blanks will be stored and transported with the sample group).

Some general considerations will be taken into account when planning and conducting sampling operations. The sampler will take into consideration the required sample volumes; sample holding times, sample handling, and special precautions for trace contaminant sampling.

The volume of the sample obtained should be sufficient to perform all required analyses with an additional amount collected to satisfy the needs for quality control, split samples, or repeat examinations. The Laboratory Coordinator should be consulted for any specific volume requirements. Multiple sample containers are always required for volatile organic compound (VOC) analyses.

The elapsed time between sample collection and initiation of each laboratory analysis will fall within a prescribed time frame. Holding times for samples required by this project are prescribed by EPA: Title 40 of the Code of Federal Regulations.

After collection, all samples should be handled as few times as possible. Technicians should use extreme care to ensure that samples are not contaminated. If samples are placed in a cooler, technicians should ensure that melted ice cannot cause sample containers to become submerged, as this may result in cross-contamination. Plastic bags, such as Ziplock® bags, should be used when small sample containers (e.g., VOC vials) are placed in coolers to prevent cross-contamination.

Some compounds can be detected in the parts per billion and/or parts per trillion range. Extreme care will be taken to prevent cross-contamination of these samples. A clean pair of new, disposable gloves will be worn for each sample location. Sample containers for source samples or samples suspected of containing high concentrations of contaminants are placed in separate plastic bags and coolers immediately after collecting, preserving and tagging. Sample collection activities will proceed progressively from the least contaminated area to the most contaminated area (when known).

Sample Storage

Place samples as soon as possible in a cooler containing bagged ice. Samples must be kept cold (4 ±2°C) at all times until delivery to the laboratory. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of fiberglass tape. Samples must be secure to prevent tampering with or loss of samples. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

Disposal

All waste generated by this process 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 environmental pollution.

Documentation

The technician(s) will document the groundwater sampling events on field log data sheets, field log cover sheets, and field log data reports. They will also document the type and number of bottles on both the field log data sheet and chain-of-custody record. The analysis for each container and the laboratory used will be documented on the chain-of-custody record.

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Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Sampling Report
Attachment 5: Field Log Cover Sheet
Attachment 6: Field Log Data Sheet

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Attachment 1
Chain of Custody Form

Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Number of Containers/Preservative

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Water: VOCs (HCl) #1	Water: SVOCs (unpreserved) #2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (unpreserved) #3	Water: Diesel Range Organics (HCl)	Water: Nutrients (H_2SO_4) #4	Soil: VOCs (tared MeOH) #1	Soil: GRO, BTEX (tared MeOH) #1	Soil: DRO (tared unpreserved)	Soil: Metals (unpreserved)	Soil: SVOCs (unpreserved) #2	Soil: % Solids (plastic vial, unpres.)	Total Number Of Containers
1.																								
2.																								
3.																								
4.																								
5.																								
6.																								
7.																								
8.																								
9.																								
10.																								

COC _____ of _____

Project Manager: _____

Project QC Contact: _____

Sampled by: _____

Laboratory: _____

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other: _____ Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:RLG\STDFORM\S\Chain Of Custody Form 2009 RLG Rev. 09\01\09

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Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

Preservative: _____

Sampled By: _____

Sample Location: _____

_____

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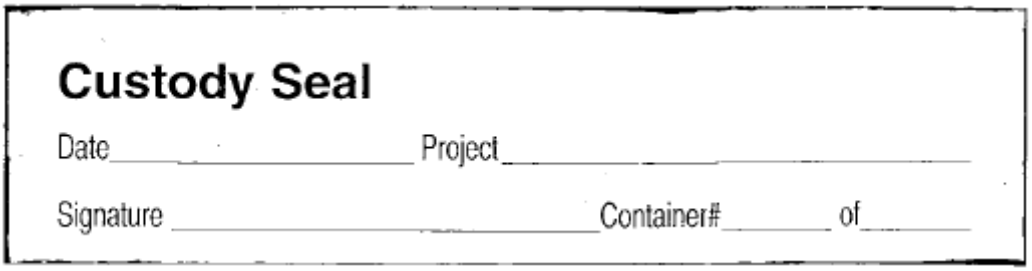
Attachment 3
Custody Seal – if applicable



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Attachment 4
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

UMP013143

Attachment 5
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

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Attachment 6
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample Time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)								
CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DMI\Templates\FieldLogDataSheet.doc

STANDARD OPERATING PROCEDURE

Maintenance and Operation of the YSI Model 556 MPS Water Quality Monitoring System

Revision 3

April 8, 2011

Approved By:

Andrea Nord	Andrea Nord	4-8-11
Print	QA Manager(s) Signature	Date
Kim Johannessen	Kim Johannessen	4-8-11
Print	Field Technician(s) Signature	Date

Barr Engineering Company
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Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for the Maintenance and Operation of the YSI Model 556 MPS Water Quality Monitoring System

Purpose

The purpose of this SOP is to clearly define the procedures required to accurately measure dissolved oxygen, conductivity, temperature, pH and oxidation reduction potential (ORP) in the field using the YSI Model 556 MPS water quality system.

Applicability

This procedure is applicable to field personnel who will be using the YSI Model 556 MPS to measure dissolved oxygen, conductivity, temperature, pH and ORP in the field.

Definitions

ORP Oxidation Reduction Potential
MPS Multi-Probe System

Equipment

YSI Model 556 MPS
O-ring lubricant
Four alkaline "C" batteries
Mild soap
Water
ChemWipes
Screwdrivers
Conductivity standard
pH buffer solution (pH 7.00 and 10.00)
ORP solution (Zobel)
Zobel solution value chart
Moist sponge
Calibration cup
Field Log Data Sheet

References

YSI Model 556 MPS water quality system Operations Manual

Responsibilities

The environmental technician(s) is responsible for the proper sample identification; field screening procedures; field equipment and calibration; quality control procedures and documentation.

Instrument

The rugged and reliable YSI 556 MPS (Multi-Probe System) combines the versatility of an easy-to-use, easy-to-read hand-held unit. It features a waterproof, impact-resistant case and it simultaneously measures dissolved oxygen, conductivity, temperature, pH and ORP.

Analysis	Conductivity	Salinity	Oxidation Reduction Potential	pH	Dissolved Oxygen
Analytical Method	Standard Method 2510	Standard Method 2520A	Standard Method 2580A	Standard Method 4500-H B	Standard Method 4500-O G

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Maintenance/Installation

1. Instrument

The 556 requires occasional battery replacement and cleaning. Four alkaline "C" cells in the 556 provide 180 hours of operation. Battery life is displayed on the keypad. When the fuel gauge is low, it is time to change batteries.

a. Loosen the four screws in the battery lid on the back of the instrument.
b. Insert four "C" batteries in the clips following the polarity labels on the bottom of the battery compartment.
c. Check the gasket for proper placement and place the lid.
d. Do not over tighten the screws.
e. Clean the display pad with a mild soap and water solution.
f. Wipe the solution on and off.
g. Follow with a clean water wipe.

2. The Probe Module

To prepare the probe module for calibration and operation, the sensors need to be installed into the connectors on the probe module bulkhead. Whenever you install, remove or replace a sensor, it is important that the probe module and all the sensors be dry. This will prevent water from entering the port.

a. Unscrew and remove the probe sensor guard.
b. Using the sensor installation tool, unscrew and remove the sensor port plugs.
c. Locate the port with the connector that corresponds to the sensor that is to be installed.
d. Apply a thin coat of o-ring lubricant to the o-rings on the connector-side of the sensor.
e. Be sure that the probe module sensor port is free of moisture and insert the sensor into the correct port.
f. Gently rotate the sensor until the two connectors align.
g. With connectors aligned, screw down the sensor nut using the installation tool.
h. Repeat these steps for all sensors.

3. Instrument/Cable Connection

a. Line up the pins and guides on the cable with the holes and indentations on the cable connector at the bottom of the 556 instrument.
b. Holding the cable firmly against the cable connector, turn the locking mechanism clockwise until it snaps into place.

Calibration

All of the sensors, except temperature, require daily calibration to assure high performance. This will show specific calibration procedures for all sensors that require calibration. Make sure that the sensors are completely submersed when calibration values are entered. For maximum accuracy, use a small amount of calibration solution to pre-rinse the probe module. Have room temperature water on hand to rinse the probes between calibration solutions.

Make sure to dry the probe module between rinses and calibration solutions. Be sure that port plugs are installed in all ports where sensors are not installed.

To access the calibration screen:

a. Press the on/off key to display the run screen.
b. Press the escape key to display the main menu screen.
c. Use the arrow keys to highlight the calibrate selection.
d. Press the enter key and the calibration screen is displayed.

1. Conductivity Calibration

a. Go to the calibrate screen as described above.
b. Use the arrow key to highlight the conductivity selection.
c. Press enter. The conductivity calibration screen is displayed.
d. Select the specific conductance selection. Press enter.
e. Place the correct volume of conductivity standard into a clean calibration cup.
f. Carefully immerse the sensor end of the probe module into the solution. The sensor must be completely immersed past its vent hole.
g. Gently move the probe up and down to remove any bubbles from the cell.
h. Use the keypad to enter the calibration value of the standard you are using. Be sure to enter the value in ms/cm@25ºC.
i. Press enter; the conductivity calibration screen is displayed. Allow at least one minute for temperature equilibration before proceeding. The current values for all enabled sensors will appear on the screen.
j. Observe the reading under specific conductance. When the reading shows no significant change for 30 seconds, press enter. The screen will indicate that the calibration has been accepted and prompt you to press enter. This returns you to the conductivity calibrate selection screen.
k. Press escape to return to the calibrate menu.
l. Rinse the probe module and dry.

2. Dissolved Oxygen Calibration

[Note: The instrument must be on for at least 20 minutes to polarize the DO sensor before calibrating. Calibrating any one option (% or µg/L) automatically calibrates the other.]

a. Go to the calibrate screen.
b. Use the arrow keys to highlight the dissolved oxygen selection. Press enter. The dissolved oxygen calibration screen is displayed.
c. Use the arrow keys to highlight the DO% selection. Press enter. The DO barometric pressure entry screen is displayed.
d. Place ⅛ inch of water in the bottom of the calibration cup and screw it on the probe module (only engage one or two threads to ensure the DO sensor is vented to the atmosphere).
e. Use the keypad to enter the current local barometric pressure. (If the unit has the optional barometer, no entry is required.)
f. Press enter and the DO% saturation calibrating screen is displayed. Allow 10 minutes for the air in the calibration cup to become water-saturated and for the temperature to equilibrate before proceeding.

g. Observe the reading under DO%. When the reading shows no significant change for 30 seconds, press enter. The screen will indicate that the calibration has been accepted and prompt you to press enter again. This will return you to the DO calibration screen.
h. Press escape to return to the calibrate menu.
i. Rinse the probe and dry.

Note: A moist sponge kept with the probe sensor guard to prevent the dissolved oxygen membrane from drying out.

3. pH Calibration

a. Go to the calibrate screen and select the pH selection.
b. Press enter, and the pH calibration screen is displayed.
c. Select the two-point option. Press enter. The pH entry screen is displayed.
d. Place the correct amount of pH buffer into a clean calibration cup. (Note: for maximum accuracy, the pH buffers you choose should be within the same pH range as the water you are sampling.)
e. Carefully immerse the sensor end of the probe module into the solution.
f. Gently rotate the probe up and down to remove any air bubbles.
g. Use the keypad to enter the calibration value of the buffer you are using. Press enter. The pH calibration screen is displayed.
h. Allow one minute for temperature equilibrium before proceeding. The current values of all enabled sensors will appear on the screen.
i. Observe the reading under pH. When the reading shows no significant change for 30 seconds, press enter. The screen will indicate the calibration has been accepted and prompt you to press enter again to continue.
j. Press enter. This returns you to the specified pH calibration screen.
k. Rinse the probe modules, calibration cup and sensors, and dry.
l. Repeat the above steps using the second pH buffer.
m. Press enter. This returns you to the pH calibration screen.
n. Press escape to return to the calibrate screen.
o. Rinse the probe and dry.

4. ORP Calibration

a. Go to the calibrate screen and use the arrows to highlight the ORP selection.
b. Press enter. The calibration screen is displayed.
c. Place the correct amount of a known ORP solution (Zobel) into a clean calibration cup. (Note: before proceeding, make sure the sensor is dry and, ideally, rinse it with ORP solution.)
d. Carefully immerse the sensor end of the probe up and down to remove any air bubbles.
e. Use the keypad to enter the correct value of the calibration solution you are using at the current temperature. Refer to the Zobel solution value chart.
f. Press enter. The ORP calibration screen is displayed.
g. Allow at least one minute for temperature equilibration before proceeding.
h. Observe the reading under ORP.
i. When the reading shows no significant change for 30 seconds, press enter. The screen will indicate that the calibration has been accepted and prompt you to press enter again to continue.

j. Rinse the probe and dry. The meter is now calibrated and ready for use.

If any calibrations fail, contact the Equipment Technician or manufacturer immediately or obtain a replacement instrument.

Quality Control Samples

Replicate sample measurements should be taken a minimum of one of twenty project samples per type of measurement. Method Blanks must be one for every batch of samples analyzed.

Safety

Please refer to the proper MSDS sheets or the Project Health and Safety Plan to determine the proper PPE required for use with the calibration solutions and reagents listed in this SOP prior to working with these chemicals.

Interferences

Rinse the probe sensor between instrument readings with water and dab dry to ensure accurate results.

Disposal

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. When feasible, implement procedures to minimize the potential for environmental pollution.

Documentation

The field technician will document the YSI Model 556 MPS dissolved oxygen, conductivity, temperature, pH and ORP data on the Field Log Data Sheet.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Cover Sheet
Attachment 3: Field Log Data Sheet
Attachment 4: Meter Calibration Summary Form

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

UMP013152

Attachment 2
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Barr Engineering Company ` 4700 W. 77th Street ` Minneapolis, MN 55435-4803 ` 952/832-2600

UMP013153

Attachment 3
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample Time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)								
CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

Attachment 4
Meter Calibration Summary Form

MCS-1

BARR ENGINEERING COMPANY
METER CALIBRATION SUMMARY

PROJECT _____

TECHNICIAN _____

Meter type and number	Date	Time	Temperature C	Standard Used	Meter Reading	Slope	Conductivity Redline
Conductivity	Date	Solution Used	Cell Result				
Cell Check							
ORP Probe	Date	Temp.	ORP Reading	Calculation Result			
Check							
231+,- 10mV @ 25C							

231mV = Display Value + [(Display Temp. - 25 C) x (1.3 mV)]

WEATHER CONDITIONS

Date	Wind Direction	Wind Speed	Temperature F	Cloud Cover	Comments

Comments: _____

UMP013155

STANDARD OPERATING PROCEDURE

Purging Groundwater Wells

Revision 3

April 27, 2009

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord	Andrea Nord		04/27/09

Print	Field Technician(s)	Signature	Date
Kim Johannessen	Kim Johannessen		04/27/09

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
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Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	KSJ	Date:	4/4/2011
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures
Purging Groundwater Wells

Purpose

The purpose of this SOP is to describe the procedures for purging a well using a variety of techniques described in this SOP.

Applicability

This SOP applies to environmental technicians who are responsible for purging wells using one of the following techniques described in this SOP.

Definitions

Well purging is the removal of a known volume of water from a well so sampling can occur. This removal can be achieved by using two techniques: (1) without in-place plumbing; or (2) with in-place plumbing.

Drawdown: the lowering of the static water level due to the removal of the groundwater.
Note: See SOP for definition of static water level.

Equipment

Trisodium phosphate (TSP) solution
Tap water
Brush
Deionized water
Container marked in volume increments
Sterile gloves
Bailer (Stainless steel or new disposable polyethylene)
Peristaltic pump
Submersible pump
Nylon line
Discharge hose

References

Groundwater Sampling Guidelines by MPCA

Responsibilities

The environmental technician(s) is responsible for the proper well purging procedures; and documentation.

Discussion

Purging of a groundwater well is an important factor in the sampling process. It prepares the well by removing required volumes of water (according to the sampling plan) prior to sampling. The purging is needed to stabilize the well to allow for representative sample collection.
One method of purging is to pump the well until three to five times the volume of standing water in the well is removed. A second method is to pump the well until the groundwater's

specific conductance, temperature, pH, dissolved oxygen and oxidation reduction potential (ORP) stabilize. Normally, a combination of the two methods is used; i.e., specific conductance, temperature, pH, ORP and dissolved oxygen are measured at intervals and the volume purged is monitored. If a well is pumped dry, this constitutes an adequate purge and the well can be sampled following recovery. All well purging equipment will be cleaned between wells with tap water and Trisodium phosphate (TSP) solution and rinsed with tap water as described in the SOP for Tool Decontamination – Level I.

Purging can be done using a bailer, a peristaltic or a submersible pump.

Procedure

Well Purging

A. Technique for purging a well without in-place pumping:

1. Bailer

a. Put on gloves for skin protection and to prevent sample contamination.

b. Remove foil from bailer top (stainless steel), bailer body (stainless steel), and check valve (Teflon). A disposable polyethylene bailer can be used in place of the stainless steel bailer.

c. Connect all three parts together. It may be possible to connect additional bailer body pieces together, increasing the volume removed with each lowering of the bailer.

d. Using a cord reel or similar device, secure the bailer to the cord reel rope.

e. Empty the water collected from the bailer into a measuring bucket.

f. Continue the process, until the correct volume of water has been purged and the well has stabilized.
Note: See SOP for well stabilization testing.

g. Cut the rope from the bailer after purging is finished.

h. Place used top, bailer, and check valve in a dirty bailer cooler to be cleaned.
Note: If a disposable bailer was used, it cannot be reused and must be disposed of.

2. Bailer (H) – bailer hose

A bailer is used for slow-recovering wells with an inside diameter less than 2 inches and a depth to groundwater greater than 25 feet. A laboratory-cleaned stainless steel bailer with a Teflon check valve or new disposable polyethylene bailer with a check valve is attached to a downrigger and support assembly. Teflon-coated wire and stainless steel wire are both acceptable for hauling stainless steel bailers. Polyethylene bailers can be hauled using stainless steel wire or new nylon line.

1. Put on gloves to protect skin.

2. Remove foil from bailer (stainless steel) and check valve (Teflon).

3. Connect these two parts together and connect them to a 40-foot suction hose.
   Note: Bailer (H) can only be used on wells with total well depths of approximately 40 feet or less.

4. Lower the hose and bailer into the well until the bailer is partially submerged below the static water level.
   Note: If well goes dry, the bailer needs to be on the bottom of the well (due to drawdown).

5. Begin to surge the hose up and down; the result will be water pumping out of the well from the suction hose.

6. Collect purged water in a measuring bucket.

7. Continue to purge until the desired amount is purged or the well goes dry (see monitoring plan for volumes required to be purged).

8. Remove hose from well, put bailer and check valve in dirty bailer cooler, rinse hose with distilled water.

3. Centrifugal Pump

a. Put on gloves to protect skin.

b. Remove foil from bailer (stainless steel) and check valve (Teflon); connect together.

c. Connect bailer assembly to a 40-foot suction hose.

   Note: Centrifugal pumps will not pump at depths greater than 30 feet without surging (bailer [C]).

d. Submerge the bailer assembly with attached hose about 2 feet into the static groundwater.

e. Screw the other end of the hose onto the intake of centrifugal pump (make sure the connection is tight to ensure suction).

f. Prime the pump by pouring water into the priming water filler cap.

g. Start centrifugal pump:
   Step 1: turn pump on by the switch on the side of the pump
   Step 2: pull recoil rope to start pump

h. Surge hose to get the water up.

i. Continue priming until the water pumps by itself.

j. Adjust flow (with check valve located on discharge of the pump) to desired flow rate.

k. Check flow rate with the measurement bucket (gpm).

Note: If flow rates are under 1 gpm, the centrifugal pump should not be used.

l. Continue pumping until desired purge volumes are achieved.

m. Remove bailer and hose from well, turn off pump and disconnect the hose from the intake.

n. Disconnect bailer from the hose, put the bailer and check valve into the dirty bailer cooler; rinse the hose with distilled water.

o. Discharge purged water from pump by unscrewing the drain plug (bottom of the pumps); rinse pump.

4. Peristaltic Pump

This pump is used when the water level is within suction lift, i.e., within about 22 feet of the ground surface. It usually is a low-volume suction pump with low pumping rates suitable for sampling shallow, small-diameter wells.

a. Cut tubing to desired length.

b. Connect tubing to pump head, leaving 1 to 2 feet for discharge line.

c. Lower tubing into the well water (1 to 2 feet below surface).

d. Turn on pump and set speed at the desired rate of flow.

5. 4-inch Submersible Pump

This pump may be used to purge water samples from any depth. Variable rate submersible pumps are available to fit inside 2-inch or larger wells.

a. Put on gloves to protect skin.

b. Attach purging hose to the pipe connected on the top submersible pump.
Note: Either a 40- or 60-foot hose can be used, or both, whichever is appropriate.

c. Lower the submersible pump slowly into the well.

d. Lower pump until it is completely submersed into the water hang in casing.
Note: It can usually be lowered 5 to 6 feet under the water, unless draw-down in the well occurs.

e. Connect the pump to the generator with an extension cord.

f. Start the generator:

Step 1: turn switch to start
Step 2: put choke on
Step 3: pull recoil rope
Step 4: let generator idle until it is running smooth

g. Turn on power (which is located on the front of the generator).
Note: Submersible should be running; if not, turn off the generator and check connections.

h. Adjust flow rate to desired rate with the valve.

i. Measure the flow rate with the measuring bucket (gpm).

j. Turn off the generator after desired purge volume has been achieved.

k. Pull up the pump. The technician must be especially careful not to let the hose and wire get under or on the side of the pump.

l. Disconnect and disassemble all of the submersible pump apparatus; rinse accordingly.

6. 1.5-inch Submersible Pump

This is a type of submersible pump that can be used in 2-inch or larger diameter wells. It can purge water from depths down to 200 feet depending on pump model and manufacturer. This pump may be used as a submersible pump alternate for lower-volume wells.

a. Attach ⅜-inch tubing to pump intake and lower to desired depth.

b. Cut off tubing, allowing additional tubing length for discharge.

c. Plug the pump into the controller. Pump will begin pumping using the variable speed controller. There are a variety of speed controllers available, typically designed for a specific pump.

d. Attach the controller battery clips to the 12v DC power supply.

e. Turn on the controller and dial the speed control to the desired flow rate. This is especially useful if the well has low recharge rates. The controller can slow the purge rate down to the optimum rate.

7. 6-Inch Submersible Pump

a. Put on gloves to protect skin.

b. Attach hose reel onto well casing.

c. Loosen retainer pins from pump holder and place in well.

d. Loosen retainer pins from hose reel and lower pump with reel handle to desired depth (about 2 feet below static water level).

e. Connecting hoses and power cords:
Step 1: connect discharge hose to hose reel
Step 2: connect (110, 220 volt) controller power patch cord to hose reel
Step 3: connect controller power cord to appropriate 110, 220 receptacle on generator

f. Start the generator:
Step 1: turn switch to start
Step 2: put choke on
Step 3: pull recoil rope
Step 4: let generator idle until it is running smoothly

g. Turn on AC switch if applicable

h. Turn controller switch on (make sure LCD display reads zero before setting flow rate), adjust the flow rate with the speed control knob to desired rate.

i. Measure the flow rate with the measuring bucket (gpm).
Note: Submersible pump should be running; if not, turn off the generator and check connections.

j. Shut down system after desired purge volume has been achieved:
Step 1: turn controller switch off and turn speed control to zero
Step 2: turn off AC switch
Step 3: turn off generator
Step 4: disconnect controller power patch cord from generator
Step 5: disconnect controller power patch cord from hose reel
Step 6: disconnect discharge hose from hose reel

k. Unlock retainer pin and reel up the hose and submersible pump and lock into pump holder.

l. Rinse the hose and pump with distilled water.

B. With In-place Plumbing

1. **Dedicated pumps** are submersible pumps that are permanently installed in a well.

    a. Put on gloves to protect skin.

    b. Start the generator:
    Step 1: turn switch to "on"
    Step 2: turn on choke
    Step 3: pull recoil rope
    Step 4: let generator idle until it is running smooth

    c. Connect the pump to the generator with an extension cord.

    d. Connect the pipe, elbow, and valve to the discharge pipe of the submersible pump (located at the top of the well).

    e. Turn on power from the generator to the pump.
    Note: If the pump does not start, check the connection from the generator to the pump.

    f. When water flows from discharge of the pump, adjust the flow according to desired flow rate (using the discharge check valve).

    g. Use measuring bucket to determine the appropriate flow rate (gpm).

    h. After the appropriate purge volume is achieved. Sample collection can occur (before shutting off the generator and pump).

    i. Turn off the generator.

    j. Disconnect all of the appropriate connections and take the pipe, elbow, and valve off.

    Note: Each dedicated pump has its own pipe, elbow, and valve. These pieces are left at each well.

Discussion

In general, peristaltic pumps are used for wells with water levels less than 22 feet in depth. Submersible pump may be used for wells with lower water levels. Bailers are used for wells with water levels below 25 feet and diameters less than 2 inches.

When peristaltic pumps are used, only the intake line is placed into the well. When submersible pump are used, the pump and discharge hose are lowered into the water column.

The pump/hose assembly used in purging should be lowered into the top of the standing water column and not deep into the water. This is done so that the purging will "pull" water from the formation into the screened area of the well and up through the casing so that the entire static volume can be removed. If the pump/hose is placed deep into the water column, the water above the pump may not be removed, and the subsequent samples collected by

bailer may not be representative of the groundwater. The exception to placing the intake at the top of the water level is during low-flow purging and sampling. For low-flow purging and sampling the intake should be placed at or just above midscreen to capture water within the formation (see Barr's Low-flow Purging and Sampling SOP's).

If well recovery (groundwater reentering the well from the surrounding formation) is at least as rapid as the pumping rate, the pump/hose may be left hanging at the initial level until an adequate volume of water is removed. If the pumping rate exceeds the well's recovery rate, the pumping rate will be adjusted.

A laboratory-cleaned bailer with a Teflon check valve or new disposable polyethylene bailer with a check valve is attached to a support base and downrigger by stainless steel or Teflon-coated wire. The bailer assembly is lowered into the top of the water column. When the bailer has filled, it is removed from the well and the water is poured into a bucket marked in quarts/liters for volume measurement.

Purge Rate. The purge rate for a given well depends on several factors including the well volume and the depth to water. The well volume and depth to water will determine the type of pump used in purging the well. Different types of pumps give different types of flow rates. The flow rate will be determined in the field according to individual well performance. The purge rate with submersible pumps will be up to approximately 25 gpm. The purge rate with a peristaltic pump will be up to approximately 1 gpm. The purge rate should be held constant during stabilization testing.

Measuring Well Pumping Rate. If a flow meter is installed on the well, simply read the meter. If no meter is available, the pumping rate can be determined by using a container marked in volume increments such as quarts or liters and a stopwatch to time how long it takes for the container to fill with purge water. Be aware that changes in the flow rate will affect the amount of time required to purge the necessary amount of water from the well.

Measuring Purge Volume. The volume of standing water in the well is calculated first to determine the amount of purge water that needs to be removed from the well. The water level must be measured in order to determine the volume of standing water. The volume of standing water in the well is calculated using the following equation:

$$V = (\pi)(r^2)(h)$$

where: V = volume, in cubic feet
π = 3.14
r = radius of the well casing or hole (in feet)
h = height of the column of water in the well (in feet)

Then convert the volume of water standing in the well from cubic feet to gallons by multiplying the volume by 7.48.

Then determine the amount of water that must be purged by multiplying the gallons of standing water in the well by the number of well volumes that are required to be purged.

Documentation

The environmental technician(s) will document the procedures used in purging wells on the Field Log Cover Sheet, Field Log Data Sheet and or Field Log book.

Attachments

Attachment 1: Field Log Data Sheet

Attachment 1
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:	**Monitoring Point:**
Location:	**Date:**
Project #:	**Sample Time:**

GENERAL DATA		STABILIZATION TEST						
Barr lock:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						

Others present:

WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)

CASING & CAP: COLLAR: LOCK: OTHER:

MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:

VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-

oil,grease- bacteria- total metal- filtered metal- methane- filter-

Others:

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\FieldLogDataSheet.doc

STANDARD OPERATING PROCEDURE

Soil Sample Collection

Revision 3

March 23, 2010

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord			3/23/10

Print	Field Technician(s)	Signature	Date
KEVIN McGILP			3/23/10

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	JWJ	Date:	4-10-11
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedure for Soil Sample Collection

Purpose

To describe the collection of soil samples for volatiles, semivolatiles, metals, inorganics, bacteria, and dioxin analysis in soil.

Applicability

This procedure applies to the collection of soil samples by the sampling technician(s). It identifies each container type (volume, construction, preservative) required for each category of analyses, their corresponding holding times and collection procedures from a variety of sources.

Definitions

Holding Time. Period of time between sample collection and when the sample is analyzed.

Sample Preservation. The stability of analytes depends upon how well the samples are preserved.

Equipment

Sampler media
Pre-cleaned-certified Sampling Containers
Stainless Steel Spoons
Balance
Coolers
Ziploc® Baggy
Ice
Water-proof ink pen or pencil
Gloves
Alconox®
Chain of Custody Form
Sample Label
Custody Seal – if applicable
Field Sampling Report
Field Log Cover Sheet
Field Log Data Sheet

Responsibilities

The Field Operations/QA Officer or the environmental technician(s) will order the sample containers prior to the sampling event. The environmental technician(s) is responsible for the proper collection of soil samples, sample identification, quality control procedures, and documentation.

Procedure

Examples of samplers include split-barrel, split-barrel with brass liners, Geoprobe® sleeves, piston samplers, backhoe, or shovels may be used to retrieve soil from sampling locations. Depending upon the analyses to be conducted on the soil sample, the soil sample will either be sealed within the liner or sleeve or the soil sample will be transferred to a certified-laboratory-supplied container. The equipment required to transfer soil from the sampler to the sample container includes: stainless steel spoons, or scoops and the appropriate personal protective equipment necessary for collection and handling of soil. Volatile samples will be collected from representative areas of soil that were least disturbed first, then the remaining soil will be mixed and collected for the remaining analyses.

All soil sampling equipment will be carefully cleaned before and during soil sampling. All sampling tools including split-barrels, stainless steel spoons and scoops will be cleaned before use and between samples in the following manner: (1) clean with tap water and a phosphate–free detergent such as Alconox®, using a brush if necessary to remove particulate matter and films; (2) rinse three times with tap water; and (3) rinse three times with deionized water. To prevent sample cross-contamination, the sampler will discard the outer pair of sample gloves and put on a new pair between each sample event.

Collecting Volatile Organic Samples

Soil samples will be collected for analysis by either a drilling apparatus equipped with a split-barrel, core barrel sampler or by hand excavation. Volatile samples should be collected first. The soil selected for collection should be the most undisturbed sample possible.

It is important to note that there are different jar sizes and sampling media available for collecting a soil sample for volatile organic compounds (VOCs). The table below describes the sample volumes and preservation techniques for the most common sampling media.

Summary of Typical Sampling Media and Soil Volumes Used for Volatile Organic Compound Determination

VOC Sample Media	Preservative	Volume of Preservative (mL)	Volume of Sample (g)
2 oz. glass jar with PTFE-lined lid	MeOH, cool 4 °	10	10
	MeOH, cool 4 °	25	25
4 oz. glass jar with PTFE-lined lid	MeOH, cool 4 °	10	10
	MeOH, cool 4 °	25	25
Encore® Sampler			
5 gram device	Freeze or extrude into chemical preservative	Maintain a 1:1 ratio of soil to preservative if chemical preservation is used.	5
25 gram device	Freeze or extrude into chemical preservative	Maintain a 1:1 ratio of soil to preservative if chemical preservation is used.	25
Terracore® Kit			
1 MeOH and 2 water preserved glass vial	MeOH, cool 4 °	5	5
	Water Submersion, cool 4 °	5	5
1 MeOH and 2 sodium bisulfite preserved glass vials	MeOH, cool 4 °	5	5
	Sodium Bisulfite, cool 4 °	5	5

The following procedure applies to soil samples retrieved with a drilling apparatus equipped with a split-barrel sampler or core barrel with liners (Skip to the next section if Encore® sampler or other coring device is used):

1. Open the split-barrel sampler.

2. Open a representative liner containing soil.

3. Using a stainless-steel spoon, weigh the desired aliquot (25 g. or 10 g.) of a representative soil sample on a field balance. Once a weight/volume estimate has been established, discard the soil and collect untouched soil, from the same source for step 4.

4. Using a stainless-steel spoon, place soil in a laboratory-provided-pre-weighed sample container containing methanol (avoid splashing the methanol).

5. Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6. Cool the sample to approximately 4±2°C immediately after collection.

The following procedure applies to the collection of hand-excavated soil samples:

1. Dig to the desired sampling interval, exposing fresh soil surface to sample.

2. Collect a large sample on a shovel or in a bucket auger and bring it to the surface or collect the sample directly from the fresh soil surface.

3. Using a stainless-steel spoon, weigh the desired aliquot (25 g. or 10 g.) of a representative soil sample on a field balance. Once a weight/volume estimate has been established, discard the soil and collect untouched soil, from the same sample source for step 4.

4. Using a stainless-steel spoon, place the desired aliquot (25 g. or 10 g.) of soil in a pre-weighed-laboratory-provided sample container containing methanol (avoid splashing the methanol).

5. Wipe the jar lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6. Cool the sample to 4±2°C immediately after collection

Collecting Volatile Organic Samples with the Encore® Sampler or other soil coring device

The following procedure applies collecting VOC samples of soil with the Encore® sampler device:

1. Hold the Encore® coring body and push plunger down until small o-ring rests against tabs to ensure the plunger moves freely.

2. Depress locking lever on T-Handle. Place coring body plunger end first into the open end of the T- Handle, aligning the slots on the coring body with the locking pins in the T-Handle. Twist coring body clockwise to lock pins in slots. Check to insure sampler is locked in place.

3. Turn T-handle with T-up and coring body down. This positions the plunger bottom flush with bottom of coring body. Using T-Handle, push sampler into soil until coring body is completely full. When full the small o-ring will be centered in the T-Handle viewing hole. Remove excess soil from the coring body exterior.

4. Cap the coring body while it is still on the T-Handle. Push and twist cap over bottom until grooves on locking arms seat over ridge on coring body. Remove from T-Handle, lock plunger by rotating extended plunger rod fully counterclockwise until wings rest firmly against tabs, and attach label.

5. Cool the sample to 4±2°C immediately after collection.

Collecting Semivolatile Organic, Wet Chemistry and Metals Samples– except WI DRO

Soil samples will be collected for analysis by either a drilling rig equipped with a Geoprobe® sleeve, split-barrel, core barrel sampler or by hand excavation.

Please review the SOP for Direct Push Soil and Groundwater Sample Collection when Geoprobe® sleeves are used.

The following procedure applies to soil samples retrieved with a drilling rig equipped with a split-barrel sampler or core barrel with brass liners:

1. Open the split-barrel sampler.

2. Select a representative brass liner filled completely with soil.

3. Wrap the ends of the brass liners with heavy-duty aluminum foil, taking care to not piece the foil. Tape the foil to the brass liner with duct tape to ensure a seal. Cover the ends of the liner with plastic caps or duct tape to fully protect the foil.

4. Cool the sample to 4±2°C immediately after collection.

The following procedure applies to the collection of hand-excavated soil samples or core barrel samples:

1. Dig to the desired sampling interval, exposing fresh soil surface to sample.

2. Collect a large sample on a shovel or in a bucket auger and bring it to the surface or collect the sample directly from the fresh soil surface.

3. Using a stainless-steel spoon, homogenize the soil, pack the soil into the sample jars, leaving no headspace.

4. Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

5. Cool the sample to 4±2°C immediately after collection.

WI Diesel Range Organic (WIDRO) Samples

Soil samples will be collected for analysis by either a drilling apparatus equipped with a split-barrel, core barrel sampler or by hand excavation. Volatile samples should be collected first. The soil selected for collection should be the most undisturbed sample possible.

The following procedure applies to soil samples retrieved with a drilling apparatus equipped with a split-barrel sampler or core barrel with liners:

1. Open the split-barrel sampler.

2. Open a representative liner containing soil.

3. Using a stainless-steel spoon, weigh 25 ± 5 grams of a representative soil sample on a field balance. Once a weight/volume estimate has been established, discard the soil and collect untouched soil, from the same source for step 4.

4. Using a stainless-steel spoon, place 25 ± 5 grams of soil in a laboratory-provided-pre-weighed sample container.

5. Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6. Cool the sample to 4±2°C immediately after collection.

The following procedure applies to the collection of hand-excavated soil samples:

1. Dig to the desired sampling interval, exposing fresh soil surface to sample.

2. Collect a large sample on a shovel or in a bucket auger and bring it to the surface or collect the sample directly from the fresh soil surface.

3. Using a stainless-steel spoon, weigh 25 ± 5 grams of a representative soil sample on a field balance. Once a weight/volume estimate has been established, discard the soil and collect untouched soil, from the same sample source for step 4.

4. Using a stainless-steel spoon, place 25 ± 5 grams of soil in a pre-weighed-laboratory-provided sample container.

5. Wipe the container lip and screw threads to remove soil and provide a good sealing surface, and immediately screw on the lid.

6. Cool the sample to 4±2°C immediately after collection

Collecting Soil Quality Control Samples

Trip blanks are only used when sampling for volatile organics. Their purpose is to determine if contamination has occurred as a result of improper sample container cleaning, sample contamination during storage and transport due to exposure to volatile organics, or other environmental conditions during sampling or analysis. Trip blank samples are prepared prior to the sampling events by the laboratory providing the sample containers. The certified-pre-weighed methanol (MeOH) containers will be free of contaminants. The trip blank samples are prepared by the lab, sealed, labeled appropriately by the lab, and transported to the field in the same containers as the sample containers. These blanks are not opened in the field. They are transferred to the cooler designated for volatile sample storage and transport and accompany the samples to the analytical laboratory.

Field (or Masked) duplicate samples will be collected to measure relative sampling precision. Five percent of all samples collected are collected in duplicate. These samples are collected at the same time using the same procedures, equipment, and types of containers as the required samples. They are also preserved in the same manner and are either co-located or split and submitted for the same analyses as the required samples.

Some general considerations will be taken into account when planning and conducting sampling operations. The sampler will take into consideration the required sample volume, sample holding times, sample handling, and special precautions for trace contaminant sampling.

The volume of the sample obtained should be sufficient to perform all required analyses with an additional amount collected to satisfy the needs for quality control, split samples, or repeat examinations. The Laboratory Coordinator should be consulted for any specific volume requirements.

The elapsed time between sample collection and initiation of each laboratory analysis will fall within a prescribed time frame. Holding times for samples required by this project are prescribed by EPA: Title 40 of the Code of Federal Regulations.

After collection, all samples should be handled as few times as possible. Samplers should use extreme care to ensure that samples are not contaminated. If samples are placed in a cooler, samplers should ensure that melted ice cannot cause sample containers to become submerged, as this may result in cross-contamination. Plastic bags, such as Ziplock® bags, should be used when small sample containers are placed in coolers to prevent cross-contamination.

Some compounds can be detected in the parts per billion and/or parts per trillion range. Extreme care will be taken to prevent cross-contamination of these samples. A clean pair of new, disposable gloves will be worn for each sample location. Sample containers for source samples or samples suspected of containing high concentrations of contaminants are placed in separate plastic bags and coolers immediately after collecting, preserving and tagging. Sample collection activities will proceed progressively from the least contaminated area to the most contaminated area (when known).

Sample Storage

Immediately after samples are collected, they will be placed in a cooler containing bagged ice. Samples will be kept cold (approximately 4±2°C) until receipt at the laboratory, where they are to be stored in a refrigerated area. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of tape. All samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

Disposal

All waste generated by this process 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 environmental pollution.

Documentation

The technician(s) will document the soil sampling events in a project dedicated field logbook or on field log data sheets. They will also document the type and number of bottles the field log data sheets and chain-of-custody record. The analysis for each bottle and the laboratory used will be documented on the chain-of-custody record. The sampling request form will document which sampling containers are used for which soil samples.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Log Data Sheet

Attachment 1
Chain of Custody Form

Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Number of Containers/Preservative

COC _____ of _____

Project Manager: _____

Project QC Contact: _____

Sampled by: _____

Laboratory: _____

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	Type: QC	Water: VOCs (HCl) #1	Water: SVOCs (unpreserved) #2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (unpreserved) #3	Water: Diesel Range Organics (HCl)	Water: Nutrients (H_2SO_4) #4	Water:	Water:	Water:	Soil: VOCs (tared MeOH) #1	Soil: GRO, BTEX (tared MeOH) #1	Soil: DRO (tared unpreserved)	Soil: Metals (unpreserved)	Soil: SVOCs (unpreserved) #2	Soil: % Solids (plastic vial, unpres.)	Soil:	Soil:	Soil:	Total Number Of Containers
1.																													
2.																													
3.																													
4.																													
5.																													
6.																													
7.																													
8.																													
9.																													
10.																													

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐Air Freight ☐Federal Express ☐Sampler ☐Other: _____ Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

HRL GUSTOF CHMS\Chain Of Custody Form 2009 RLG Rev. 09/01/09

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

Preservative: _____

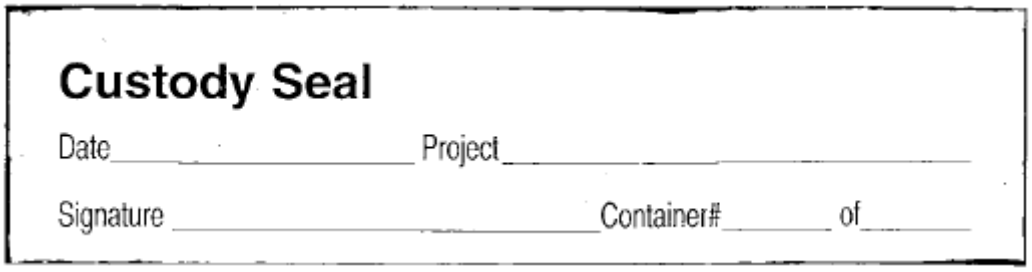
Sampled By: _____

Sample Location: _____

_____

UMP013176

Attachment 3
Custody Seal



Attachment 4
Field Log Data Sheet

Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:									Number of Containers/ Analysis																			
Location:																												
Project #:																												
Project Name:																												
Sample Identification	Collection		Matrix			Type																						
	Date	Time	Soil	Sludge		Grab	Comp.	QC	2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:				
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STANDARD OPERATING PROCEDURE

Soil Sample Collection Tools Decontamination – Level I

Revision 3

March 23, 2010

Approved By:

Andrea Nord		3/23/10
Print	QA Manager(s) Signature	Date
KEVIN McGILP		3/23/10
Print	Field Technician(s) Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	JWJ	Date:	4-10-11
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for the Soil Sample Collection Tools Decontamination – Level I

Purpose

The purpose of this SOP is to describe the proper techniques for equipment decontamination to meet level I protocol.

Applicability

This SOP applies to any field technician who is collecting environmental samples or is otherwise tasked with decontaminating field equipment for level I decontamination protocol.

Equipment

Tap water
Alconox®
Brush
Deionized water or distilled water
Bucket
Gloves

Responsibilities

The environmental technician(s) and/or Equipment technician is responsible for the proper equipment decontamination; quality control procedures and documentation.

Discussion

A variety of samplers (split-barrel, split-barrel with brass liners, piston sampler, backhoe, hand-auger, or shovel) may be used to retrieve soil from sampling locations. The soil sample will either be sealed within the sampler (e.g., collecting volatile samples) or the soil sample will be transferred to laboratory-supplied containers depending on the analysis to be conducted on the soil sample. The equipment required to transfer the soil from the sampler to the laboratory-supplied sample containers includes: stainless-steel spoons or scoops and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan.

Decontamination Procedures

All soil sampling equipment will be carefully cleaned before and during soil or sediment sampling. All sampling tools including split-barrels, stainless-steel spoons and scoops will be cleaned before use and between samples in the following manner:

1. Clean in a tap water and Alconox® solution, using a brush if necessary to remove particulate matter and films.

2. Rinse three times with tap water.

3. Rinse three times with deionized or distilled water.

4. Inspect equipment and repeat procedure if any residual soil or visible contaminants are present.

Disposal

All waste generated by this process 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 environmental pollution.

Documentation

The environmental and/or equipment technician is responsible for the proper decontamination of the equipment and the proper documentation in the Field Sampling Report and/or Field Log book.

Attachments

Attachment 1: Field Sampling Report

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

UMP013182

STANDARD OPERATING PROCEDURE

Soil Sample Collection Tools Decontamination – Level II

Revision 4

April 10, 2011

Approved By:

Andrea Nord		4/10/11
Print	QA Manager(s) Signature	Date

John W. Juntilla		4/10/11
Print	Field Technician(s) Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for the Soil Sample Collection Tools Decontamination – Level II

Purpose

The purpose of this SOP is to describe the proper techniques for equipment decontamination to meet level II protocol.

Applicability

This SOP applies to any field technician who is collecting environmental samples or is otherwise tasked with decontaminating field equipment for level II decontamination protocol.

Equipment

Tap water
Alconox®
Brush
Deionized water or distilled water
Bucket
Methanol
Aluminum Foil
Chem-wipe™
Gloves

Responsibilities

The environmental technician(s) and/or equipment technician is responsible for the proper equipment decontamination; quality control procedures and documentation.

Discussion

A variety of samplers (split-barrel, split-barrel with brass liners, piston sampler, backhoe, hand-auger, or shovel) may be used to retrieve soil from sampling locations. The soil sample will either be sealed within the sampler (e.g., collecting volatile samples) or the soil sample will be transferred to laboratory-supplied containers depending on the analysis to be conducted on the soil sample. The equipment required to transfer the soil from the sampler to the laboratory-supplied sample containers includes: stainless-steel spoons or scoops and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Project Health and Safety Plan.

Decontamination Procedures

All soil sampling equipment will be carefully cleaned before and during soil sampling. All sampling tools including split-barrels, stainless-steel spoons and scoops will be cleaned before use and between samples in the following manner:

1. Clean in a tap water and Alconox® solution, using a brush if necessary to remove particulate matter and films.

2. Rinse three times with tap water. Discharge water to the ground or collect investigative derived waste (IDW) as required by project-specific SAP or Work Plan.

3. Rinse three times with deionized or distilled water. Discharge water to ground or collect IDW as required by project-specific SAP or Work Plan.

.

4. Rinse once with methanol. Collect and containerize the methanol rinse.

5. Inspect equipment and repeat procedure if any residual soil or visible contaminants are present.

6. Dry sampler with Chem-wipe™ or appropriate disposable replacement.

At the completion of the work day, the samplers should be decontaminated following the procedure outlined above and wrapped in aluminum foil for storage.

Disposal

All waste generated by this process 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 environmental pollution.

Documentation

The environmental and/or equipment technician is responsible for the proper decontamination of the equipment and the proper documentation in the Field Sampling Report and /or Field Log book.

Attachments

Attachment 1: Field Sampling Report

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

_____
<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

UMP013186

STANDARD OPERATING PROCEDURE

Soil Sample Compositing

Revision 2

March 23, 2010

Approved By:

Print	QA Manager(s)	Signature	Date
Andrea Nord			3/23/10

Print	Field Technician(s)	Signature	Date
KEVIN McGILP			3/23/10

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	JWJ	Date:	4-10-11
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for Soil Sample Compositing

Purpose

The purpose of this SOP is not to define the representative number of sub-samples, but to describe the procedures of compositing several discrete samples into one representative sample for analysis.

Applicability

This SOP applies to samples collected from any site where it is determined that samples be composited prior to analysis at the laboratory.

Definitions

Sub-sample A representative, homogeneous portion or aliquot of a sample that is removed from an individual sample or the aggregate sample for preparation and measurement of the sample submitted for analysis.
Composite Sample A collection of more than one sample of the same medium from the same type of surface, such that multiple samples can be combined and analyzed as a single sample.
Discrete Sample A sample that originated from a specific area at a specific time.

Equipment

Stainless steel spoons or scoops
Large stainless steel mixing bowl
Pre-cleaned-certified Sampling Containers
Coolers
Ziploc® Baggy
Ice
Water-proof ink pen or pencil
Gloves
Tap water
Deionized or distilled water
Alconox®

References

Barr Engineering Co. SOP Soil Sample Collection

Responsibilities

The environmental technician(s) is responsible for the proper sample identification; field screening procedures; quality control procedures and documentation. The role of the Health and Safety Officer is to oversee all aspects of job safety. The Barr Project Manager in conjunction with the client develops the site specific work plan or sample and analysis plan to define the scope of work.

Discussion

Both composite and discrete samples can be used for environmental investigations. Composite samples are valuable in characterizing a large area or volume of soil. Detailed guidance for sub-sample collection is given from specific programs (e.g., Minnesota Department of Agriculture Agricultural Chemical Incidents; MCPA's Petroleum Remediation Program). In general, sampling the total investigation area and final numbers of sub-samples should be appropriate to meet the data quality objectives for the project. The work plan or SAP should contain the detailed information regarding the ratio of the total investigation soil area to final composited sample numbers.

Discrete soil samples identified for compositing can be collected in several ways. These include a drilling rig equipped with a split-barrel or core-barrel sampler, direct-push sampling equipment or by hand excavation. Additional information on soil sample collection can be found in the SOP for soil sample collection.

Procedure

The samples should be labeled discretely, and stored at 4ºC until each individual sample is obtained. A minimum volume of soil obtained during discrete sampling will be dependent on the final analytical requirements for the composite sample. A minimum volume of soil sufficient to fill two 4- or 8-ounce glass or Teflon containers should be obtained for compositing. This volume would be ample for analysis of semivolatiles, PCBs, pesticides, metals.

Note: Analytical samples should not be collected from polyethylene bags sometimes used for field screening purposes. Volatile organic samples should not be composited, due to aeration of the sample during mixing.

A. Sampling Equipment Preparation
   All soil compositing equipment will be carefully cleaned between uses in following manner: (1) clean with tap water and Alconox® using a brush, if necessary, to remove particulate matter and films; (2) rinse three times with tap water; and (3) rinse three times with deionized or distilled water. To prevent sample cross-contamination, the sampler will discard the outer pair of sample gloves and put on a new pair between each compositing event.

B. Compositing Discrete Samples

   1. After individual samples have been obtained, compositing begins by documenting the discrete sample locations to be included in a final composited sample. Appropriate laboratory containers should be labeled with this final sample identifier and the date of collection.

   2. Retrieve from storage the samples selected for compositing. One container from each discrete sample location should remain in storage in case individual sample confirmations are necessary.

   3. Empty the entire contents of each container into the stainless steel mixing bowl, removing any large debris or rocks. Mix thoroughly.

4. Fill appropriate laboratory sample containers.

5. Complete chain-of-custody documentation.

6. Immediately after samples are composited, they should be placed in a cooler containing ice or ice packs and cooled at 4°C for shipment to the laboratory.

Sample Storage

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Samples will be kept cold (approximately 4°C) until receipt at the laboratory, where they are to be stored in a refrigerated area. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of fiberglass tape. All samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or over night delivery courier in accordance with all Federal, State and Local shipping regulations.

Note: Samples may have to be stored indoors in winter to prevent freezing.

Investigative Derived Waste Sample Collection

Ensure enough representative sample is collected to provide adequate sample volume for all analyses used to characterize the IDW for disposal purposes. When collecting solid IDW sample volume, take moisture content into consideration when determining how much sample to provide to the laboratory for TCLP analysis. If the solid sample contains <5 percent by volume solid material, then enough liquid/solid sample must be obtained to provide the laboratory with an adequate supply to meet TCLP sample volume requirement guidelines. See the IDW Sample Collection SOP for further information.

Disposal

All waste generated by this process 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 environmental pollution.

Documentation

The technician(s) will document the soil sampling events on field log data sheets or project dedicated Field Log book. They will also document the type and number of bottles on both the field log data sheet and chain-of-custody record. The analysis for each bottle and the laboratory used will be documented on the chain-of-custody record. The sampling request form will document which sampling containers are used for which soil samples.

Attachments

Attachment 1: Chain of Custody Form
Attachment 2: Sample Label
Attachment 3: Custody Seal – if applicable
Attachment 4: Field Log Data Sheet - Soil

Attachment 1
Chain of Custody Form

Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Number of Containers/Preservative

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil	Type: Grab	Type: Comp.	QC	Water: VOCs (HCl) #1	Water: SVOCs (unpreserved) #2	Water: Dissolved Metals (HNO_3)	Water: Total Metals (HNO_3)	Water: General (unpreserved) #3	Water: Diesel Range Organics (HCl)	Water: Nutrients (H_2SO_4) #4	Water:	Water:	Water:	Soil: VOCs (tared MeOH) #1	Soil: GRO, BTEX (tared MeOH) #1	Soil: DRO (tared unpreserved)	Soil: Metals (unpreserved)	Soil: SVOCs (unpreserved) #2	Soil: % Solids (plastic vial, unpres.)	Soil:	Soil:	Total Number Of Containers
1.																												
2.																												
3.																												
4.																												
5.																												
6.																												
7.																												
8.																												
9.																												
10.																												

COC _____ of _____

Project Manager: _____

Project QC Contact: _____

Sampled by: _____

Laboratory: _____

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐Air Freight ☐Federal Express ☐Sampler ☐Other: _____ Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

HRL GUSTOF CHMS\Chain Of Custody Form 2009 RLG Rev. 09/01/09

Attachment 2
Example - Sample label

Client _____

Project Number. _____

Date: _____ Time _____

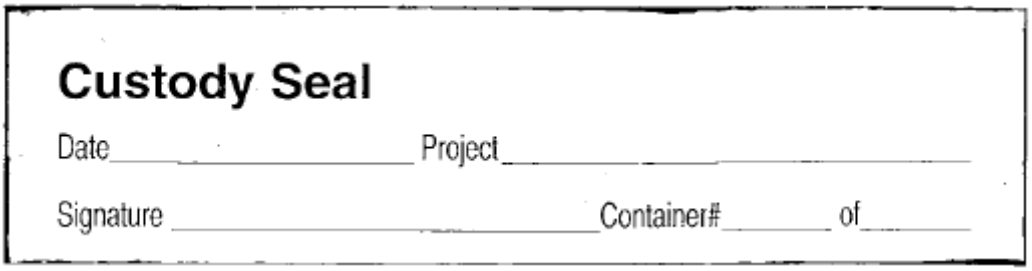
Preservative: _____

Sampled By: _____

Sample Location: _____

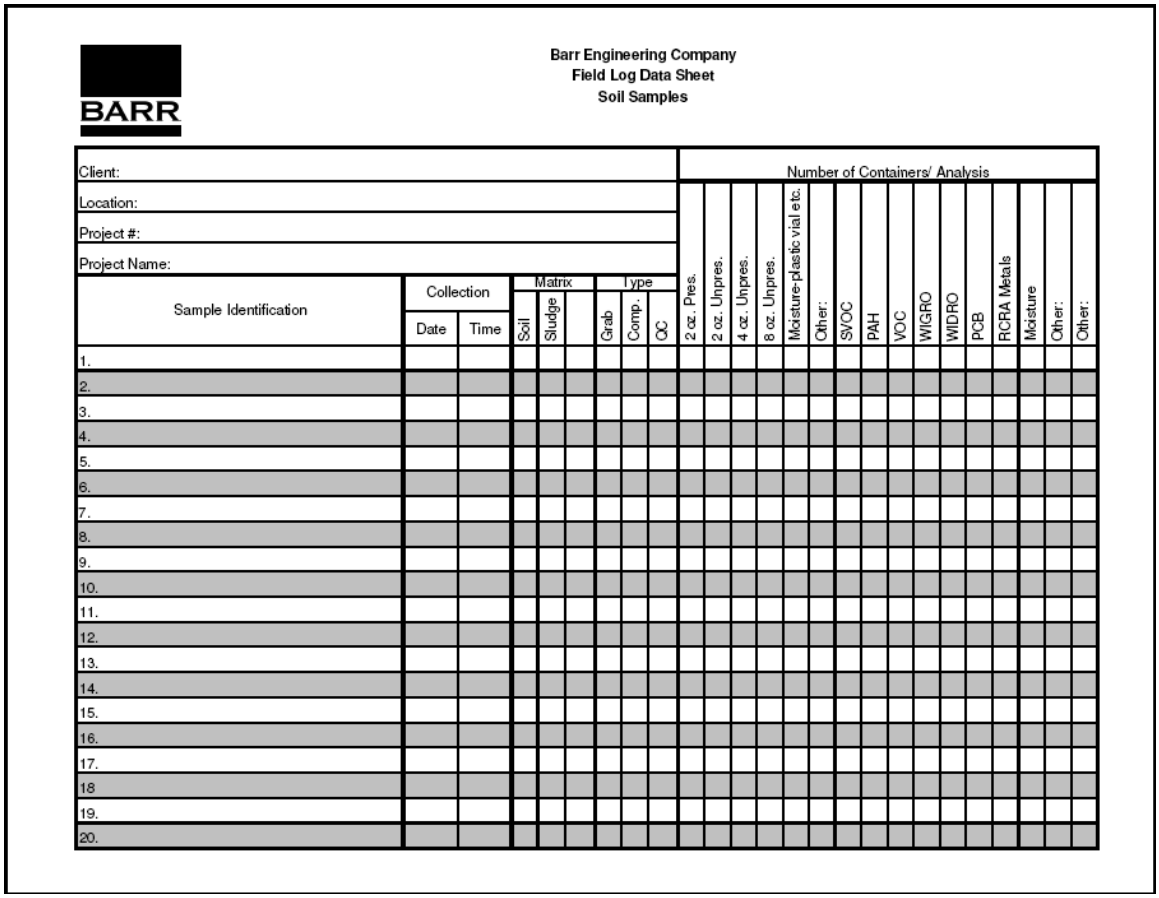
_____

Attachment 3
Custody Seal – if applicable



UMP013194

Attachment 4
Field Log Data Sheet



Barr Engineering Company
Field Log Data Sheet
Soil Samples

Client:
Location:
Project #:
Project Name:

Sample Identification	Collection		Matrix			Type			Number of Containers/ Analysis															
	Date	Time	Soil	Sludge		Grab	Comp.	QC	2 oz. Pres.	2 oz. Unpres.	4 oz. Unpres.	8 oz. Unpres.	Moisture-plastic vial etc.	Other:	SVOC	PAH	VOC	WIGRO	WIDRO	PCB	RCRA Metals	Moisture	Other:	Other:
1.																								
2.																								
3.																								
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STANDARD OPERATING PROCEDURE

For Well Stabilization and Well Stabilization Testing

Revision 2

March 1, 2011

Approved By:

Andrea Nord		03-01-11
Print	QA Manager(s) Signature	Date
Marta Nelson		03-01-11
Print	Field Technician(s) Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Standard Operating Procedures for
Well Stabilization and Well Stabilization Testing

Purpose

The purpose of procedure is to describe the methods used for well stabilization and stabilization testing of a well.

Applicability

Well stabilization is important to ensure that the water sampled will be representative of aquifer conditions.

Definitions

Stabilized When the required amount of water has been purged and the specific conductance, temperature, pH and potentially ORP of the groundwater are within acceptable limits for three consecutive readings.

ORP Reduction/oxidation potential. ORP is the potentiometric measurement in which the potential (or tendency) of the medium for electron transfer is sensed by an inert metal electrode and read relative to a reference electrode that is immersed in the same medium.

References

Quality Assurance Manual: Ground Water and Surface Water Sampling Procedures; Barr Engineering Co. Procedures for Groundwater Monitoring: MPCA Guidelines.

YSI Environmental. YSI Model 556 MPS Water Quality Monitoring System Operations Manual

YSI Environmental. *Measuring ORP on YSI 6-series Sondes: Tips, Cautions, and Limitations Tech Note*. YSI 2001/2005.

Responsibilities

The environmental technician(s) will be responsible for testing and recording stabilization test information.

Procedures

A. Well Stabilization:

1) Field Water Quality Measurements

Specific conductance, pH, temperature, dissolved oxygen and potentially ORP (oxidation reduction potential) will be measured in the field immediately before sample collection. These measurements, as well as measurement conditions and the steady-state value for each

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field water-quality parameter, will be recorded on the Field Log Data Sheet. Instrument calibration information will be recorded as part of the field sampling report.

All measurements will be taken within a closed flow-through cell designed to allow measurement of these parameters while minimizing changes in temperature, pressure, and dissolved gases from the in-situ aquifer environment. The flow-through cell has:

- Airtight fittings for installation of all probes.
- An intake that is connected directly to the pump discharge line.
- A discharge line that is connected to the flow-through cell with an airtight connection.

The following rules should be followed when using the flow-through cell:

- The flow-through cell will be shielded from strong winds and, on hot days, it will be shielded from direct sunlight.
- The flow of groundwater through the cell will be maintained as continuous and steady as practical throughout the measurement period.
- Discharge velocities through the cell should be kept below 1.5 gallons per minute.

The operation of the probes will be as follows:

- All probes will be fully immersed without touching the sides of the airtight, non-metallic flow-through cell.
- All probes will be allowed to equilibrate with well water before recording measurements.

Specific procedural details for measurement of individual field water-quality parameters are specified below. General care, maintenance, calibration procedures, and operation of each measurement device will follow manufacturer's specifications as detailed in the instruction/owner's manual for each device. Specific procedures for measurement of individual field water-quality parameters are described below.

Specific Conductance, Temperature, pH, and ORP (reduction/oxidation potential)

These measurements will be taken using the YSI Model 556 MPS Water Quality Monitoring System or equivalent. This device will be operated (including calibration) following the manufacturer's instructions.

Dissolved Oxygen

Dissolved oxygen measurements will be taken using the YSI Model 556 MPS dissolved oxygen meter or equivalent. Personnel using dissolved oxygen measuring equipment will have read the manufacturer's instruction manual once carefully before making dissolved oxygen measurements. Special care will be taken to store the probe in a humid environment and to otherwise protect the delicate membrane on the end of the probe. The membrane will be replaced every two to four weeks.

The dissolved oxygen meter will be calibrated according to manufacturer's specifications before taking measurements. When dissolved oxygen readings less than or equal to approximately 1.0 mg/L are expected, the meter will be calibrated in a mode that enhances accuracy at low concentrations. The calibration details will be recorded in the field log.

Measurements will be taken as follows:

a. The membrane at the tip of the probe will be checked visually to verify that it is in good condition.

b. After allowing the dissolved oxygen probe to equilibrate with a continuously replenished supply of aquifer water, the first measurement will be recorded.

To be considered valid, readings should appear stable on the display. If unstable readings are recorded, they will be footnoted and the unstable measurement conditions will be clearly stated in the final field sampling report. Readings will be reported to the nearest 0.01 mg/L dissolved oxygen.

2) Criteria for Stabilization

Field water-quality parameters will be measured for stabilization after each water-column volume is purged. One water-column volume is defined as the volume of a cylinder with a height (h) equal to that of the static water-column inside the well and a diameter (d) equal to the diameter of the well casing.

Volume = $\pi (d/2)^2 h$

Three consecutive measurements which meet the criteria listed below will be used to demonstrate stabilization:

- Temperature ± 0.5°C of the most recent reading (in degrees Celsius)
- Specific conductance (temperature corrected EC) - readings from 0 to 500 must be within ±5 µmhos/cm @ 25°C. Readings from 500 to 5,000 must be within ±50 µmhos/cm @ 25°C.
- Dissolved oxygen ±5% of the most recent reading (in mg/L)
- pH ±0.1 standard units of the most recent reading (in pH units)
- ORP Reading must be within ±0.01 units depending on the accuracy of the meter used.

Samples for laboratory analysis will be collected only after purging a maximum of five water-column volumes and achieving stabilization of field water-quality parameters. If field parameters do not stabilize after five water-column volumes, then field staff will verify that the probes and related equipment are functioning properly and that operator error is not an issue. Samples will be collected after five (or more) water-column volumes have been purged, even if field measurements have not stabilized. In such a case, the field log sampling and analysis report will clearly state that stabilization was not achieved.

B. Well Stabilization Testing:

Stabilization test samples are collected either from the flowing well discharge water or a bailer (depending on the purging method used). The sample is collected in a plastic bottle that has been rinsed three times with the sample.

Probes from both meters are placed in the collection bottle; readings are allowed to stabilize. Record the readings.

Documentation

The technician(s) shall document readings on the Field Log Data Sheet in the stabilization columns.

Attachments

Attachment 1: Field Sampling Report
Attachment 2: Field Log Cover Sheet
Attachment 3: Field Log Data Sheet

Attachment 1
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

-
-
-
-
-
-
-

Laboratory Analysis Status

<Name inserts here>
Environmental Technician

Document1

Barr Engineering Company ` 4700 W. 77th Street ` Minneapolis, MN 55435-4803 ` 952/832-2600

Attachment 2
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

Barr Engineering Company ' 4700 W. 77th Street ' Minneapolis, MN 55435-4803 ' 952/832-2600

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Attachment 3
Field Log Data Sheet

Barr Engineering Company
Field Log Data Sheet

Client:				**Monitoring Point:**				
Location:				**Date:**				
Project #:				**Sample time:**				
GENERAL DATA		**STABILIZATION TEST**						
Barr lock:		Time/ Volume	Temp. ºC	Cond. @ 25	PH	ORP mV	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*		NA						
Static well level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume purged:								
Duplicate collected:								
Sample collection by:								
Others present:		Well condition:						
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment Other: sump								
VOC Semi-volatile General Nutrient Cyanide DRO Sulfide								
Oil, grease Bacteria Total Metal Filtered Metal Methane Filter								
Others:								

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Appendix F

Barr Data Validation Standard Operating Procedures

STANDARD OPERATING PROCEDURE

Routine Level General Chemistry Data Validation

Revision 3.0

April 8, 2011

Approved By:	Andrea Nord		4/8/11
	Print QA Manager(s)	Signature	Date

Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone: 952-832-2600 · Fax: 952-832-2601 · www.barr.com

Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO • Bismarck, ND

Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	
Initials:		Date:	

Standard Operating Procedures for Routine Level General Chemistry Data Validation

Purpose

This SOP is intended as a guidance document for the routine level validation of general chemistry data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine general chemistry data validation including a variety of approved methods not limited to the following analyses:

Chromium VI (Hexavalent Chromium)	Nitrate (or Nitrite) only
Alkalinity as $CaCO_3$	Nitrate + Nitrite
Ammonia	pH – *in lab*
BOD (Biological Oxygen Demand)	Phosphorus, total
COD (Chemical Oxygen Demand)	Sulfate
Chloride	Sulfide
Conductance, Specific – *in lab*	Total Dissolved Solids (TDS)
Cyanide (CN^- as HCN)	Total Kjeldahl Nitrogen (TKN)
Fluoride	Total Organic Carbon (TOC)
Hardness	Total Suspended Solids (TSS)
HEM (Oil and Grease)	

In the case of specific analyses not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

Definitions

Blank. A sample designed to assess specific sources of contamination.

BOD. Biological Oxygen Demand. The BOD test is an empirical bioassay-type test which measures the dissolved oxygen consumed by microbial life while assimilating and oxidizing organic matter in a sample.

COD. Chemical Oxygen Demand. The COD test determines the quantity of oxygen required to oxidize organic matter in a waste sample.

Contamination. A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

Duplicate. A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank. Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the Laboratory. A field blank includes trip blanks, equipment blanks, etc.

Field Duplicate. A duplicate sample generated in the field, not in the Laboratory.

HCl. Hydrochloric acid. Used as a sample preservative in some analyses.

HNO_3. Nitric acid. Used as a sample preservative in some analyses.

H_2SO_4. Sulfuric acid. Used as a sample preservative for some analyses.

Initial Calibration. Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument.

Instrument Blank. A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD). A control sample of known composition. LCSs and LCSDs are processed using the same sample preparation, reagents, and analytical methods employed for samples received. Sometimes referred to as a LFB (Laboratory Fortified Blank).

LFB. Laboratory Fortified Blank. See *Laboratory Control Sample*.

Matrix. The predominant material of which the sample to be analyzed is composed (e.g. water, soil, sediment, etc.)

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS). Aliquot of the sample 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.

Matrix Spike Duplicate (MSD). A second aliquot of the same as the MS sample that is fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine precision of the method.

Method Detection Limit (MDL). The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99% probability that it is different from the blank. MDL studies performed by the laboratory should be consistent with SW-846, Ch. 1.

Method Blank. An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

NaOH. Sodium hydroxide. Used as a preservative in some analyses.

Narrative. Portion of the data package which includes laboratory, contact, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Retention Time (RT). The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

TDS. Total Dissolved Solids. The amount of filterable residue in a given water sample.

TKN. Total Kjeldahl Nitrogen. The combination of organically bound nitrogen and ammonia (NH_3 and NH_4^+) in biological wastewater.

TOC. Total Organic Carbon. The carbon bound in an organic compound in waters and used as an indicator of water quality. Source of nutrients for undesirable biological growth.

TSS. Total Suspended Solids. The amount of non-filterable residue in a given water sample.

ZnAc + NaOH. Zinc acetate and sodium hydroxide. Used as a preservative of samples in the analysis for sulfide.

Equations

For % Recovery (%R or %Rec):

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

Where,

%R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample
In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of the associated approved analytical methods (EPA, ASTM, NPDS, etc.) and *Standard Methods for the Examination of Water and Wastewater*, 20th Ed. (Parts 1020A and 1020B).

Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

All samples should meet acceptance criteria for their respective analyses (and matrices) in the charts attached to the end of this SOP

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Attachments 1* and *2*, consider qualification with an "**h**".

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before (receipt). While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

It is understood that the method recommends that pH is a parameter that should be measured in the field. However, for conformational measurements in the laboratory, a recommended maximum holding time of 7 days from sample collection will be used for as a guideline for qualification. QAPP and SAP requirements may differ from this recommendation and professional judgment should be applied before qualifying any data.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

While not required for all methods, method blanks are recommended for all but pH analyses. Refer to *Attachments 1 and 2* at the end of this SOP for individual method requirements for method blank evaluation.

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the method blank. In such cases, it may be appropriate to qualify the affected data with "**" (usable value, QA/QC criteria not met).

Table 1 – Guidelines for the Evaluation of Blank Contamination	
Sample Result	**Recommended Action**
Non-detect	No action required
<5x blank concentration	Qualify with "**b**"
>5x blank concentration	Use professional judgment
Gross contamination	Qualify associated samples with "**"

Note: "**" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. Per method (laboratories may have developed other acceptable QC measures), LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix

Not all methods require an LCS (or equivalent, such as a LFB). *Attachments 1 and 2* should be consulted to determine those analyses that require an LCS.

Laboratory control samples contain a known amount of each target compound and the percent recoveries are evaluated based either on the laboratory's internally-generated acceptance windows or default acceptance criteria when laboratory limits are not assigned generally fall between 75-125% recovery. *Table 2* presents the recommended guidelines for evaluating LCS/LCSD recoveries and qualification of samples from the associated batch.

Table 2 – LCS/LCSD Recovery Guidelines		
Spike Recovery	**Sample Concentration**	**Recommended Action**
< Lower Limit	Non-detect	Qualify with "*****" If LCS recovery is < 10%, consider "******"
	Detected	Qualify with "*****"
Between Lower and Upper Limits	Non-detect or Detected	Acceptable, no qualification.
> Upper Limit	Non-detect	No qualification required.
	Detected	Qualify with "*****"; If LCS recovery is >> upper limit, use professional judgment

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met.

IV. Laboratory Duplicates

Laboratory duplicates are analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and performance evaluation (PE) standards may not be used for duplicate analysis and are only evaluated for samples with concentrations greater than five times (>5x) the MDL. When methods require duplicates, they should be analyzed for each matrix.

In general, laboratory duplicates should be analyzed 1 duplicate in every 20 sample (where required). In some cases, a matrix spike duplicate may be considered an acceptable laboratory duplicate for methods requiring a matrix spike.

Duplicate results are compared to the associated native result for evaluation, using the equation for RPD defined in the *Equations* section in the beginning of this SOP.

RPD values are calculated only for results above the reporting limit and only if the following qualifiers do not apply: b, U, < and **.

Use laboratory acceptance criteria to evaluate RPDs, when available. The guidelines in *Table 3* may be used when laboratory acceptance criteria is not available.

Table 3 – Duplicate RPD Guidelines	
Matrix	**Recommended Action**
aqueous	if RPD is <20%, no action is required
	if RPD is >20%, but both results are <5x RL, no action is required
	if RPD is >20% and both results are >5x RL, qualify with *****
soil/sediment	if RPD is <35%, no action is required
	if RPD is >35%, but both results are <5x RL, no action is required
	if RPD is >35% and both results are >5x RL, qualify with *****

If both samples are non-detect, the RPD is not calculated.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are also used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the same equation as found in the *Equations* section in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs of <20-30% for aqueous samples and <30-40% for soil or sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample is detected and greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results based on field duplicate RPDs.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix may have on the sample preparation procedures and analytical results. While not required by every method, matrix spikes are typically analyzed 1 in 20 samples where required.

However, the frequency may also be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.).

If a matrix spike does not meet acceptance criteria and is not associated with the specific project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (greater than 4 times the native concentration (>4x)), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery defined in the *Equations* section in the beginning of this SOP.

If laboratory or QAPP acceptance criteria are provided for the percent recoveries of matrix spikes, those criteria should be used for the evaluation of the data. If acceptance criteria are not provided, percent recoveries between 75-125% are generally acceptable for matrix spikes.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity.

In general, matrix spikes and matrix spike duplicates may be evaluated as follows:

Table 4 – MS/MSD Recovery Guidelines

% Recovery of MS/MSD	Native Concentration	Recommended Action
<< Lower Limit (e.g. < 20%)	Non-detect	Consider qualifying with "**"
	Detected	Qualify with "*"
< Lower Limit	Non-detect	Qualify with "*"
	Detected	Qualify with "*"
Between Lower and Upper Limits	Non-detect or Detected	No qualification required
> Upper Limit	Non-detect	No qualification required
	Detected	Qualify with "*"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS/MSD pairs using the equation for RPD evaluation in the *Equations* section in the beginning of this document. Generally, acceptance criteria for MS/MSD are <20-30% RPD for aqueous samples and <30-40% for soils/sediments, but professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Attachments

Attachment 1: QC/QA Recommendations and Requirements Chart for Water Samples
Attachment 2: QC/QA Recommendations and Requirements Chart for Soil Samples
Attachment 3: Routine Level Quality Control Report
Attachment 4: Barr Qualifiers/Footnotes
Attachment 5: Revisions to SOP

Attachment 1
QC/QA Recommendations and Requirements Chart for Water Samples

Parameter (*Alternate Name*)	Recommended Hold Time						Required Preservation						QC Requirements					
	24 Hours (or ASAP)	48 Hours	7 Day	14 Day	28 Day	180 Day	Ice Only (or ≤ 6°C)	HCl	HNO_3	H_2SO_4	NaOH, Ascorbic Acid	ZnAc + NaOH	Method Blank	LCS	LSCD	Duplicate	MS	MSD
Chromium VI (*Hexavalent Chromium*)	X						X						X	X				
Alkalinity, as $CaCO_3$				X			X						R	R		R	R	
Ammonia					X		X			X			X	X		R	X	
BOD (*Biological Oxygen Demand*)		X					X						R			R		
COD (*Chemical Oxygen Demand*)					X		X			X			X			R		
Chloride by EPA 300.0					X		B						X	X	O	O	X	O
Chloride by EPA 9056	X						X						X	X		X	X	X
Conductance, specific – *in lab*					X		X						R	R		R		
Cyanide (*CN^- as HCN*)				X			X				X		X	X			X	
Fluoride by EPA 300.0					X		B						X	X	O	O	X	O
Fluoride by EPA 9056	X						X						X	X		X	X	X
Hardness						X			X				R	R		R		
Nitrate (or Nitrite) only by EPA 300.0		X					X						X	X		O	X	O
Nitrate (or Nitrite) only by EPA 9056	X						X						X	X		X	X	X
Nitrate + Nitrite					X					X			X	X			X	
Oil and Grease (HEM)					X		X	X[b]		X[b]			X	X			X	R
pH[a] – *in lab*			X				X							R		R		
Phosphorus, total					X		X			X			R	R		R	R	
Sulfate by EPA 300.0					X		X						X	X	O	O	X	O
Sulfate by EPA 9056	X						X						X	X		X	X	X
Sulfide			X									X	R	R		R	X	
Total Dissolved Solids (*TDS*)			X				X						R	R	R	R		
Total Kjeldahl Nitrogen (*TKN*)					X					X			R	R		R	R	
Total Organic Carbon (*TOC*)					X			X[b]		X[b]			X	R		R	X	
Total Suspended Solids (*TSS*)			X				X						R	R	R	R		

a Preferably in the field, otherwise 7 days
b Either preservative may be used (to pH <2)
R Recommended QA/QC test, not method requirement
X Method requirement
O Optional requirement (one must be used)
B No preservation is required, but ice is recommended for all samples

UMP013216

Attachment 2
QC/QA Recommendations and Requirements Chart for Soil Samples

Parameter (*Alternate Name*)	Recommended Hold Time					Required Preservation					QC Requirements					
	24 Hours (or ASAP)	48 Hours	7 Day	14 Day	28 Day	Ice Only (or ≤ 6°C)	HCl	H_2SO_4	NaOH	ZnAc + NaOH	Method Blank	LCS	LSCD	Duplicate	MS	MSD
Chromium VI (*Hexavalent Chromium*)					X	X					X	X		O	O	
Ammonia					X			X			X	X		R	X	
Chloride					X	X					X	X	O	O	X	O
Cyanide (*CN^- as HCN*)				X					X		X	X			X	
Fluoride					X	X					X	X	O	O	X	O
Nitrate (or Nitrite) only		X				X					X	X		O	X	O
Nitrate + Nitrite					X			X			X	X			X	
pH[a] – *in lab*			X			X						R		R		
Phosphorus, total					X			X			R	R		R	R	
Sulfate					X	X					X	X	O	O	X	O
Sulfide			X							X	R	R		R	X	
Total Kjeldahl Nitrogen (*TKN*)					X			X			R	R		R	R	
Total Organic Carbon (*TOC*)					X		X[b]	X[b]			X	R		R	X	

Preferably in the field, otherwise 7 days
b Either preservative may be used (to pH <2)
R Recommended QA/QC test, not method requirement
X Method requirement
O Optional requirement (one must be used)

Attachment 3
Routine Level Quality Control Report

Barr Engineering Company
Routine Level Quality Control Report

Project #		**Project Name**	
Laboratory		**COC(s)/Event**	
Lab Report #		**Matrix**	
Report Date		**Review Date**	
Holding Times Met	Yes No	**Reviewed By**	
If no, comments		**Exceptions**	
Temps on Receipt (°C)			

Method Blanks	**LCS/LCSD**
Field Blanks	**MS/MSD**
Trip Blanks (VOCs Only)	
Field Duplicates (if applicable)	**Surrogates (if applicable)**
	Lab Duplicates (if applicable)

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Additional Notes (include historical comparison, if appropriate)

Qualifier Summary		Qualifier Changes		
Sample Name	Parameter	Added	Removed	Retained

Additional Notes to DM

For DM Use Only	
Equis Y / N	Data Tech Init:
Facility ID:	Date entered into Equis:

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Attachment 4
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes	
Qualifier	**Definition**
--	Not analyzed/not available.
a	Estimated value, calculated using some or all values that are estimates.
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Relative percent difference is >40% (25% CLP pesticides) between primary and confirmation GC columns.
pp	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.
N	Sample Type: Normal
FD	Sample Type: Field Duplicate
AT	Sample chromatogram is noted to be atypical of a petroleum product.
DLND	Not detected, detection limit not determined.
DF	Did not flash
EMPC	Estimated maximum possible concentration.
NA – (Not applicable)	NA indicates that a fractional portion of the sample is not part of the analytical testing or field collection procedures.
ND	Not detected.
TIC	Tentatively identified compound
BQA	Barr-applied project specific qualifier: extraction and/or analyses conducted using an alternative method and/or procedure.
BQC	Barr-applied project specific qualifier: plant shut down.
BQD	Barr-applied project specific qualifier: equipment malfunction.
BQE	Barr-applied project specific qualifier: equipment adjustment.
BQM	Barr-applied project specific qualifier: manual measurement.
BQN	Barr-applied project specific qualifier: unable to be sampled or measured due to various reasons.
BQP	Barr-applied project specific qualifier: atypical chromatographic pattern.
BQQ	Barr-applied project specific qualifier: some aspect of QA/QC was not met.
BQR	Barr-applied project specific qualifier: location was re-sampled.
BQS	Barr-applied project specific qualifier: data is considered suspect.
BQT	Barr-applied project specific qualifier: summed value not displayed due to insufficient field length.
BQU	Barr-applied project specific qualifier: historical qualifier - definition unknown.
BQV	Barr-applied project specific qualifier: estimated value.
BQX	Barr-applied project specific qualifier: see notes for qualifier definition.
BQZ	Barr-applied project specific qualifier: data is considered unusable.

Attachment 5
Revisions to SOP

Revision Number	Date of Revision	Section	Revision Made
2.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		Attachments	Added Attachment 5
		Attachment 1	Corrections to hold times and preservation requirements
		Attachment 2	Corrections to hold time requirements
3.0	04/2011	Attachment 1, 3, 4	Updated Attachments 1,3 and 4 to include current forms and added/updated analytical method requirements.

STANDARD OPERATING PROCEDURE

Routine Level Metals Data Validation

Revision 3.3

April 18, 2011

Approved By:	Andrea Nord	Cindy Nord	04-08-11
	Print	QA Manager(s) Signature	Date

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Annual Review of the SOP has been performed
and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for Routine Level Metals Data Validation

Purpose

This SOP is intended as a guidance document for the routine level validation of metals data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine metals data validation for analysis by:

- ICP/AES (Methods EPA 200.7, EPA 6010B or EPA 6010C)
- ICP/MS (Methods EPA 200.8, EPA 6020 or EPA 6020A)
- Mercury (Methods EPA 245.1/245.5, EPA 7470A7471A/7471B and EPA 1631E (including appendix)
- Any of the above in conjunction with TCLP procedure (EPA 1311)

Validation of Contract Laboratory Program (CLP) data should be performed in accordance with to the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (January 2010)*.

Definitions

AFS. Atomic Fluorescence Spectroscopy. A flame is used to solvate and atomize the sample, and a lamp emits light at a specific wavelength into the flame to excite the analyte atoms in the flame. The atoms of certain elements fluoresce and emit light in a different direction. The intensity of this fluorescing light is used for quantifying the amount of analyte element in the sample.

Blank. A sample designed to assess specific sources of contamination.

Duplicate. A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank. Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the Laboratory. A field blank includes trip blanks, equipment blanks, etc.

Holding Time. The maximum recommended amount of time samples may be held before they are processed.

HNO_3. Nitric acid. Used as a preservative.

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD). A control sample of known composition. LCSs and LCSDs are processed using the same sample preparation, reagents, and analytical methods employed for samples received.

UMP013223

Matrix. The predominant material of which the sample to be analyzed is composed (e.g. water, soil, sediment, etc.)

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike* (MS) *and Matrix Spike Duplicate (MSD). Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology.

Method Detection Limit (MDL). The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99% probability that it is different from the blank.

Method (Preparation) Blank. An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Narrative. The portion of the data package which includes laboratory, contact, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Reporting Limit (RL). The RL is the lowest reported concentration, provided on the sample-analysis data report, after corrections have been made for sample dilution, sample weight, and (for soils and sediments) amount of moisture in the sample.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

TCLP. Toxicity Characteristic Leaching Procedure. A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

Equations

For % Recovery (%R or %Rec):

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

Where: %R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample

In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where: RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004)* and quality control recommendations outlined in:

- **EPA Methods 200.7/6010B/6010C** – *"Determination of Metals in Waters and Wastes by ICP-AES"*, 1994/February 2007
- **EPA Methods 200.8/6020/6020A –** *"Determination of Trace Elements in Waters and Wastes by ICP-MS"*, 1994/February 2007
- **EPA Methods 245.1/245.5** – *"Determination of Mercury in Water by CVAAS/ Automated Cold Vapor Technique"*, 1994/1974
- **EPA Method 1631E (including Appendix)** – *"Mercury in Water by Oxidation, Purge and Trap, and CVAAS"*, August 2002
- **EPA Methods 7470A/7471B** – *"Mercury in Liquid/Solid Waste (Manual Cold Vapor Technique)"*, September 1994/February 2007

Responsibilities

The laboratory is responsible for generating metals data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the metals data in accordance with this document, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

The recommended hold time and preservation acceptance criteria are in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temperature	Preservative	Maximum Hold Time
Mercury	aqueous	--	HNO_3 < 2 pH	28 days
	aqueous (low level)	< 6° C	Pre-tested hydrochloric acid or bromium chloride	28 days
	sediment/soil	< 6° C	ice	28 days
	sediment/soil (low level)	< 6° C	Pre-tested hydrochloric acid or bromium chloride	28 days
All other metals	aqueous	< 6° C	HNO_3 < 2 pH	180 days
	sediment/soil	< 6° C	ice	180 days

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil) may not be necessarily be subjected to the same hold time recommendations.

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before receipt by the laboratory. While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

Low-level mercury considerations

Low-level mercury (Method 1631E) must be collected directly into a specially cleaned, pretested, fluoropolymer bottle using sample handling techniques specially designed for collection of mercury at trace levels and preserved with pre-tested hydrochloric acid (required for methyl mercury) or bromium chloride. Borosilicate glass bottles may be used if mercury is the only target analyte. Samples not collected in the correct type of container may be qualified with an "**h**". These samples may be shipped unpreserved provided:

- the sample is collected in a fluoropolymer bottle
- the bottle contains no headspace and is capped tightly
- sample temperature was maintained between 0-4°C, and
- the samples are acid-preserved within 48 hours of sampling.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group, or each batch digested (whichever is more frequent).
- Field blank collection and analysis frequency is project-specific.
- Low-level mercury method requires *at least* three method blanks per run per analytical batch.

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the method blank. In such cases, it may be appropriate to qualify the affected data with "******" (usable value, QA/QC criteria not met).

Table 2 – Guidelines for the Evaluation of Blank Contamination

Sample Result	Recommended Action
Non-detect	No action required
<5x blank concentration	Qualify with "b"
>5x blank concentration	Use professional judgment
Gross contamination	Qualify associated samples with "******"

Note: "******" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix
- For low-level mercury, ongoing precision and recovery (OPR) samples are run before and after each analytical batch. Quality control samples (QCS) should be from a different source and analyzed once per analytical batch.

Laboratory control samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 9) and the percent recoveries are evaluated based either on the laboratory's internally generated acceptance windows or default method criteria (as given in *Table 3*).

Table 3 – Guidelines for Laboratory Control Sample Recoveries

Matrix	Acceptance Criteria	Action
aqueous	80% to 120% recovery	if LCS > upper limit and samples are non-detect, no action; if detections, qualify with "*"
		if LCS is between < lower limit, use professional judgment when considering qualifying with "*"
		if LCS is << lower limit and samples are non-detect, qualify with "**"; if detections, qualify with "*"
sediment/soil	70% to 130% recovery	if LCS > 130%, and samples are non-detect, no action; if detections, qualify with "*"
		if LCS < 70% qualify detections with "*"; use professional judgment when considering non-detections with "**"

Note: "*"indicates the reported value is estimated and QA/QA criteria were not met. "**" indicates the reported value is unusable and QA/QC criteria were not met.

IV. Laboratory Duplicates

Laboratory duplicates are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and performance evaluation (PE) standards may not be used for duplicate analysis and duplicate RPDs are only evaluated for samples with concentrations greater than five times (>5x) the MDL. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water), or
- One from each SDG

MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Duplicate results are compared to the associated native result for evaluation, using the equation for RPD defined above.

Use laboratory acceptance criteria to evaluate RPDs, where available. When acceptance criteria is not available, use the following:

Table 4 – Guidelines for Laboratory Duplicate RPDs	
% RPD	**Action**
RPD is < upper limit	no action is required
RPD is > upper limit	if both results are <5x RL, no action is required
RPD is > upper limit	if both results are >5x RL, consider qualifying with "***".**

Note: "*"indicates the reported value is estimated and QA/QA criteria were not met.

If both samples are non-detect, the RPD is not calculated.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are also used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs between 20-30% for aqueous samples and 30-40% for soil and sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample is detected and greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix may have on the sample preparation procedures and analytical results. Matrix spike and matrix spike duplicate samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 9).

Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix, or
- 1 per sample delivery group

However, the frequency may be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.). In some cases, MS/MSD analysis is not required.

If a matrix spike does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery can not be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery provided in the *Equations* section in the beginning of this SOP.

If laboratory acceptance criteria are provided for the percent recoveries of matrix spikes, those criteria should be used for the evaluation of the data. If acceptance criteria are not provided, percent recoveries between 75-125% are generally considered acceptable for matrix spikes.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity.

Table 5 – Guidance for Matrix Spike Evaluation	
Spike Result	**Recommended Action**
% recovery > upper acceptance limit	Non-detects, no qualification
	Detects, qualify with "*****"
% recovery meets acceptance limits	No qualification
% recovery is between 20% and lower acceptance limit	Non-detects, qualify with "*****"
	Detects, qualify with "*****"
% recovery is below 20%	Non-detects, use professional judgment; consider qualifying with "******"
	Detects, qualify with "*****"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation for RPD evaluation in the *Equations* section in the beginning of this document. Generally, acceptance criteria for MS/MSD are <20-30% RPD for aqueous samples and <30-40% for soils/sediments, but professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Additional Tables

Table 9 – Number of Target Compounds Required by NELAP and MN Rules for LCS/LCSD and MS/MSD samples	
Number of Target Parameters	**Required Number of Spiked Compounds**
1-10 analytes	Spike *all* compounds
11-20 analytes	*At least* 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike *at least* 16 compounds

X. Attachments

Attachment 1: Routine Level Quality Control Report
Attachment 2: Barr Qualifiers/Footnotes
Attachment 3: Revisions to SOP

Attachment 1
Routine Level Quality Control Report

Barr Engineering Company
Routine Level Quality Control Report

Project #		**Project Name**	
Laboratory		**COC(s)/Event**	
Lab Report #		**Matrix**	
Report Date		**Review Date**	
Holding Times Met	Yes No	**Reviewed By**	
If no, comments		**Exceptions**	
Temps on Receipt (°C)			

Method Blanks	**LCS/LCSD**
Field Blanks	**MS/MSD**
Trip Blanks (VOCs Only)	
Field Duplicates (if applicable)	**Surrogates (if applicable)**
	Lab Duplicates (if applicable)

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Additional Notes (include historical comparison, if appropriate)

Qualifier Summary		Qualifier Changes		
Sample Name	Parameter	Added	Removed	Retained

Additional Notes to DM

For DM Use Only	
Equis Y / N	Data Tech Init:
Facility ID:	Date entered into Equis:

W:\Business Units\AR\Subunit Admin\Practices-Processes\QCTRACK\MASTER SOP File\Final SOPs\2011 Final\Data Val Wrksht Rev 2010.xlsx

Attachment 2
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes	
Qualifier	**Definition**
--	Not analyzed/not available.
a	Estimated value, calculated using some or all values that are estimates.
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Relative percent difference is >40% (25% CLP pesticides) between primary and confirmation GC columns.
pp	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.
N	Sample Type: Normal
FD	Sample Type: Field Duplicate
AT	Sample chromatogram is noted to be atypical of a petroleum product.
DLND	Not detected, detection limit not determined.
DF	Did not flash
EMPC	Estimated maximum possible concentration.
NA – (Not applicable)	NA indicates that a fractional portion of the sample is not part of the analytical testing or field collection procedures.
ND	Not detected.
TIC	Tentatively identified compound
BQA	Barr-applied project specific qualifier: extraction and/or analyses conducted using an alternative method and/or procedure.
BQC	Barr-applied project specific qualifier: plant shut down.
BQD	Barr-applied project specific qualifier: equipment malfunction.
BQE	Barr-applied project specific qualifier: equipment adjustment.
BQM	Barr-applied project specific qualifier: manual measurement.
BQN	Barr-applied project specific qualifier: unable to be sampled or measured due to various reasons.
BQP	Barr-applied project specific qualifier: atypical chromatographic pattern.
BQQ	Barr-applied project specific qualifier: some aspect of QA/QC was not met.
BQR	Barr-applied project specific qualifier: location was re-sampled.
BQS	Barr-applied project specific qualifier: data is considered suspect.
BQT	Barr-applied project specific qualifier: summed value not displayed due to insufficient field length.
BQU	Barr-applied project specific qualifier: historical qualifier - definition unknown.
BQV	Barr-applied project specific qualifier: estimated value.
BQX	Barr-applied project specific qualifier: see notes for qualifier definition.
BQZ	Barr-applied project specific qualifier: data is considered unusable.

Attachment 3
Revisions

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections;
		IX	Changed to Section X
		Attachments	Added Attachment 3
		IX (new)	Added Table 9.
3.2	04/2011	Document Wide	Added missing analytical method references.
		Attachments	Updated Attachments to current forms.
3.3	04/2011	References	Update the reference to the current NFG Metals data validation document.

STANDARD OPERATING PROCEDURE

for Routine Level Polychlorinated Biphenyls (PCB), Aroclor™, Pesticide and Herbicide Data Validation

Revision 1.2

April 8, 2011

Approved By: Andrea Nord | Andrea Nord | 04/08/2011

Print | QA Manager(s) | Signature | Date

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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Standard Operating Procedures for Routine Level Polychlorinated Biphenyls (PCB), Aroclor™, Pesticide and Herbicide Data Validation

Purpose

This SOP is intended as a guidance document for the routine level validation of polychlorinated biphenyls (PCBs), Aroclor™, herbicide and pesticide data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine level PCB, Aroclor™, herbicide and pesticide data validation by the analytical methods including, but not limited to:

- GC/ECD for Pesticides (EPA Methods 608/8081B)
- GC/ECD or GC/ELCD for PCBs/Aroclor™ (EPA Method 8082A)
- GC/FPD or GC/NPD for Organophosphorous Compounds (EPA Method 8141B)
- GC/ECD for Herbicides (EPA Method 8151A)

Validation of Contract Laboratory Program (CLP) data should be performed in accordance with to the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008).*

Definitions

Aroclor™. A trademarked name for a mixture of polychlorinated biphenyls (PCBs) used in a variety of applications including additives in lubricants, heat transfer dielectric fluids, adhesives, etc.

Blank. An analytical sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

Contamination. A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

Field Blank. A blank used to provide information about contaminants that may be introduced during sample collection.

Herbicide. Any substance, or mixture of substances, intended to prevent the growth of or to destroy terrestrial or aquatic weeds. Weeds are any woody or non-woody undesirable vegetation.

GC/ECD. Gas Chromatography/Electron Capture Detector. Measures electron capturing compounds (usually halogenated) by creating an electrical field in which molecules exiting a GC column can be detected by the drop in current in the field. Identifies potential compounds based on retention time in a GC column, but the identification of the compound is not selective with one detector. Additional conformational analyses must be performed (either GC/MS or a separate GC/ECD with a different stationary phase on the column).

GC/FPD. Gas Chromatography/Flame Photometric Detector. The flame photometric detector (FPD) measures sulfur and phosphorus containing compounds, measuring chemiluminescent reactions from these compounds in a hydrogen / air flame.

GC/MS. Gas Chromatography/Mass Spectrometry (sometimes Mass Selective Detector (MSD)). An analytical technique used to measure the mass-to-charge ratio of ions. A MS detector can selectively identify a compound based on the compound's mass-to-charge ratio and generally does not require secondary confirmation.

GC/NPD. Gas Chromatography/Nitrogen-Phosphorus Detector. The nitrogen phosphorus detector (NPD) is a highly sensitive but specific detector similar to an FID. It gives a strong response to organic compounds containing nitrogen and/or phosphorus.

GC/PID. Gas Chromatography/Photoionization Detector. Uses ultraviolet light to ionize analyte exiting from a GC column. The resultant electrical charge produces a measurable current on a detector.

Initial Calibration. Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument.

Instrument Blank. A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS). The LCS is an internal laboratory Quality Control (QC) sample designed to assess the capability of the laboratory to perform the analytical method.

Matrix. The predominant material of which the sample to be analyzed is composed.

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS). Aliquot of the sample 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.

Matrix Spike Duplicate (MSD). A second aliquot of the same sample that is fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine precision of the method.

Method Blank. A reagent aqueous sample spiked with internal standards, and surrogate standards (or DMCs), that is carried throughout the entire analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

Narrative. The portion of the data package which includes laboratory, contact person, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

Pesticide. Any substance or mixture of substances intended for preventing, destroying, repelling, or lessening the damage of any pest.

Polychlorinated Biphenyls (PCBs). A group of toxic, persistent chemicals used in electrical transformers and capacitors for insulating purposes, and in gas pipeline systems as a lubricant. The sale and new use of PCBs were banned by law in 1979.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Retention Time (RT). The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

Semi-Volatile Organic Compounds (SVOC). An organic compound which has a boiling point higher than water and which may vaporize when exposed to temperatures above room temperature. Semi-volatile organic compounds include phenols and polynuclear aromatic hydrocarbons (PAH).

Surrogates (Surrogate Standard). Compounds added to every blank, sample, including Laboratory Control Sample (LCS/LCSD), Matrix Spike/Matrix Spike Duplicate (MS/MSD), and standards; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

TCLP. Toxicity Characteristic Leaching Procedure. A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

Equations

For % Recovery (%R or %Rec):

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

Where,

%R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample
In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008)*, and quality control recommendations outlined in:

- **EPA Methods 608** – *"Organochlorine Pesticides and PCBs"*
- **EPA Method 8081B** – "Organochlorine Pesticides by Gas Chromatography", February 2007.
- **EPA Method 8082A –** *"Polychlorinated Biphenyls (PCBs) by Gas Chromatography"*, February 2007.
- **EPA Method 8141B –** *"Organophosphorus Compounds by Gas Chromatography: Capillary Column Technique"*, February 2007.
- **EPA Method 8151A** – *"Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization"*, December 1996.
- **EPA Method 1311** – *"Toxicity Characteristic Leaching Procedure"* July 1992.

Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the data in accordance with this SOP, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

The recommended hold time and preservation acceptance criteria are in *Table 1*.

Table 1 – Recommended Hold Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Hold Time
PCBs/Aroclor™/ Pesticides (EPA 8081/8082)	aqueous	< 6° C	ice	7 days extraction/ addl. 40 days analysis
	sediment/ soil	< 6° C	ice	14 days extraction/ addl. 40 days analysis
PCBs/Pesticides (EPA 608)	aqueous	< 6° C	ice (if >72 hrs to extraction, preserve to pH 5-9 with NaOH and/or H_2SO_4)	72 hours extraction unpreserved/ 7 day extraction preserved/ addl. 40 days analysis
Herbicides (EPA 8151)	all matrices	< 6° C	ice	7 day extraction/ addl. 40 days analysis

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil) may not be necessarily be subjected to the same hold time recommendations.

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before receipt by the laboratory. While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group, or each batch digested (whichever is more frequent).
- The laboratory should analyze a method blanks at least once every 20 samples.
- Field blank collection and analysis frequency is project-specific.

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the method blank. In such cases, it may be appropriate to qualify the affected data with "**" (usable value, QA/QC criteria not met).

Table 2 – Guidelines for the Evaluation of Blank Contamination

Sample Result	Recommended Action
Non-detect	No action required
<5x blank concentration	Qualify with "**b**"
>5x blank concentration	Use professional judgment
Gross contamination	Qualify associated samples with "**"

Note: "**" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Surrogates Standards

Recovery limit guidelines are presented in the table below. Keep in mind that the laboratory may have different limits and compounds than those recommended. Recommended surrogate compounds are in *Tables 6 and 7* in ***Section IX*****.** Laboratory or QAPP assigned limits should be used where provided.

All samples (blanks, spiked samples, project samples, QC samples) should contain surrogates. If a sample does not contain surrogates, professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of surrogate spikes may not be applicable if dilution of the sample was required.

Table 3 – Guidelines for Surrogate Standard Recoveries			
Analysis	**Sample Concentration**	**Surrogate recovery**	**Recommended Action**
PCB/ Aroclor™/ Pesticides/ Herbicides	Non-detect	< 10% recovery	Qualify associated compounds with "**"
		< lower recovery limit	Qualify associated compounds with "*"
		Within or > acceptance criteria	No action
	Detections above reporting limits	< lower recovery limit	Qualify associated compounds with "*"
		Within acceptance criteria	No action
		> upper recovery limit	Qualify associated compounds with "*"

Note: "*"indicates that the reported value is estimated and QA/QA criteria were not met;
"**" indicates that the reported value is unusable and QA/QC criteria were not met.

In instances where surrogate recoveries are marginally outside acceptance criteria (within 10% of recovery limits), additional consideration should be given to historical results (where available) and general recovery trends in related samples of the same report before qualifying the sample.

IV. Laboratory Control Samples (LCS)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. Per method (laboratories may have developed other acceptable QC measures), LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix

Laboratory control samples contain a known amount of each target compound and the percent recoveries are evaluated based either on the laboratory's internally generated acceptance windows or default method criteria (as presented in *Table 4*). Herbicides do not currently have EPA-recommended recovery acceptance criteria. For the purposes of this SOP, use the recommended guidelines for LCS spike recoveries of PCBs/Aroclor™ to evaluate data (50-150% recoveries are acceptable).

Table 4 – Guidelines for Laboratory Control Sample Recoveries		
Analysis	**Acceptance Criteria**	**Recommended Action**
PCBs/Aroclor™	50-150% recovery (Aroclor™ 1016 and Aroclor™ 1260 are the recommended spike compounds)	if LCS > 150% & samples are non-detect, no action; if detections, qualify with "*"
		if LCS < 50%, qualify samples with "*"
		if LCS < 10%, qualify detects with "*" qualify non-detects with "**"
Pesticides	See Table 6 in **Section IX** for EPA-recommended compounds and recoveries	if LCS > upper limit & samples are non-detect, no action; if detections, qualify with "*"
		if LCS < lower limit, qualify samples with "*"
		if LCS < 10%, qualify detects with "*" qualify non-detects with "**"

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs of <20-30% for aqueous samples and <30-40% for soil or sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results are dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix may have on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix, or
- 1 per sample delivery group

However, the frequency may be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.). In some cases, MS/MSD analysis is not required.

If a matrix spike does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (greater than 4 times (>4x)), spike recovery criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery provided in the *Equations* section in the beginning of this SOP.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity.

In general, matrix spikes and matrix spike duplicates may be evaluated as follows:

Table 5 – Guidelines for Matrix Spike Evaluation	
Spike Result	**Recommended Action**
% recovery > upper acceptance limit	Non-detects, no qualification
	Detects, qualify with "*****"
between upper and lower limits	No qualification
% recovery is between 20% and lower acceptance limit	Non-detects, qualify with "*****"
	Detects, qualify with "*****"
% recovery is below 20%	Non-detects, use professional judgment; consider qualifying with "******"
	Detects, qualify with "*****"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS/MSD pairs using the equation for RPD evaluation provided in the *Equations* section in the beginning of this SOP. Generally, acceptance criteria for MS/MSD are <20-30% RPD for aqueous samples and <30-40% for soils or sediments, but professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

Note: Pesticides, herbicides, PCBs and Aroclors™ **require** additional ECD or GC/MS confirmation of tentatively identified compounds (TIC), using a separate column. This may occur at the same time as the initial analysis using a dual-column GC with an additional detector; or a second, separate analysis via EPA 8270 (See Barr SOP for SVOC Data Validation if positive detections occur). Herbicides are sufficiently identified by a single column if a GC/MS is used for analysis. If there is indication that confirmational analysis was not performed for the remaining parameters, professional judgment should be used to critically evaluate the usability of the data as reported.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Additional Tables

The following tables should be used for guidance purposes *only*. Priority should be given to laboratory limits and associated target compounds when provided by the laboratory. Use professional judgment before applying any of the data in the following tables to the validation process.

Table 6 – Recommended Guidance for LCS Compounds and Recovery for Pesticides

Compound	Recovery limits (%)
4,4'-DDE	50-150
Dieldrin	30-130
Endosulfan sulfate	50-120
Endrin	50-120
gamma-BHC	50-120
gamma-Chlordane	30-130
Heptachlor epoxide	50-150

Table 7 – Recommended Surrogates

Analysis	Recommend Surrogate
PCBs/Aroclor™/Pesticides	Tetrachloro-m-xylene (TCX)
	Decachlorobiphenyl (DCB)
Herbicides	2,4-Dichlorophenylacetic acid (DCAA)

X. Attachments

Attachment 1: Routine Level Quality Control Report
Attachment 2: Barr Qualifiers/Footnotes
Attachment 3: Revisions to SOP

Attachment 1
Routine Level Quality Control Report

Barr Engineering Company
Routine Level Quality Control Report

Project #		**Project Name**	
Laboratory		**COC(s)/Event**	
Lab Report #		**Matrix**	
Report Date		**Review Date**	
Holding Times Met	Yes No	**Reviewed By**	
If no, comments		**Exceptions**	
Temps on Receipt (°C)			

Method Blanks	**LCS/LCSD**
Field Blanks	**MS/MSD**
Trip Blanks (VOCs Only)	
Field Duplicates (if applicable)	**Surrogates (if applicable)**
	Lab Duplicates (if applicable)

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Additional Notes (include historical comparison, if appropriate)

Qualifier Summary		Qualifier Changes		
Sample Name	Parameter	Added	Removed	Retained

Additional Notes to DM

For DM Use Only	
Equis Y / N	Data Tech Init:
Facility ID:	Date entered into Equis:

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Attachment 2
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes	
Qualifier	**Definition**
--	Not analyzed/not available.
a	Estimated value, calculated using some or all values that are estimates.
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Relative percent difference is >40% (25% CLP pesticides) between primary and confirmation GC columns.
pp	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.
N	Sample Type: Normal
FD	Sample Type: Field Duplicate
AT	Sample chromatogram is noted to be atypical of a petroleum product.
DLND	Not detected, detection limit not determined.
DF	Did not flash
EMPC	Estimated maximum possible concentration.
NA – (Not applicable)	NA indicates that a fractional portion of the sample is not part of the analytical testing or field collection procedures.
ND	Not detected.
TIC	Tentatively identified compound
BQA	Barr-applied project specific qualifier: extraction and/or analyses conducted using an alternative method and/or procedure.
BQC	Barr-applied project specific qualifier: plant shut down.
BQD	Barr-applied project specific qualifier: equipment malfunction.
BQE	Barr-applied project specific qualifier: equipment adjustment.
BQM	Barr-applied project specific qualifier: manual measurement.
BQN	Barr-applied project specific qualifier: unable to be sampled or measured due to various reasons.
BQP	Barr-applied project specific qualifier: atypical chromatographic pattern.
BQQ	Barr-applied project specific qualifier: some aspect of QA/QC was not met.
BQR	Barr-applied project specific qualifier: location was re-sampled.
BQS	Barr-applied project specific qualifier: data is considered suspect.
BQT	Barr-applied project specific qualifier: summed value not displayed due to insufficient field length.
BQU	Barr-applied project specific qualifier: historical qualifier - definition unknown.
BQV	Barr-applied project specific qualifier: estimated value.
BQX	Barr-applied project specific qualifier: see notes for qualifier definition.
BQZ	Barr-applied project specific qualifier: data is considered unusable.

UMP013252

Attachment 3
Revisions

Revision Number	Date of Revision	Section	Revision Made
1.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		Attachments	Added Attachment 3
1.2	04/2011	Attachments	Updated Attachment 1 and 2 to current forms.

UMP013253

STANDARD OPERATING PROCEDURE

for Routine Level Semivolatile Organic Compounds (SVOC) and Polycyclic Aromatic Hydrocarbons (PAH) Data Validation

Revision 3.2

April 8, 2011

Approved By:	Andrea Nord		04/08/11
	Print	QA Manager(s) Signature	Date

Barr Engineering Company
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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	_____	Date:	_____
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Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for Routine Level Semivolatile Organic Compounds (SVOC) and Polycyclic Aromatic Hydrocarbons (PAH) Data Validation

Purpose

This SOP is intended as a guidance document for the routine level validation of semivolatile organic compounds (SVOC) and Polycyclic Aromatic Hydrocarbons (PAH) data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine SVOC (including PAHs, PCPs) and diesel range organics (DRO) data validation by the analytical methods including, but not limited to:

- GC/MS for SVOCs (EPA Method 8270C/8270D and 8270C/8270D SIM)
- GC/FID for PAHs (EPA Method 8100)
- HPLC for PAHs (EPA Method 8310)
- Wisconsin (WI) DRO (SW-141)
- GC/FID for DRO (EPA Method 8015C)
- TCLP/SVOC (EPA Methods 1311/8270C/8270D)

Validation of Contract Laboratory Program (CLP) data should be performed in accordance with to the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008).*

Definitions

Blank. An analytical sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

Contamination. A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

DRO. Diesel Range Organics. Organic range corresponding to a hydrocarbon range of C_{10} - C_{28} and a boiling point range between approximately 170°C and 430°C. Other organic compounds, including chlorinated hydrocarbons, phenols, phthalate esters, polynuclear aromatic hydrocarbons, kerosene, fuel oils and heavier oils, are measurable.

Deuterated Monitoring Compounds (DMCs). Compounds added to every semivolatile calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge and trap procedures, and the performance of the Gas Chromatograph/Mass Spectrometer (GC/MS) systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

Field Blank. A blank used to provide information about contaminants that may be introduced during sample collection.

GC/FID. Gas Chromatography/Flame Ionization Detector. As components elute from the GC's column they pass through the flame and are burned, producing ions. The ions propagate an electric current, which is the signal output of the detector. The greater the concentration of the component, the more ions are produced, and the greater the current.

GC/MS. Gas Chromatography/Mass Spectrometry (sometimes Mass Selective Detector (MSD)). An analytical technique used to measure the mass-to-charge ratio of ions. A MS detector can selectively identify a compound based on the compound's mass-to-charge ratio and generally does not require secondary confirmation.

HCl. Hydrochloric acid.

HPLC. High Performance Liquid Chromatography. A chromatographic technique for separating and analyzing mixtures of substances, using a packed column with small particles coated with the stationary phase and where the mobile phase is pumped through the column with a high pressure pump. For the purposes of these analyses, a fluorescence or UV (ultraviolet) detector is used to identify the chromatographic separations.

Initial Calibration. Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument.

Internal Standards. Compounds added to every volatile calibration standard, blank, sample, or sample extract, including the Laboratory Control Sample (LCS), at a known concentration, prior to analysis. Internal standards are used to monitor instrument performance and quantitation of target compounds.

Instrument Blank. A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS). The LCS is an internal laboratory Quality Control (QC) sample designed to assess the capability of the laboratory to perform the analytical method.

Matrix. The predominant material of which the sample to be analyzed is composed.

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS). Aliquot of the sample 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.

MeOH. Methanol.

Method Blank. A reagent aqueous sample spiked with internal standards, and surrogate standards (or DMCs), that is carried throughout the entire analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

Narrative. Portion of the data package which includes laboratory, contact, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP

Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Retention Time (RT). The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

SIM. Selected Ion Monitoring. Method of GC/MS scanning that focuses on only a few ions for detection. Using this technique increases the sensitivity of a mass spectrometry detector as much as 500-fold.

Semivolatile Organic Compounds (SVOC). An organic compound which has a boiling point higher than water and which may vaporize when exposed to temperatures above room temperature. Semivolatile organic compounds include phenols and polynuclear aromatic hydrocarbons (PAH)

Surrogates (Surrogate Standard). Compounds added to every blank, sample, including Laboratory Control Sample (LCS/LCSD), Matrix Spike/Matrix Spike Duplicate (MS/MSD), and standards; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

TCLP. Toxicity Characteristic Leaching Procedure. A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

Equations

For % Recovery (%R or %Rec):

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

Where,

%R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample
In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (January 2005)*, and quality control recommendations outlined in:

- **SW-141** – *"Wisconsin DRO"*, September 1995;
- **EPA Method 1311** – *"Toxicity Characteristic Leaching Procedure"*, July 1992;
- **EPA Method 8015B** – *"Nonhalongenated Organics Using GC/FID"*, February 2007;
- **EPA Method 8100** – *"Polynuclear Aromatic Hydrocarbons"*, September 1986;
- **EPA Method 8270** – *"Semivolatile Organic Compounds by GC/MS"*, February 2007; and
- **EPA Method 8310** – *"Polynuclear Aromatic Hydrocarbons*, September 1986.

Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the data in accordance with this SOP, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

The recommended hold time and preservation acceptance criteria are in *Table 1*.

Table 1 – Recommended Hold Times and Preservation

Compound	Matrix	Temperature	Preservative	Maximum Hold Time
SVOCs /PAHs	aqueous	< 6° C	ice	7 days extraction/ addl. 40 days analysis
	sediment/soil	< 6° C	ice	14 days extraction/ addl. 40 days analysis
WI DRO	aqueous	< 6° C	HCl <2 pH	7 days extraction/ addl. 40 days analysis
	sediment/soil	< 6° C	ice	14 days extraction/ addl. 40 days analysis
TCLP	all matrices	< 6° C	ice	14 days TCLP extraction / 7 days prep. extraction / addl. 40 days analysis

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil) may not be necessarily be subjected to the same hold time recommendations.

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before receipt by the laboratory. While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

A separate sample (without preservative) should be collected for each soil/sediment sample to be analyzed for DRO for the determination of moisture content. Samples without moisture content determination should be reported as wet weight.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group, or each batch digested (whichever is more frequent).
- The laboratory should analyze a method blanks at least once every 20 samples.
- At least one method blank should be analyzed with each concentration level (e.g. low or medium).
- Field blank collection and analysis frequency is project-specific.

Table 2 – Guidance for the Evaluation of Blank Contamination

Analyses	Positive Detection in Blank	Sample Result	Recommended Action
SVOCs/ DRO/ PAHs	Common laboratory contaminants (e.g. common phthalate esters)	Non-detect	No action required
		<10x blank concentration	Qualify with "**b**"
		>10x blank concentration	Use professional judgment
	All other target parameters	Non-detect	No action required
		<5x blank concentration	Qualify with "**b**"
		>5x blank concentration	Use professional judgment
Any analysis	Gross contamination (e.g. saturated peaks of target analytes in blanks)	All detections	Qualify with "******"
SVOC 8270 SIM	All target parameters	Non-detect	No action required
		< 20x blank concentration	Qualify with "**b**"
		> 20x blank concentration	Use professional judgment

Note: "******" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Deuterated Monitoring Compounds (DMC), (Surrogates)

Deuterated Monitoring Compounds (DMC) are also frequently known as System Monitoring Compounds (SMC) or Surrogates. Keep in mind that the laboratory may have different limits and compounds than those recommended. *Table 7* in **Section IX** presents the *recommended* compounds and recovery limits (per Guidelines) for DMCs used by laboratories (SVOCs only). Associated methods may provide additional guidance. Laboratory or QAPP assigned limits should be used where provided.

All samples (blanks, spiked samples, project samples, QC samples) should contain DMCs or surrogates. If a sample does not contain DMCs or surrogates, professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of DMCs or surrogates may not be applicable if dilution of the sample was required.

Table 3 – Guidance for the Recovery of Deuterated Monitoring Compounds

Analysis	Sample Concentration	DMC/surrogate recovery	Recommended Action
SVOC/ SVOC SIM	Sample is non-detect or has concentrations of associated target compounds less than reporting limit (RL)	< 10% recovery	Qualify associated target compounds with "**"
		< lower recovery limit	Qualify with associated target compounds with "*"
		within or > acceptance limits	No action
	Sample has detectable concentrations of associated target compounds above reporting limit (RL)	< lower recovery limit	Qualify with associated target compounds with "*"
		within acceptance limits	No action
		> upper recovery limits	Qualify with associated target compounds with "*"

Note: "*"indicates that the reported value is estimated and QA/QA criteria were not met;
"**" indicates that the reported value is unusable and QA/QC criteria were not met.

In instances where the DMC or surrogate recoveries are marginally outside acceptance criteria (within 10% of recovery limits), additional consideration should be given to historical results (where available) and general recovery trends in related samples of the same report before qualifying the sample.

In all cases, only the target compound(s) associated with the DMC that fails to meet acceptance criteria may require qualification.

Table 8 in **Section IX** presents the recommended DMCs with their associated target compounds for SVOCs *only*. Bear in mind that laboratory methods may deviate from the recommended guidance and surrogate failures should be evaluated based on laboratory SOPs, especially in regard to which DMCs (surrogates) are associated with which target compounds.

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Not all DMC/surrogates are utilized in all SVOC analyses. If alternate or fewer surrogates are used, the following guidelines are recommended:

Table 4 – Guidance for the Recovery of Deuterated Monitoring Compounds (If Fewer DMCs than National Function Guidelines Recommend Are Used)	
DMC/Surrogate recoveries	**Recommended Action**
One DMC < 10% recovery	All detects of same fraction (Acid or Base/Neutral), qualify with "*****"
	All non-detects, qualify with "******"
One DMC (or two DMC of different fractions), between 10% recovery and lower recovery limit	No action required
Two or more DMC of the same acid or base/neutral fraction between 10% recovery and lower recovery limit	All detects of same fraction (Acid or Base/Neutral), qualify with "*****"
	All non-detects, qualify with "******"
Two or more DMC of the same acid or base/neutral fraction above the upper recovery limit	All detects of same fraction (Acid or Base/Neutral), qualify with "*****"
	All non-detects, qualify with "******"
One DMC above the upper recovery limit	No action

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met.

PAH analysis by Method 8100 (GC/FID) requires only that one surrogate be used and does not specify which surrogate is to be used. 2-fluorobiphenyl and 1-fluoronaphthalene are the recommended surrogate compounds, but the choice is open to the laboratory performing the analysis, provided adequate chromatographic separations can be demonstrated. PAH analysis by Method 8310 (HPLC) has similar recommendations and requirements. The recommended (but not required) surrogate is decafluorobiphenyl for this method.

For DRO analysis, surrogates are not required by the method. If used, the method requires that the surrogates must not elute within the diesel range organics (DRO) window. Surrogates recommended by the method are nonane (C_9) and nonacosane (C_{29}). Use professional judgment and the above table as guidance for evaluating surrogates in DRO samples.

IV. Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. Per method (laboratories may have developed other acceptable QC measures), LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix
- One LCS every 20 samples of the same matrix (WI DRO methods require an additional LCSD analysis every 20 samples)

Laboratory control samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 10) and the percent recoveries are evaluated based either on the laboratory's internally generated acceptance windows or default method criteria (as presented in the following table).

Table 5 – Guidelines for Evaluating Laboratory Control Sample Recoveries

Analysis	Matrix	Acceptance Criteria	Recommended Action
SVOC and associated analyses	aqueous/ sediment/ soil	no guidance from EPA, use laboratory acceptance criteria or professional judgment (generally, 75-125% recovery is acceptable)	if LCS > upper limit & samples are non-detect, no action; if detections, qualify with "*"
			if LCS < lower limit, qualify samples with "**"
			if LCS << lower limit, qualify detects with "**" qualify non-detects with "**"
DRO	aqueous	75-115% recovery <20% RPD	if LCS > 115% & samples are non-detect, no action; if detections, qualify with "*"
			if LCS < 75%, qualify samples with "**"
	soil/sediment	70-120% recovery <20%RPD	if LCS > 120% & samples are non-detect, no action; if detections, qualify with "**"
			if LCS < 70%, qualify samples with "**"

Note: "**"indicates that the reported value is estimated and QA/QA criteria were not met;
"**" indicates that the reported value is unusable and QA/QC criteria were not met.

RPDs are calculated for LCS/LCSD pairs using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are also used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs <20-30% for aqueous samples and <30-40% for soil and sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample is detected and much greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results based upon field duplicate RPDs.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix may have on the sample preparation procedures and analytical results. Matrix spike and matrix spike duplicate samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 10).

Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to WI DRO)
- 1 per preparation batch per matrix, or
- 1 per sample delivery group

However, the frequency may be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.). In some cases, MS/MSD analysis is not required.

If a matrix spike does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (greater than four times (>4x)), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery provided in the *Equations* section in the beginning of this SOP.

If laboratory acceptance criteria are provided for the percent recoveries of matrix spikes, those criteria should be used for the evaluation of the data. If acceptance criteria are not provided by the laboratory, the percent recoveries recommended by the Guidelines are presented in *Table 9* in **Section IX** can be used for guidance.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be limited by the sampling precision and inherent sample homogeneity.

In general, matrix spikes and matrix spike duplicates may be evaluated as follows:

Table 6 – Guidance for Matrix Spike Evaluation

Spike Result	Recommended Action
% recovery > upper acceptance limit	Non-detects, no qualification
	Detects, qualify with "*****"
% recovery meets acceptance limits	No qualification
% recovery is between 20% and lower acceptance limit	Non-detects, qualify with "*****"
	Detects, qualify with "*****"
% recovery is below 20%	Non-detects, use professional judgment; consider qualifying with "******"
	Detects, qualify with "*****"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS/MSD pairs using the equation for RPD evaluation provided in the *Equations* section in the beginning of this SOP. Generally, acceptance criteria for MS/MSD is <20-30% RPD for aqueous samples and <30-40% for soils/sediments, but professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

IX. Additional Tables

The following tables should be used for guidance purposes *only* for samples being analyzed for SVOCs. Priority should be given to laboratory limits and associated target compounds when provided by the laboratory. Use professional judgment before applying any of the data in the following tables to the validation process.

Table 7 – Recommended Guidance for DMC/Surrogate Recovery

DMC	Recovery limits (%) for aqueous samples	Recovery limits (%) for soil/sediment samples
2,4-Dichlorophenol-d_3	37-105	23-104
2-Chlorophenol-d_4	41-106	13-101
2-Nitrophenol-d_4	40-108	16-104
4-6-Dinitro-2-methylphenol-d_2	22-104	1-121
4-Chloroaniline-d_4	1-145	1-145
4-Methylphenol-d_8	25-111	8-100
4-Nitrophenol-d_4	33-116	16-166
Acenaphthylene-d_8	41-107	20-97
Anthracene-d_{10}	44-110	22-98
Benzo(a)pyrene-d_{12}	32-121	43-111

Table 7 – Recommended Guidance for DMC/Surrogate Recovery		
DMC	**Recovery limits (%) for aqueous samples**	**Recovery limits (%) for soil/sediment samples**
Bis-(2-chloroethyl) ether-d_8	40-105	12-98
Dimethylphthalate-d_6	47-114	43-111
Fluorene-d_{10}	42-111	40-108
Nitrobenzene-d_5	43-108	16-103
Phenol-d_5	39-106	17-103
Pyrene-d_{10}	52-119	51-120
Fluoranthene-d_{10} **(SIM)**	50-150	50-150
2-Methylnaphthalene-d_{10} **(SIM)**	50-150	50-150

Table 8 – DMC and Associated Target Compounds		
DMC (alphabetical)	**Associated Target Compounds**	
2,4-Dichlorophenol-d_3	2,3-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol	2,3,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol
2-Chlorophenol-d_4	2-Chlorophenol	
2-Nitrophenol-d_4	Isophorone	2-Nitrophenol
4-6-Dinitro-2-methylphenol-d_2	4,6-Ditritro-2-methylphenol	
4-Chloroaniline-d_4	4-Chloroaniline Hexachlorocyclopentadiene	3,3'-Dichlorobenzidine
4-Methylphenol-d_8	2-Methylphenol 4-Methylphenol	2,4-Dimethylphenol
4-Nitrophenol-d_4	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol	4-Nitrophenol 4-Nitroaniline
Acenaphthylene-d_8	Naphthalene 2-Methylnaphthalene 2-Chloronapthalene	Acenaphthylene Acenaphthene
Anthracene-d_{10}	Hexachlorobenzene Atrazine	Phenanthrene Anthracene
Benzo(a)pyrene-d_{12}	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
Bis-(2-chloroethyl) ether-d_8	Bis-(2-chloroethyl) ether 2,2'-oxybis(1-chloropropane)	bis(2-Choloethoxy) methane
Dimethylphthalate-d_6	Caprolactum 1,1'-Biphenyl Dimethylphthalate Diethylphthalate	Di-n-butylphthalate Butylbenzylphthalate bis(2-ethylhexyl)phthalate Di-n-octylphthalate
Fluorene-d_{10}	Dibenzofuran Fluorene 4-Chlorophenyl-phenylether	4-Bromophenyl-phenylether Carbazole
Nitrobenzene-d_5	Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene	2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosdiphenylamine
Phenol-d_5	Benzaldehyde	Phenol

Table 8 – DMC and Associated Target Compounds		
DMC (alphabetical)	**Associated Target Compounds**	
Pyrene-d_{10}	Fluoranthrene Pyrene	Benzo(a)anthracene Chrysene
SIM DMC and Associated Target Compounds		
Fluoranthene-d_{10}	Fluoranthene Pyrene Benzo(a)antheacene Chrysene Benzo(b)fluoranthene	Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
2-Methylnaphthalene-d_{10}	Naphthalene 2-Methylnaphthalene Ancenaphthylene Acenaphthene	Fluorene Pentachlorophenol Phenanthrene Anthracene

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Table 9 – Recommended MS/MSD Recoveries and RPD				
Compound	**%Recovery for Water Samples**	**RPD for Water Samples**	**%Recovery for Soil/Sediment Samples**	**RPD for Soil/Sediment Samples**
2,4-Dinitrotoluene	24 – 96	0 – 38	28 – 89	0 – 47
2-Cholorphenol	27 – 123	0 – 40	25 – 102	0 - 50
4-Chloro-3-methylphenol	23 - 97	0 – 42	26 – 103	0 – 33
4-Nitrophenol	10 – 80	0 – 50	11 – 114	0 – 50
Acenaphthene	46 – 118	0 – 31	31 – 137	0 – 19
N-Nitroso-di-n-propylamine	41 – 116	0 – 38	41 – 126	0 – 38
Pentachlorophenol	9 – 103	0 – 50	17 – 109	0 – 47
Phenol	12 - 110	0 - 42	26 - 90	0 - 35
Pyrene	26 – 127	0 - 31	35 – 142	0 – 36

Table 10 – Number of Target Compounds Required by NELAP and MN Rules for LCS/LCSD and MS/MSD samples	
Number of Target Parameters	**Required Number of Spiked Compounds**
1-10 analytes	Spike *all* compounds
11-20 analytes	*At least* 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike *at least* 16 compounds

X. Attachments

Attachment 1: Routine Level Quality Control Report
Attachment 2: Barr Qualifiers/Footnotes
Attachment 3: Revisions to SOP

Attachment 1
Routine Level Quality Control Report

Barr Engineering Company
Routine Level Quality Control Report

Project #		**Project Name**	
Laboratory		**COC(s)/Event**	
Lab Report #		**Matrix**	
Report Date		**Review Date**	
Holding Times Met	Yes No	**Reviewed By**	
If no, comments		**Exceptions**	
Temps on Receipt (°C)			

Method Blanks	**LCS/LCSD**
Field Blanks	**MS/MSD**
Trip Blanks (VOCs Only)	
Field Duplicates (if applicable)	**Surrogates (if applicable)**
	Lab Duplicates (if applicable)

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Attachment 1 (continued)
Routine Level Quality Control Report

Additional Notes (include historical comparison, if appropriate)

Qualifier Summary		Qualifier Changes		
Sample Name	Parameter	Added	Removed	Retained

Additional Notes to DM

For DM Use Only	
Equis Y / N	Data Tech Init:
Facility ID:	Date entered into Equis:

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Attachment 2
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes	
Qualifier	**Definition**
--	Not analyzed/not available.
a	Estimated value, calculated using some or all values that are estimates.
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Relative percent difference is >40% (25% CLP pesticides) between primary and confirmation GC columns.
pp	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.
N	Sample Type: Normal
FD	Sample Type: Field Duplicate
AT	Sample chromatogram is noted to be atypical of a petroleum product.
DLND	Not detected, detection limit not determined.
DF	Did not flash
EMPC	Estimated maximum possible concentration.
NA – (Not applicable)	NA indicates that a fractional portion of the sample is not part of the analytical testing or field collection procedures.
ND	Not detected.
TIC	Tentatively identified compound
BQA	Barr-applied project specific qualifier: extraction and/or analyses conducted using an alternative method and/or procedure.
BQC	Barr-applied project specific qualifier: plant shut down.
BQD	Barr-applied project specific qualifier: equipment malfunction.
BQE	Barr-applied project specific qualifier: equipment adjustment.
BQM	Barr-applied project specific qualifier: manual measurement.
BQN	Barr-applied project specific qualifier: unable to be sampled or measured due to various reasons.
BQP	Barr-applied project specific qualifier: atypical chromatographic pattern.
BQQ	Barr-applied project specific qualifier: some aspect of QA/QC was not met.
BQR	Barr-applied project specific qualifier: location was re-sampled.
BQS	Barr-applied project specific qualifier: data is considered suspect.
BQT	Barr-applied project specific qualifier: summed value not displayed due to insufficient field length.
BQU	Barr-applied project specific qualifier: historical qualifier - definition unknown.
BQV	Barr-applied project specific qualifier: estimated value.
BQX	Barr-applied project specific qualifier: see notes for qualifier definition.
BQZ	Barr-applied project specific qualifier: data is considered unusable.

Attachment 3
Revisions

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 10
		Attachments	Added Attachment 3
3.2	04/2011	Document Wide	Added analytical methods to applicability section.
		Attachments	Updated Attachment 1 and 2 to include current forms.

STANDARD OPERATING PROCEDURE

Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO) and Total Petroleum Hydrocarbons (TPH) Data Validation

Revision 3.2

April 8, 2011

Approved By:	Andrea Nord	Andrea Nord	4/8/11
	Print QA Manager(s)	Signature	Date

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Annual Review of the SOP has been performed and the SOP still reflects current practice.

Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____
Initials:	_____	Date:	_____

Standard Operating Procedures for Routine Level Volatile Organic Compounds (VOC) Gasoline Range Organics (GRO) and Total Petroleum Hydrocarbons (TPH) Data Validation

Purpose

This SOP is intended as a guidance SOP for the routine level validation of volatile organic compounds (VOC) data provided by laboratories to be used in Barr Engineering Company (Barr) projects or by Barr clients.

Applicability

This SOP applies to routine VOC (including BTEX and TPH) and gasoline range organics (GRO) data validation by the analytical methods including, but not limited to:

- GC/MS and GC/MS SIM (EPA Method 8260B)
- GC/PID or GC/ECD (EPA Method 8021B)
- Wisconsin (WI) GRO (EPA Method 8015C)
- TCLP VOCs (EPA Methods 1311/8260B)

Validation of Contract Laboratory Program (CLP) data should be performed in accordance with to the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008)*.

Definitions

Blank. An analytical sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

BTEX. An acronym that stands for Benzene, Toluene, Ethylbenzene, and Xylenes.

Contamination. A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

Deuterated Monitoring Compounds (DMCs). Compounds added to every volatile calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge and trap procedures, and the performance of the Gas Chromatograph/Mass Spectrometer (GC/MS) systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

Field Blank. A blank used to provide information about contaminants that may be introduced during sample collection.

GC/ECD. Gas Chromatography/Electron Capture Detector. Measures electron capturing compounds (usually halogenated) by creating an electrical field in which molecules exiting a GC column can be detected by the drop in current in the field. Identifies potential compounds based on retention time in a GC column, but the identification of the compound is not selective with one detector. Additional conformational analyses must be performed (either GC/MS or a separate GC/ECD with a different stationary phase on the column).

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GC/FID. Gas Chromatography/Flame Ionization Detector. As components elute from the GC's column they pass through the flame and are burned, producing ions. The ions propagate an electric current, which is the signal output of the detector. The greater the concentration of the component, the more ions are produced, and the greater the current.

GC/MS. Gas Chromatography/Mass Spectrometry (sometimes Mass Selective Detector (MSD)). An analytical technique used to measure the mass-to-charge ratio of ions. A MS detector can selectively identify a compound based on the compound's mass-to-charge ratio and generally does not require secondary confirmation.

GC/PID. Gas Chromatography/Photoionization Detector. Uses ultraviolet light to ionize analyte exiting from a GC column. The resultant electrical charge produces a measurable current on a detector.

GRO. Gasoline Range Organics. Light-range petroleum products, including gasoline, with petroleum hydrocarbon compounds corresponding to an alkane range from the beginning of n-hexane (C_6) to beginning of n-decane (C_{10}) and with a boiling point range between approximately 60 - 170 degrees Centigrade.

HCl. Hydrochloric acid.

Initial Calibration. Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument.

Internal Standards. Compounds added to every volatile calibration standard, blank, sample, or sample extract, including the Laboratory Control Sample (LCS), at a known concentration, prior to analysis. Internal standards are used to monitor instrument performance and quantitation of target compounds.

Instrument Blank. A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS). The LCS is an internal laboratory Quality Control (QC) sample designed to assess the capability of the laboratory to perform the analytical method.

Matrix. The predominant material of which the sample to be analyzed is composed.

Matrix Effect. In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS). Aliquot of the sample 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.

Matrix Spike Duplicate (MSD). A second aliquot of the same as the MS sample that is fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine precision of the method.

MeOH. Methanol.

Method Blank. A reagent aqueous sample spiked with internal standards, and surrogate standards (or DMCs), that is carried throughout the entire analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

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MTBE. Methyl-Tertiary-Butyl-Ether. A gasoline additive, intended to reduce air pollution, that has sometimes contaminated groundwater through releases from leaking underground fuel storage tanks.

Narrative. The portion of the data package which includes laboratory, contact person, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NELAP. National Environmental Laboratory Accreditation Program. NELAP adopts standards (e.g. rules) that are based on the International Organization for Standardization (ISO) and are developed through a consensus process. Oversight is provided by the NELAP Board which consists of one representative and one alternate from each of the recognized accreditation bodies.

$Na_2S_2O_4$. Sodium Hydrosulfite. A chemical used to preserve aqueous VOC samples if residual chlorine is present.

PE Sample. Performance Evaluation Sample. A reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified.

PT Sample. Proficiency Testing Sample. A sample, the composition of which is unknown to the laboratory and is provided to test whether a laboratory can produce analytical results within the specified acceptance criteria. Required for accreditation by MN Rules and NELAP.

QAPP. Quality Assurance Project Plan. A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

QA/QC. Quality Assurance/Quality Control.

Retention Time (RT). The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SAP. Sampling and Analysis Plan. The purpose of the SAP is to ensure that sampling data collection activities are comparable to and compatible with previous data collection activities performed at the site and to document the details of all field activities and laboratory analyses prior to the work being conducted.

Sample Delivery Group (SDG). Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

SIM. Selected Ion Monitoring. Method of GC/MS scanning that focuses on only a few ions for detection. Using this technique increases the sensitivity of a mass spectrometry detector as much as 500-fold.

Surrogates (Surrogate Standard). Compounds added to every blank, sample, including Laboratory Control Sample (LCS/LCSD), Matrix Spike/Matrix Spike Duplicate (MS/MSD), and standards; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

TCLP. Toxicity Characteristic Leaching Procedure. A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

TPH. Total Petroleum Hydrocarbons. A measure of the concentration or mass of petroleum hydrocarbon constituents present in a given amount of soil or water. The term "total" is a misnomer--few, if any, of the procedures for quantifying hydrocarbons are capable of measuring all fractions of petroleum hydrocarbons present in the sample. Volatile hydrocarbons are usually lost in the process and not quantified, and some non-petroleum hydrocarbons are sometimes included in the analysis.

Trip Blank. A blank used to provide information about contaminants that may be introduced during sample transport.

Volatile Organic Compounds (VOC). Organic chemical compounds that have high enough vapor pressures under normal conditions to significantly vaporize and enter the atmosphere.

Equations

For % Recovery (%R or %Rec):

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

Where,

%R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample
In the case of LCS and other laboratory-prepared samples, the sample result (SR) is zero.

For RPD:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = relative percent difference
S = original sample result
D = duplicate sample result

References

This SOP is based on the recommendations of *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (January 2005),* and quality control recommendations outlined in:

- **Minnesota Rules 4740.2020 – 4740.2120** – *State of Minnesota Rules,* October 2006,
- **SW-140 –** *Wisconsin GRO (WI GRO),* September 1995,
- **EPA Method 8260B** – *"Volatile Organic Compounds by GC/MS",* December 1996,
- **EPA Method 8015C** – *"Nonhalogenated Organics Using GC/FID",* February 2007,
- **EPA Method 8021B** – *"Aromatic and Halogenated Volatiles by GC using PID and/or ECD",* December 1996, and
- **EPA Method 1311** – *"Toxicity Characteristic Leaching Procedure"* July 1992.

Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.).

The QA/QC officer is responsible for validating the data in accordance with this SOP, in addition to using professional judgment where necessary or appropriate.

Procedure

I. Hold Time and Preservation Evaluation

The purpose of hold time evaluation is to ascertain the validity of the analytical results based on the sample condition, proper preservation and the time elapsed between the date of sample collection and date of analysis.

The recommended hold time and preservation acceptance criteria are in *Table 1*.

Table 1 – Recommended Hold Times and Preservation

Compound	Matrix	Temperature	Preservative	Maximum Hold Time
VOC (including BTEX and MTBE)	aqueous	< 6° C	HCl <2 pH	14 days
	aqueous	< 6° C	unpreserved	7 days
	sediment/soil	< 6° C	25 mL MeOH in jar pre-weighed by laboratory	14 days
WI GRO	aqueous	< 6° C	HCl <2 pH	14 days
	sediment/soil	< 6° C	25 mL MeOH in jar pre-weighed by laboratory	14 days
TPH	aqueous	< 6° C	HCl or H_2SO_4 <2 pH	7 day extraction/ addl. 40 days analysis
	sediment/soil	< 6° C	not required	14 days extraction/ addl.40 days analysis
TCLP	all matrices	< 6° C	no preservative	14 days extraction/ addl. 14 days analysis

If samples do not meet hold time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil,) may not be necessarily be subjected to the same hold time recommendations.

It is noted that the temperature of sample upon laboratory receipt may exceed the recommended temperature if the sample was collected the same day or shortly before receipt by the laboratory. While Minnesota requires that samples are stored and received on ice this may have little impact on the temperature of samples collected within five (5) hours before submission to the laboratory. Other states may have additional or different requirements. Use professional judgment when evaluating the application of qualifiers in these cases.

Professional judgment should be applied (considering matrix and magnitude of exceedence, etc.) when evaluating the application of qualifiers when criteria are not met.

Special considerations for Holding Times of VOC samples

Aqueous samples should be received without headspace and soil samples typically require 25 grams of soil to 25 mL methanol (other volumes may be used, but the ratio of grams of soil to mL of methanol should be 1:1). Some headspace may be self-evolving in aqueous samples at sites with characteristically high pH levels and this should be considered before qualification of the results.

Aqueous samples with residual chlorine present should additionally have a 10% $Na_2S_2O_4$ solution added in addition to the HCl preservative to dechlorinate the sample. Samples with residual chlorine might warrant qualification with an "**h**" if not preserved correctly.

A separate sample (without preservative) should be collected for each soil sample to be analyzed for VOC, BTEX or WI GRO for the determination of moisture content. Samples without moisture content determination should be reported as wet weight.

II. Blanks

Blank evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group, or each batch digested (whichever is more frequent). The laboratory should analyze a method blanks at least once every 12 hours.

- Field blank collection and analysis frequency is project-specific.

- Trip blanks should be placed in each transport cooler containing VOC sample containers prior to shipment into the field and remain with the associated VOC samples submitted to the laboratory for VOC analysis; including sample storage through analysis.

Table 2 – Guidance for the Evaluation of Blank Contamination		
Positive Detection in Blank	**Sample Result**	**Recommended Action**
Common laboratory contaminants (e.g. methylene chloride, acetone, toluene, 2-butanone (MEK), carbon disulfide, and cyclohexane)	Non-detect	No action required
	<10x blank concentration	Qualify with "**b**"
	>10x blank concentration	Use professional judgment
All other target parameters	Non-detect	No action required
	<5x blank concentration	Qualify with "**b**"
	>5x blank concentration	Use professional judgment
Gross contamination (e.g. saturated peaks of target analytes in blanks)	All detections	Qualify with "******"

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met;
"b" indicates the reported value may be a potential false positive based on blank data validation procedures.

III. Deuterated Monitoring Compounds (DMC aka Surrogates)

Deuterated Monitoring Compounds (DMC) are also frequently known as System Monitoring Compounds (SMC) or Surrogates. Keep in mind that the laboratory may have different limits and compounds than those recommended. *Table 6* in **Section IX** presents the *recommended* compounds and recovery limits (per Guidelines) for DMCs used by laboratories (VOCs only). Laboratory-assigned limits should be used where provided.

All samples (blanks, spiked samples, project samples, QC samples) should contain DMCs or surrogates. If a sample does not contain DMCs or surrogates, professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of DMCs or surrogates may not be applicable if dilution of the sample was required.

Table 3 – Guidance for the Recovery of Deuterated Monitoring Compounds		
Sample Concentration	**DMC or surrogate recovery**	**Recommended Action**
Sample is non-detect or has concentrations of associated target compounds less than reporting limit (RL)	< 10% recovery	Qualify associated target compounds with "******"
	< lower recovery limit	Qualify with associated target compounds with "*****"
	within acceptance limits	No action
	> upper recovery limits	No action
Sample has detectable concentrations of associated target compounds above reporting limit (RL)	< 10% recovery	Qualify with associated target compounds with "*****"
	< lower recovery limit	Qualify with associated target compounds with "*****"
	within acceptance limits	No action
	> upper recovery limits	Qualify with associated target compounds with "*****"

Note: "*****"indicates that the reported value is estimated and QA/QA criteria were not met;
"******" indicates that the reported value is unusable and QA/QC criteria were not met;

In instances where the DMC or surrogate recoveries are marginally outside acceptance criteria (within 10% of recovery limits), additional consideration should be given to historical results (where available) and general recovery trends in related samples of the same report before qualifying the sample.

In all cases, only the target compound(s) associated with the DMC that fails to meet acceptance criteria may require qualification.

Table 7 in **Section IX** presents the recommended DMCs with their associated target compounds. Bear in mind that laboratory methods may deviate from the recommended guidance and surrogate failures should be evaluated based on laboratory SOPs, especially in regard to which DMCs (surrogates) are associated with which target compounds.

For WI GRO analysis, surrogates are not required by the method. If used, the method requires that the surrogates must not elute within the gasoline range organics (GRO) window. Surrogates recommended by the method are nonane (C_9) and nonacosane (C_{29}). Use professional judgment and the above table as guidance for evaluating surrogates in WI GRO samples.

IV. Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. Per method (laboratories may have developed other acceptable QC measures), LCSs should be analyzed:

- Once every preparation batch
- Once for each matrix
- One LCS/LCSD pair every 20 samples of the same matrix for WI GRO analysis

Laboratory control samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 9) and the percent recoveries are evaluated based either on the laboratory's internally generated acceptance windows or default method criteria (as given below).

Table 4 – Guidelines for Evaluating Laboratory Control Sample Recoveries

Analysis	Matrix	Acceptance Criteria	Recommended Action
VOC and associated analyses	aqueous/ sediment/ soil	no guidance from EPA, use laboratory acceptance criteria or professional judgment (generally, 75-125% recovery is acceptable)	if LCS > upper limit & samples are non-detect, no action; if detections, qualify with "*"
			if LCS < lower limit, qualify samples with "**"
			if LCS << lower limit, qualify detects with "**" qualify non-detects with "**"
GRO	aqueous	75-115% recovery <20% RPD	if LCS > 115% & samples are non-detect, no action; if detections, qualify with "*"
			if LCS < 75%, qualify samples with "**"
	soil/sediment	70-120% recovery <20%RPD	if LCS > 120% & samples are non-detect, no action; if detections, qualify with "**"
			if LCS < 70%, qualify samples with "**"

Note: "**"indicates that the reported value is estimated and QA/QA criteria were not met;
"**" indicates that the reported value is unusable and QA/QC criteria were not met.

RPDs are calculated for LCS/LCSD pairs using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

V. Field Duplicates

Field duplicates (also known as "masked or "blind" duplicates) are also used to demonstrate acceptable precision and reproducibility of results by the laboratory. Frequency of collection is project-specific. RPDs are calculated using the equation as provided in the *Equations* section found in the beginning of this SOP, and are not calculated where data is already qualified with b, U, < or **.

Acceptance criteria for field duplicates is subject to the professional judgment of the QC officer, but typically RPDs between 20-30% for aqueous samples and 30-40% for soil and sediment samples are considered acceptable. In cases where the either of samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample is detected and greater than two times (>>2x) the RL, professional judgment should be used to determine if qualification is appropriate.

It is noted that RPD results will be dependent on the heterogeneity of the samples. Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. Use professional judgment when considering qualification of associated results based upon field duplicate RPDs.

VI. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD)

Matrix spikes provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spike and matrix spike duplicate samples contain a known amount of a prescribed number of target compounds (per MN Rules and NELAP; see Table 9).

Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix, or
- 1 per sample delivery group

However, the frequency may be project-specific and the QC officer should review the documents outlining the needs of the project (SAP, QAPP, etc.). In some cases, MS/MSD analysis is not required.

If a matrix spike does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (greater than four times (>4x)), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spikes (and matrix spike duplicates) should be calculated using the appropriate equation for percent recovery provided in the *Equations* section in the beginning of this SOP.

If laboratory acceptance criteria are provided for the percent recoveries of matrix spikes, those criteria should be used for the evaluation of the data. If acceptance criteria are not provided by the laboratory, the percent recoveries recommended by the Guidelines are presented in *Table 8* in **Section IX** may be used for guidance.

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity.

In general, matrix spikes and matrix spike duplicates may be evaluated as follows:

Table 5 – Guidance for Matrix Spike Evaluation

Spike Result	Recommended Action
% recovery > upper acceptance limit	Non-detects, no qualification
	Detects, qualify with "*****"
% recovery meets acceptance limits	No qualification
% recovery is between 20% and lower acceptance limit	Non-detects, qualify with "*****"
	Detects, qualify with "*****"
% recovery is below 20%	Non-detects, use professional judgment; consider qualifying with "******"
	Detects, qualify with "*****"

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation for RPD evaluation provided in the *Equations* section in the beginning of this SOP. Generally, acceptance criteria for MS/MSD is <20-30% RPD for aqueous samples and <30-40% for soils/sediments, but professional judgment should be used for difficult matrices and the acceptance criteria should be adjusted accordingly.

VII. Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project-specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

VIII. Documentation

The QC officer performing the validation should complete a Routine Level Quality Control Report as part of the validation process, making sure that all exceedances of acceptance criteria are documented in the appropriate sections. If revised reports are required, copies should be given to the appropriate data management personnel for record maintenance.

All qualifiers, added, removed or retained, should be documented on the Control Report.

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IX. Additional Tables

The following tables should be used for guidance purposes *only*. Priority should be given to laboratory limits and associated target compounds when provided by the laboratory. Use professional judgment before applying any of the data in the following tables to the validation process.

Table 6 – Recommended Guidance for DMC/Surrogate Recovery (alphabetical)		
DMC	**Recovery limits (%) for aqueous samples**	**Recovery limits (%) for soil samples**
1,1,2,2-Tetrachloroethane-d_2	73-125	56-161
1,1-Dichloroethane-d_2	55-104	45-132
1,2-Dichlorobenzene-d_4	80-131	70-131
1,2-Dichloroethane-d_4	78-129	79-122
1,2-Dicloropropane-d_6	79-124	74-124
1,4-Dioxane-d_8	50-150	50-150
2-Butanone-d_5	49-155	20-182
2-Hexanon-d_5	28-135	17-184
Benzene-d_6	77-124	80-121
Chloroethane-d_5	71-131	61-130
Chloroform-d	78-121	72-123
Toluene-d_8	77-121	78-121
trans-1,3-Dichloropropene-d_4	73-121	72-130
Vinyl Chloride-d_3	65-131	68-122

Table 7 – Target Compounds Associated with DMCs (alphabetical)		
DMC	**Associated Target Compounds**	
1,1,2,2-Tetrachloroethane-d_2	1,1,2,2-Tetrachloroethane	1,2-Dibromo-3-chloropropane
1,1-Dichloroethane-d_2	trans-1,2-Dichloroethene 1,1-Dichloroethene	cis-1,2-Dichloroethene
1,2-Dichlorobenzene-d_4	Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene	1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene
1,2-Dichloroethane-d_4	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride	Methyl-tert-butyl ether 1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
1,2-Dicloropropane-d_6	Cyclohexane Methylcyclohexane	1,2-Dichloropropane Bromodichloromethane
1,4-Dioxane-d_8	1,4-Dioxane	
2-Butanone-d_5	Acetone	2-Butanone
2-Hexanon-d_5	4-Methyl-2-pentanone	2-Hexanone
Benzene-d_6	Benzene	
Chloroethane-d_5	Dichlorodifluoromethane Chloromethane Bromomethane	Chloroethane Carbon disulfide
Chloroform-d	1,1-Dichloroethane Bromochloromethane Chloroform	Dibromochloromethane Bromoform
Toluene-d_8	Trichloroethene Toluene Tetrachloroethene Ethylbenzene	o-Xylene m,p-Xylene Styrene Isopropylbenzene
trans-1,3-Dichloropropene-d_4	cis-1,3-Dichloropropene trans-1,3-Dichloropropene	1,1,2-Trichloroethane
Vinyl Chloride-d_3	Vinyl chloride	

Table 8 – EPA-recommended MS/MSD limits for VOCs				
Compound	**% Rec., Aqueous**	**% RPD, Aqueous**	**% Rec., Soil/Sediment**	**% RPD, Soil/Sediment**
1,1-Dichloroethane	61-145	< 14	59-172	< 22
Trichloroethene	71-120	< 14	62-137	< 24
Benzene	76-127	< 11	66-142	< 21
Toluene	76-125	< 13	59-139	< 21
Chlorobenzene	75-130	< 13	60-133	< 21

Table 9 – Number of Target Compounds Required by NELAP and MN Rules for LCS/LCSD and MS/MSD samples	
Number of Target Parameters	**Required Number of Spiked Compounds**
1-10 analytes	Spike *all* compounds
11-20 analytes	*At least* 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike *at least* 16 compounds

X. Attachments

Attachment 1: Routine Level Quality Control Report
Attachment 2: Barr Qualifiers/Footnotes
Attachment 3: Revisions to SOP

Attachment 1
Routine Level Quality Control Report

Barr Engineering Company
Routine Level Quality Control Report

Project #		**Project Name**	
Laboratory		**COC(s)/Event**	
Lab Report #		**Matrix**	
Report Date		**Review Date**	
Holding Times Met	Yes No	**Reviewed By**	
If no, comments		**Exceptions**	
Temps on Receipt (°C)			

Method Blanks	**LCS/LCSD**
Field Blanks	**MS/MSD**
Trip Blanks (VOCs Only)	
Field Duplicates (if applicable)	**Surrogates (if applicable)**
	Lab Duplicates (if applicable)

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Additional Notes (include historical comparison, if appropriate)

Qualifier Summary		Qualifier Changes		
Sample Name	Parameter	Added	Removed	Retained

Additional Notes to DM

For DM Use Only	
Equis Y / N	Data Tech Init:
Facility ID:	Date entered into Equis:

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Attachment 2
Barr Qualifiers/Footnotes

Data Qualifiers/Footnotes	
Qualifier	**Definition**
--	Not analyzed/not available.
a	Estimated value, calculated using some or all values that are estimates.
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
I	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
j	Reported value is less than the stated laboratory quantitation limit and is considered an estimated value.
p	Relative percent difference is >40% (25% CLP pesticides) between primary and confirmation GC columns.
pp	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
s	Potential false positive value based on statistical analysis of blank sample data.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.
N	Sample Type: Normal
FD	Sample Type: Field Duplicate
AT	Sample chromatogram is noted to be atypical of a petroleum product.
DLND	Not detected, detection limit not determined.
DF	Did not flash
EMPC	Estimated maximum possible concentration.
NA – (Not applicable)	NA indicates that a fractional portion of the sample is not part of the analytical testing or field collection procedures.
ND	Not detected.
TIC	Tentatively identified compound
BQA	Barr-applied project specific qualifier: extraction and/or analyses conducted using an alternative method and/or procedure.
BQC	Barr-applied project specific qualifier: plant shut down.
BQD	Barr-applied project specific qualifier: equipment malfunction.
BQE	Barr-applied project specific qualifier: equipment adjustment.
BQM	Barr-applied project specific qualifier: manual measurement.
BQN	Barr-applied project specific qualifier: unable to be sampled or measured due to various reasons.
BQP	Barr-applied project specific qualifier: atypical chromatographic pattern.
BQQ	Barr-applied project specific qualifier: some aspect of QA/QC was not met.
BQR	Barr-applied project specific qualifier: location was re-sampled.
BQS	Barr-applied project specific qualifier: data is considered suspect.
BQT	Barr-applied project specific qualifier: summed value not displayed due to insufficient field length.
BQU	Barr-applied project specific qualifier: historical qualifier - definition unknown.
BQV	Barr-applied project specific qualifier: estimated value.
BQX	Barr-applied project specific qualifier: see notes for qualifier definition.
BQZ	Barr-applied project specific qualifier: data is considered unusable.

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Attachment 3
Revisions to SOP

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 9
		Attachments	Added Attachment 3
3.2	04/2011	Attachments	Updated Attachments 1 and 2 to include the current forms.

Appendix G

Field Forms

Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number:

Project Name:

Sample Origination State __ __ (use two letter postal state abbreviation)

COC Number:

Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix: Water	Matrix: Soil	Matrix:	Type: Grab	Type: Comp.	Type: QC	Number of Containers/Preservative — Water: VOCs (HCl) #1	SVOCs (unpreserved) #2	Dissolved Metals (HNO_3)	Total Metals (HNO_3)	General (unpreserved) #3	Diesel Range Organics (HCl)	Nutrients (H_2SO_4) #4				Soil: VOCs (tared MeOH) #1	GRO, BTEX (tared MeOH) #1	DRO (tared unpreserved)	Metals (unpreserved)	SVOCs (unpreserved) #2	% Solids (plastic vial, unpres.)			Total Number Of Containers
1.																													
2.																													
3.																													
4.																													
5.																													
6.																													
7.																													
8.																													
9.																													
10.																													

COC _____ of _____

Project Manager: _____

Project QC Contact: _____

Sampled by: _____

Laboratory: _____

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time
Relinquished By:	On Ice? Y N	Date	Time	Received by:	Date	Time

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other: _____

Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:RLG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

Attachment 2
Example - Sample label

Client _____

Project Number _____

Date: _____ Time _____

Preservative: _____

Sampled By: _____

Sample Location: _____

_____

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Attachment 3
Custody Seal – if applicable

Custody Seal

Date_____ Project_____

Signature _____ Container#_____ of_____

UMP013299

Attachment 4
Field Sampling Report

BARR

FIELD SAMPLING REPORT

Date:

Project:

Contact:

Barr Engineering Company
4700 W. 77th Street
Minneapolis, MN 55435-4803

Field Sampling

Field Report

Attachments:

*
*
*
*

*
*
*

Laboratory Analysis Status

<Name inserts here>
Environmental Technician

Attachment 5
Field Log Cover Sheet

BARR

FIELD LOG COVER SHEET
WATER SAMPLING

Client: **Project No.:**

Technician: **Sampling Period:**

Date	Temperature	Wind Speed	Wind Direction	Cloud Cover

Summary of Field Activities

Document1

UMP013301

Attachment 6
Field Log Data Sheet

BARR

Barr Engineering Company
Field Log Data Sheet

Client:		Monitoring Point:						
Location:		Date:						
Project #:		Sample Time:						
GENERAL DATA		STABILIZATION TEST						
Barr lock:		Time/ Volume	Temp. °C	Cond. @ 25	pH	Eh	D.O.	Turbidity Appearance
Casing diameter:								
Total well depth:*								
Static water level:*								
Water depth:*								
Well volume: (gal)								
Purge method:								
Sample method:								
Start time:		Odor:						
Stop time:		Purge Appearance:						
Duration: (minutes)		Sample Appearance:						
Rate, gpm:		Comments:						
Volume, purged:								
Duplicate collected?								
Sample collection by:		CO2- Mn2- Fe(T)- Fe2-						
Others present:								
WELL INSPECTION (answer for each category, state if lock replaced, detail any repairs needed on back of form)								
CASING & CAP: COLLAR: LOCK: OTHER:								
MW: groundwater monitoring well WS: water supply well SW: surface water SE: sediment other:								
VOC- semi-volatile- general- nutrient- cyanide- DRO- Sulfide-								
oil,grease- bacteria- total metal- filtered metal- methane- filter-								
Others:								

*Measurements are referenced from top of riser pipe, unless otherwise indicated.

S:\DM\Templates\MW op\Datasheet.doc

UMP013302

Appendix H

References

References

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Appendix I

Barr Field Audit Program – Field Audit Checklist

BARR ENGINEERING COMPANY
FIELD AUDIT PROGRAM

FIELD AUDIT CHECKLIST

Site/Project Number: _____

Date of Audit: _____

Field Personnel

Name	Title
_____	_____
_____	_____

Auditing Personnel

Name	Title
_____	_____
_____	_____

1.0 Advance Preparation for Sampling

A. Coordination

1. Does MPCA or client need notification of sampling at this site Was that completed? ___

2. Were appropriate sample containers obtained from the laboratory? ___

3. Were sample containers received in good condition? ___

B. Purging and Samping Equipment

The Barr Engineering Company Field Work Check Lists provides a comprehensive overview of the items necessary for successful field event. Sections include: project reference material, miscellaneous tools and supplies, transportation, pumps, bailers, power supplies, documentation and labeling, decontamination, health and safety, other personal gear.

Has a Field Work Check List been completed for the event? ___

If no field work check list was completed, does the field technician have all the proper equipment to perform proper groundwater sampling operations based on the project specific requirements? ___

2.0 Preliminary Field Work

A. Water Level Measurements

1. Was the water level read to the nearest 0.01 foot? ___

2. Was a product interface probe necessary to measure LNAPL or DNAPL? ___

3. Was the water level recorded on the Field Log Data Sheet? ___

4. Was the water level verified with a second reading? ___

5. Was the water level marker decontaminated appropriately? ___

2.0 Sampling of Monitoring Wells

A. Well Purging/Stabilization

1. Verify the correct order of purging/sampling is being followed? ___

2. Field stablization parameters should be measured after several existing well volumes have been removed. Typically, between 3 and 5 well volumes are removed with stablization readings obtained after the third, fourth and fifth column volumes. Was this or equivalent completed? ___

3. Target stablization criteria is given below:
   Temperature +/- 0.1oC
   Specific Conductance (temperature corrected EC) +/-5%
   Dissolved Oxygen +/- 0.5 mg/L
   Redox Potential (-50 to +50) +/-20 mV
   (-100 to +100) +/- 40 mV
   (-200 to +200) +/- 60 mV
   Turbidity: < or = to 10 NTU or +/- 5% if > 10 NTU

   Was the target stabilization criteria met prior to sampling? ___

4. Was calibration of all field instrument completed and documented prior to sampling? ___

5. Was documentation completed as purging activities progressed? ___

6. For low-yielding wells, were they purged dry and allowed to recharge? ___

7. Were a minimum of 3 to 5 well volumes of removed? ___

8. If containerization of purge water is required was it performed? ___

9. Was care given to avoid placing clean sampling equipment (hoses, lines, etc.) on the ground or other potentially contaminated areas prior to use at the well? ___

10. Was appropriate or required purging equipment employed? (i.e., bladder, peristaltic, submerible pumps, clean disposable tubing, etc.) ___

11. Were decontamination procedures for non-dedicated equipment employed? ___

B. Sample Collection

1. Was a clean bailer and line used for sample collection? ___

2. Was the bailer slowly lowered into the well (minimizing aeration)? ___

3. Was the sampling completed "in-line" using dedicated equipment? ___

4. Were vehicles or generator running during sample collection? ___

5. Were the vehicles or generators downwind from the monitoring point? ___

5. Were new sampling gloves worn at the time of collection? ___

6. Were dirty gloves replaced as necessary? ___

7. Were containers filled in the correct order? (i.e., volatiles, semivolatiles, metals, general chemical) ___

8. Were samples filtered as necessary (0.45 micron)? ___

9. Were in-line filtered employed for dedicated wells? ___

10. Was a chain-of-custody completed at the monitoring point? ___

11. Were field QA/QC samples collected as required? ___

12. Were samples placed for "storage" within an acceptable time-frame and on ice (@4oC)? ___

13. Was all non-dedicated or disposable sampling equipment decontaminated as required? ___

Comments: