QUALITY ASSURANCE PROJECT PLAN
HOWARD'S BAY – ST. LOUIS RIVER AOC SEDIMENT INVESTIGATION
SUPERIOR, DOUGLAS COUNTY, WISCONSIN

Revision 1

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
Great Lakes National Program Office
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A.1 TITLE OF PLAN AND APPROVAL

Quality Assurance Project Plan
Howard's Bay – St. Louis River AOC Sediment Investigation
Superior, Douglas County, Wisconsin

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Tonya Balla, WESTON START Team Project Quality Assurance Officer

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Howard Holmes, Columbia Analytical Services Project Manager

Reviewed by: _____ Date: _____
Jim Madison, Test America Burlington Project Manager

Reviewed by: _____ Date: _____
Rick Wilburn, TriMatrix Laboratories Quality Assurance Manager

Reviewed by: _____ Date: _____
Sara Goehl, U.S. EPA GLNPO Task Monitor

Approved by: _____ Date: _____
Louis Blume, U.S. EPA GLNPO Quality Assurance Manager

ACRONYM LIST

%	Percent
°C	Celsius
AOC	Area of Concern
AVS/SEM	Acid Volatile Sulfide/Simultaneously Extracted Metal
CLP	Contract Laboratory Program
COC	Contaminants of Concern
DRO	Diesel Range Organics
FS	Feasibility Study
FSP	Field Sampling Plan
FTL	Field Team Leader
GLLA	Great Lakes Legacy Act
GLNPO	Great Lakes National Program Office
GPS	Global Positioning System
HASP	Health and Safety Plan
IATA	International Air Transport Association
IDW	Investigative-derived Wastes
MQO	Measurement Quality Objective
MS/MSD	Matrix spikes/matrix spike duplicates
ORO	Oil Range Organics
OSWER	Office of Solid Waste and Emergency Response
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PPE	Personal Protective Equipment
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAP	Remedial Action Plan
RI	Remedial Investigation
RSCC	Region V Sample Control Coordinator
R/V Mudpuppy II	Research Vessel Mudpuppy II
SMC	Sample Management Coordinator
SOP	Standard Operating Procedure
START	Superfund Technical Assessment and Response Team
TAL	Target Analyte List
TCL	Target Compound List
TPH	Total Petroleum Hydrocarbon
U.S. DOT	United States Department of Transportation
U.S. EPA	United States Environmental Protection Agency
WDNR	Wisconsin Department of Natural Resources
WESTON	Weston Solutions, Inc.

A.2 INTRODUCTION

The United States Environmental Protection Agency (U.S. EPA) requires that all environmental monitoring and measurement efforts mandated or supported by U.S. EPA participate in a centrally managed quality assurance and quality control (QA/QC) program. Any party generating data under this program has the responsibility to implement minimum procedures to assure that the precision, accuracy, completeness, and representativeness of its data are known and documented. To ensure the responsibility is met uniformly, each party must prepare a written Quality Assurance Project Plan (QAPP) covering each project it is to perform.

This QAPP has been prepared in accordance with U.S. EPA - Region 5, Instructions on the Preparation of a Superfund Division QAPP, based on U.S. EPA QA/R-5, Revision 0, June 2000 and the U.S. EPA Requirements for QAPPs for Environmental Data Operations (U.S. EPA QA/R-5), March 2001. As described in the instructions, this QAPP is organized into four Sections as described below:

SECTION A: The elements in this section cover aspects of project management, objectives, and history. This section identifies the roles and responsibilities of project personnel and describes the communication procedures. This section identifies the goal of the technical assistance at the site.

SECTION B: The elements in this section describe the design and implementation of measurement systems that will be used during the technical assistance at the site. This section describes sampling procedures, analytical methods/procedures, and data handling and documentation procedures. Standard Operating Procedures (SOPs) for sampling and testing are referenced and included as attachments to this QAPP. Quality control procedures, frequency requirements, acceptance criteria, and corrective action procedures associated with all methods are also provided in this section.

SECTION C: The elements in this section describe the procedures used to ensure proper implementation of Section B.

SECTION D: The elements in this section describe the quality assurance (QA) activities that are expected to occur after the data collection phase of the removal assessment is completed.

A.3 DISTRIBUTION LIST

The distribution list is provided in Table A-1.

A.4 PROJECT ORGANIZATION AND RESPONSIBILITIES

Key personnel responsibilities are discussed in the following subsections. Figure A-1 presents the project organization chart.

A.4.1 Project Management

U.S. EPA Great Lakes National Program Office (GLNPO) Task Monitor (TM) - Ms. Sara Goehl is the U.S. EPA GLNPO TM for this project. Ms. Goehl is responsible for all phases of sediment/source investigation for the Howard's Bay-St. Louis River Area of Concern (AOC) project. In addition, all corrective actions will be reported to Ms. Goehl and she will be overall responsible for approving any corrective action or scope changes.

Weston Solutions, Inc. (WESTON®) Program Manager - Ms. Pamela Bayles is the WESTON Program Manager. The Program Manager has overall responsibility for the work assignment. The Program Manager is responsible for ensuring that the project meets all U.S. EPA and overall objectives and quality standards. She is also responsible for ensuring that all work is executed in accordance with the U.S. EPA's technical directives. The WESTON Program Manager is responsible for assigning and monitoring the functions and responsibilities of the WESTON Project Manager. In addition, she will commit the necessary resources and personnel to meet the objectives of this project.

WESTON Project Manager - Mr. Rick Mehl is the WESTON Project Manager. The WESTON Project Manager is responsible for implementing the project objectives utilizing the personnel assigned. The WESTON Project Manager's primary function is to ensure that the technical, financial, and scheduling objectives are achieved successfully. The WESTON Project Manager will coordinate with the WESTON Program Manager, the WESTON Quality Assurance Officer, and other WESTON staff or WESTON subcontractors (as appropriate) and will be the major point of contact and control for matters concerning the project. His other responsibilities include:

- Coordination and management of project personnel
- Project scheduling
- Coordination and review of required deliverables

- General quality assurance (QA) of field activities
- Represent the project team at meetings and public hearings
- Communicating correction action to the U.S. EPA GLNPO TM

U.S. EPA GLNPO Contract Laboratory Program (CLP) Coordinator - Ms. Brenda Jones is the U.S. EPA GLNPO Contract Laboratory/Sample Management Coordinator (SMC) for coordinating the Contractor Laboratory Program Assignments. Ms. Jones will coordinate with the U.S. EPA Sample Management Office (SMO) for sample scheduling and tracking and is the U.S. EPA GLNPO point of contact for funding for use of the CLP.

WESTON SMC - Ms. Tonya Balla is the WESTON Region 5 SMC for coordinating the CLP Assignments with the U.S. EPA GLNPO CLP Coordinator. Ms. Balla will also serve as WESTON's SMC for any WESTON procured subcontractor laboratories, as applicable. Ms. Balla will coordinate all Howard's Bay site sampling requirements and schedules. Ms. Balla will also be responsible for coordinating the review of or performing the analytical data review to ensure that all laboratory protocols and methods were followed and all data is properly validated.

A.4.2 Quality Assurance

U.S. EPA GLNPO Quality Manager - Mr. Louis Blume of the U.S. EPA GLNPO will be responsible for reviewing and approving the QAPP (or his designee). The U.S. EPA Quality Assurance Reviewer also has the discretion to conduct external performance and system audits of field and laboratory activities.

WESTON Quality Assurance Officer (QAO) - Ms. Tonya Balla will be the WESTON project QAO for this assignment. The WESTON QAO has the responsibility to implement and administer the WESTON QA Program. She is responsible for coordinating all procedures and tasks pertaining to QA and reporting to the WESTON Program Manager on QA issues. Other duties include:

- Exercise overall responsibility for all audits under the contract
- Determine projects and activities to be audited
- Establish audit schedules
- Notify the audited entity of nonconformance and the need for corrective actions

- Approve the disposition of nonconformance
- Update and/or develop new standard operating procedures (SOP) in response to an observed need

Ms. Balla or her designee(s) will perform the analytical data quality reviews for the project data. Ms. Balla will function as the WESTON SMC and the WESTON project QAO. The WESTON SMC does procure the subcontractor laboratories. Those procurements are done according to federal procurement requirements. The WESTON SMC role does not generate the data. The WESTON SMC is independent of the WESTON Project Manager. The WESTON SMC and QAO roles should be considered as independent and non-conflicting. Data review and quality control will be also be supported by additional qualified WESTON START staff. WESTON does have an overall START Quality Assurance Officer. That role is supported by Mr. Jim Burton.

A.4.3 Field Team

U.S. EPA GLNPO Project Leader/Field Team Leader (FTL) – Ms. Sara Goehl will act as the GLNPO Project Leader/Field Team Leader (FTL) as well as the GLNPO TM. She will be responsible for the daily direction of the team members regarding the Field Sampling Plan (FSP)-specific tasks. The FSP is contained in Appendix A. The Field Team Leader will provide the initial technical review of all deliverables and data collection activities.

WESTON Project Leader/Field Team Leader (FTL) – Mr. Tim Walls will act as the FTL. He will be responsible for the daily direction of the WESTON team members regarding the work plan-specific tasks. The FTL will provide the initial technical review of all deliverables and data collection activities. In essence, this person will be responsible for the management of the field team and the supervision of all field activities in the absence of the U.S. EPA GLNPO TM. Any deviations or discrepancies to the field sampling plan will be directly related to the U.S. EPA GLNPO TM and the rest of the project team members, as applicable.

WESTON Site Field Safety Officer (FSO) - Mr. Tim Walls will act as the FSO. He will be responsible for implementing the site Health and Safety Plan (HASP). The FSO will perform health and safety monitoring and ensure compliance with all health and safety requirements.

A.4.4 Laboratory

Analysis will be conducted by several laboratories to cover the required scope and analysis. U.S. EPA GLNPO has funding in place on the Region 5 contract for Great Lakes Legacy Act (GLLA) projects. Therefore, the U.S. EPA Contract Laboratory Program (CLP) will be utilized to conduct target analyte list (TAL) metals, including mercury; polychlorinated biphenyls (PCB) as Aroclors; polycyclic aromatic hydrocarbons (PAH); target compound list (TCL) pesticides; and acid volatile sulfide/simultaneously extracted metal (AVS/SEM) analyses. A Weston-procured subcontractor laboratory will perform the tri-butyl tin, total organic carbon (TOC), grain size, total petroleum hydrocarbon (TPH) as diesel range organic (DRO) and oil range organic (ORO) analyses. The WESTON SMC is responsible for initiating and scheduling the laboratory analysis with the U.S. EPA GLNPO CLP Coordinator, and any WESTON procured subcontractor laboratories. The specific analytes being analyzed are included in QAPP Tables A-2 and A-3.

U.S. EPA CLP - The U.S. EPA CLP will be utilized to conduct TAL metals, mercury, PCB Aroclors, PAH, TCL pesticides, and AVS/SEM analyses. The actual CLP laboratories scheduled to conduct the analysis will be assigned at a later date. The U.S. EPA Sample Management Office (SMO) will provide the laboratory(s) assigned for analysis to the GLNPO CLP Coordinator and the WESTON SMC. The CLP modified analysis documentation for PAHs and AVS/SEM are presented in Appendix B.

WESTON Procured Subcontractor Laboratory – Analysis will be conducted by several WESTON procured subcontractor laboratories in order to cover the required scope of work and requested analysis. The parameters include: tri-butyl tin, TOC, grain size, TPH DRO, and TPH ORO analyses.

Columbia Analytical Services - Columbia Analytical Services (CAS) in Kelso, Washington will conduct the TOC analysis. CAS was chosen through a competitive bid process and has provided quality analytical support for other GLNPO projects. Laboratory SOPs for CAS are presented in Appendix C.

The project manager for CAS is Howard Holmes and QA manager is Julie Gish. Mr. Holmes will be responsible for ensuring overall data quality and compliance. Ms. Gish will be responsible for overall implementation of the laboratory QA program. Further detail can be found in Appendix D.

Test America Burlington - Test America Burlington in Burlington, Vermont will conduct the grain size and tri-butyl tin analyses. Test America Burlington was chosen through a competitive bid process.

Laboratory SOPs for Test America Burlington are presented in Appendix C.

The project manager for Test America Burlington is Jim Madison and QA manager is Kristen McCracken. Mr. Madison has 25 years of experience and will be responsible for ensuring overall data quality and compliance. Ms. McCracken has 15 years of experience and will be responsible for overall implementation of the laboratory QA program. Further detail can be found in Appendix D.

TriMatrix Laboratories - TriMatrix Laboratories in Grand Rapids, Michigan will conduct the TPH as DRO and ORO analyses. TriMatrix Laboratories was chosen through a competitive bid process.

Laboratory SOPs for TriMatrix Laboratories are presented in Appendix C.

The laboratory senior project chemist for TriMatrix Laboratories is Lisa Harvey and QA manager is Rick Wilburn. Ms. Harvey will be responsible for ensuring overall data quality and compliance. Mr. Wilburn will be responsible for overall implementation of the laboratory QA program. Further detail can be found in Appendix D.

A.5 PROBLEM DEFINITION/BACKGROUND INFORMATION

The purpose of the Howard's Bay site characterization is to further define the extent of chemical contaminants in the sediment, locate contaminated areas of focus for further evaluation, and identify priority areas for remediation and habitat restoration. The characterization will build on data from 2007 to identify areas to move into the Remedial Investigation/Feasibility Study

(RI/FS) phase, and identify non-impacted areas to help de-list the contaminated sediments for the St. Louis River AOC.

A preliminary scoping meeting was conducted in June prior to the sampling event to determine the accessibility of the proposed sampling locations, identify access issues, identify necessary sampling equipment, and determine the accessibility of the U.S. EPA Research Vessel (R/V) Mudpuppy II to proposed sampling locations.

Howard's Bay is located in the St. Louis River AOC Superior, Douglas County, Wisconsin (Figure A-2). The investigation area for Howard's Bay has been segregated into Area 1 and Area 2 (Figure A-3). Area 1 is an area where limited investigation has previously occurred, while Area 2 is an area that was last investigated in 2007.

Contaminated sediments in the study area contribute to beneficial use impairments in the AOC. The subsequent investigation of the Howard's Bay area will provide insight into contaminated sediments and what influence that may have on beneficial use impairments. Impairment of beneficial use is defined as a change in the chemical, physical, or biological integrity of the Great Lakes ecosystem. The Remedial Action Plan (RAP) identifies the following beneficial use impairments for the St. Louis River AOC:

- Restrictions on fish and wildlife consumption
- Excessive loading of sediment and nutrients
- Degradation of fish and wildlife populations
- Beach closings
- Fish tumors or other deformities
- Degradation of aesthetics
- Degradation of benthos
- Restriction on dredging activities
- Loss of fish and wildlife habitat

In 2004, the St. Louis River Remedial Action Committee identified "clean-up all hotspot contaminated sediments sites by 2020" a goal as part of the AOC's desisting strategy and named Howard's Bay as a target for sediment remediation.

The land surrounding Howard's Bay is primarily industrial and commercial and includes an active ship yard. In general, sediments in the area are suspected to have elevated concentrations of oil and grease, mercury, and heavy metals.

A.6 PROJECT/TASK DESCRIPTION AND SCHEDULE

The field activities directed under this QAPP will focus on known and suspected areas of deposition and contamination. The approximate area of the Howard's Bay project is 300 acres. Water depth is expected to range from 2 to 30 feet and sediment depth is anticipated to be approximately 4 to 5 feet. The data collected during this study will be used by U.S. EPA GLNPO and WDNR to evaluate the locations of the most heavily contaminated sediments, and focus on areas for further evaluation and/or remediation.

The sediment sampling locations were selected using Visual Sampling Plan (VSP) software. The sample design for Area 1 is based on detecting a "hot spot" (local areas of elevated concentration) with a 140 foot radius and a 95% confidence probability. The result of this method yielded a sampling grid of 31 sample locations, based upon these inputs. The sample design for Area 2 is based on detecting a "hot spot" (local areas of elevated concentration) with a 192 foot radius and a 95% confidence probability. The result of this method yielded a sampling grid of 11 sample locations, based upon these inputs. This sampling approach requires systematic grid sampling with a random start. The algorithm used to calculate the grid size (and hence, the number of samples) is based on work by Singer and Wickman for locating geologic deposits (see Singer and Wickman [1969] and Hassig et al. [2004] for details). Inputs to the algorithm include the size, shape, and orientation of a hot spot of interest, an acceptable probability of finding a hot spot. The VSP Reports are presented in Appendix A-A of the FSP (Appendix A). Based on review of the proposed sampling grid during the preliminary scoping meeting held on June 8, 2010, two sample locations were relocated from Area 1 to Area 2 and three sample locations were added to Area 2. As a result, 29 sample locations are proposed for Area 1 and 16 sample locations are proposed for Area 2.

It is estimated that a total of 180 sediment samples (45 sampling locations) will be collected from Howard's Bay. Area 1 will be comprised of 29 sample locations and Area 2 will be comprised of 16 sample locations. Samples will be collected from the following sampling intervals: 0 to 6

inches, 6 to 12 inches, 1 to 3 feet, 3 to 5 feet, etc. to native material. All sediment samples will be analyzed for TAL metals, mercury, PAH list 17, tri-butyl tin, TCL pesticides, TOC, grain size, TPH as DRO, and TPH as ORO. In addition, approximately 30 percent (%) of all samples collected will also be analyzed for AVS/SEM, PAH list 34 (in lieu of PAH list 17), and PCBs as Aroclors. The additional analysis will primarily be conducted on near shore samples from the 0 to 6 inch interval, as well as from all intervals of select cores.

Each location is anticipated to be sampled on a one time basis. Due to sample volume requirements, additional cores may be collected adjacent to original locations. In these cases, the cores will be homogenized together and then the sample collected.

A Global Positioning System (GPS) unit will be used to identify all sample locations. A minimum of two GPS reference points will be located, referenced, photographed, and recorded on the Field Collection Sheet presented in Appendix A-B of the FSP (Appendix A). These GPS reference points will be easily visible, stationary, and will facilitate verification of the locational data for any future activities. In addition to the GPS reference points, the GPS instrument and calibration information, accuracy, and the coordinate system will be recorded. This information will be provided to the project partners for inclusion into their site files. All sample locations shall be uploaded as way-points or point files into a real-time differential GPS with sub-meter accuracy capabilities. Using the navigation capabilities of the GPS, the U.S. EPA R/V Mudpuppy II and any other boat used for sample collection, will be navigated to the pre-determined sample locations shown on Figure A-4.

Field duplicates, for this project, are defined as a duplicate sample collected adjacent to the investigative sample. Field duplicate samples will be collected on a 1:10 frequency and will use procedures identical to those used for the investigative samples. Sample containers and handling and shipment procedures that will be used are identical to those used for the investigative samples. Matrix Spike/Matrix Spike Duplicate (MS/MSD) are samples or sample volume designated for spiking by the laboratory running the analysis. MS/MSD samples will be collected on a 1:20 frequency. MS/MSDs will not be collected for the geophysical parameters (grain size).

The sediment sampling activities in Howard's Bay are tentatively planned to begin the week of October 6, 2010, and is projected to last 5 days. The CLP initial data review should be completed 21 days after receipt by U.S. EPA. Sampling schedule and analytical turnaround times will be coordinated to meet this deadline. Preliminary data will be shared with the U.S. EPA GLNPO TM and other parties as directed by the U.S. EPA GLNPO TM. WESTON will then need two weeks after data receipt to perform a compliance screening and data usability assessment in correlation with the project objectives. WESTON will complete the data review and usability assessment of the WESTON procured subcontractor laboratory parameters within 45 days of receipt. As stated in the scope of work, an Investigation Report that details an overview of the site, summary of historic sampling performed and sampling techniques, data tables, field logs, and photographs is projected to be submitted by December 20, 2010. Project status reports will be submitted to the U.S. EPA GLNPO TM on a monthly basis as required by the applicable contracts.

A.7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The objective of this QAPP is to establish standard procedures so that the integrity, accuracy, precision, completeness, and representativeness of collected samples are maintained and that the required objectives of the work plan are achieved. The objectives of the field activities are to collect geophysical and chemical samples needed to support project area characterization and potential remediation activities.

A.7.1 Data Quality Objectives

Data quality objectives (DQOs) are required for all environmental data collection activities. DQOs are statements that define the type, quality, quantity, purpose and use of data to be collected. Data quality is defined in terms of the study objectives, rather than in terms of equipment or equipment analysis method characteristics. The design of a study is closely tied to the data quality objectives, which serve as the basis for important decisions regarding key design features such as the number and location of samples to be collected, the chemical analyses to be performed, etc. The DQOs must address the hypotheses that are to be proved or disproved and the necessary quality to support or defend the results obtained.

In brief, the DQO process follows a seven-step scientific method that is designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for the intended application, as follows:

1. State the problem that the study is designed to address
2. Identify the decisions to be made with the data obtained
3. Identify the types of data inputs needed to make the decision
4. Define the boundaries (in space and time) of the study
5. Define the decision rule that will be used to make decisions
6. Define the acceptable limits on decision errors
7. Optimize the design for obtaining data in an iterative fashion using information and DQOs identified in Steps 1-6

Following these seven steps helps ensure that the project plan is carefully thought out and that the data collected will provide sufficient information to support the key decisions which must be made. The following sections summarize the application of the DQO process to the design of sediment contamination collection and characterization of contamination in the vicinity of Howard's Bay.

Step 1: State the Problem

It is believed that contaminated sediments in the study area contribute to beneficial use impairments in the AOC. Impairment of beneficial use is defined as a change in the chemical, physical, or biological integrity of the Great Lakes ecosystem.

In 2004, the St. Louis River Remedial Action Committee identified a goal to "clean-up all hotspot contaminated sediments sites by 2020" as part of the AOC's desisting strategy and named Howard's Bay as a target for sediment remediation.

Further evaluation is needed to define the extent of chemical contaminants in the sediment, and identify priority areas for remediation and habitat restoration. The characterization will build on data from 2007 to identify areas to move into the RI/FS phase.

Step 2: Identify Decisions to Be Made

The overall decisions to be made are: 1) what is the extent of spatial contamination within the study area; 2) begin to understand how contamination influences beneficial use impairments; and 3) based on the overall study, what further investigations, additional monitoring, or potential remedial activities are needed.

Step 3: Identify Types of Input Needed

Sediment sampling will be conducted using purposeful sampling. The sampling is purposeful as it focuses on areas of interest for further delineation of the horizontal and vertical extent of contamination. The X and Y coordinates of the proposed locations are provided in FSP Table 1. The core samples will be sampled from 0 to 6 inches, 6 to 12 inches, and then in 2 foot intervals (1 to 3 feet, 3 to 5 feet, etc.) to native material. All sediment samples will be analyzed for TAL metals, mercury, PAH list 17, tri-butyl tin, TCL pesticides, TOC, grain size, TPH as DRO, and TPH as ORO. In addition, approximately 30% of all samples collected will be analyzed for AVS/SEM, PAH list 34 (in lieu of PAH list 17), and PCBs as Aroclors. The additional analysis will primarily be conducted on near shore samples from the 0 to 6 inch interval, as well as from all intervals of select cores. Each location is anticipated to be sampled on a one time basis.

Sampling locations were generated using VSP. Based on review of the proposed VSP sampling grid during the preliminary scoping meeting held on June 8, 2010, two sample locations were relocated from Area 1 to Area 2 and three sample locations were added to Area 2. As a result, 29 sample locations are proposed for Area 1 and 16 sample locations are proposed for Area 2.

Step 4: Define the Bounds of the Study

The investigation area for Howard's Bay has been segregated into Focus Area 1 and Focus Area 2.

It is estimated that a total of 180 sediment samples (45 sampling locations) will be collected from Howard's Bay. The vertical boundary will vary across the areas and is dependent on sediment depth. These vertical boundaries were set to further characterize the vertical extent of contamination for sediment removal volume estimates. Horizontal boundaries are dictated by shoreline, areas of sediment accumulation, and area borders. In the event that access, water

depth, or other factors does not allow for the collection of cores at all of the proposed locations then ponar samples will be collected where possible.

Step 5: Define the Decision Rules

The design of a sampling program depends on a number of factors, but most importantly, the questions that must be answered and the decisions that must be made as a result of the study. Portions of Howard's Bay have been the subject of considerable study. Most recently, studies have indicated that contaminants are present in sediment in the vicinity of Howard's Bay at concentrations above threshold effect concentrations (TECs) and probable effect concentrations (PECs) set forth in the document *Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems"* (MacDonald, et. al., 2000).

The TECs and PECs provide screening criteria to evaluate sediment chemistry data. TECs are defined as concentrations below which adverse effects are not expected to occur. PECs are defined as concentrations above which adverse effects are expected to occur more often than not. Contaminant concentrations above the PECs can often be correlated to observed toxicity in sediment. If the concentrations are above the PEC/TECs, then the area will be considered for potential remedial activities, additional monitoring, or other actions. The data generated will be used to determine if TECs and PECs are applicable or if project specific remedial objectives should be set. The data generated will also likely be used to produce kriging contour maps, and volume estimates for an engineering design for the Howard's Bay investigation area.

Total PAHs will be determined by summing all of the PAH detects and using a value of half the reporting limit for non-detects.

Step 6: Define the Acceptable Limits on Decision Errors

All laboratory data will be reviewed for compliance with established methods and guidelines for acceptability. A decision error of 95% probability of detecting a "hot spot" is used to provide the most adequate data. All usable data will be used to determine the vertical and horizontal extent of contamination. It is the intent of the sampling program that methods and laboratories were chosen that will meet these objectives. However, matrix interferences in the sediment or surface

water could elevate reporting limits. Multiple sampling locations and multiple depths should help ensure enough usable results to meet the DQOs.

Step 7: Optimize Sampling Design

The sampling approach is based on reviewing all of the historical data and supplementing that data with additional sampling locations that will help define the horizontal and vertical extent of contamination. It is the intent of the sampling program that methods and laboratories were chosen to meet these objectives. However, matrix interferences in the sediment could elevate reporting limits. Multiple sampling locations and multiple depths should help ensure usable results to meet the DQOs. Ultimately, the locations of each of the sediment samples will be based on sufficient sediment and the practicality of sampling at each of the locations. Field duplicates will be collected for quality control purposes and to estimate variability due to sampling, sample handling, and analysis. MS/MSD will also be designated to help assess precision and accuracy of the chemical parameters. Once the horizontal and vertical extent of sediment contamination is further delineated, the options to remediate the contaminated sediments will be evaluated.

A.7.2 Precision, Accuracy and Sensitivity of Analysis

Table A-2 presents the reporting limits for the parameters being analyzed through the CLP. Table A-3 presents the reporting, accuracy, and precision limits for the WESTON procured subcontractor laboratories. The CLP accuracy and precision limits are part of the applicable CLP Statement of Works (SOW) SOM01.2 and ILM05.4. The modifications to the standard CLP SOWs for AVS/SEM and PAHs are presented in Appendix B. The WESTON procured subcontractor laboratory SOPs are presented in Appendix C.

Precision

Precision is a measure of the agreement between multiple measurements of the same property carried out under similar conditions. Precision thus reflects the reproducibility of the measurement. Precision is evaluated most directly by recording and comparing multiple measurements of the same parameter made on the same sample under similar conditions.

Precision is expressed in terms of the relative standard deviation (RSD) of the values resulting from the replicate analysis or the relative percent difference (RPD) between the values resulting from duplicate analysis.

Duplicate precision is evaluated by calculating a RPD using the following equation (the smaller the RPD, the greater the precision):

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Field Precision - Field precision will be assessed through the collection and measurement of field duplicates and MS/MSD samples. Field duplicate and MS/MSD frequency (by parameter) is detailed in FSP Section 4.5 (Quality Control Samples). The total number of proposed field duplicates and MS/MSD samples for this field program is presented in the FSP Table 3.

Laboratory Precision - Precision in the laboratory will be assessed through the calculation of RPD. Because the concentration of analytes may be below detection limits in many environmental samples, the RPD data may be generated from the MS/MSD results.

Accuracy

Accuracy is a measure of the agreement between an observed value and an accepted reference value. It is a combination of the random error (precision) and systematic error (bias), which are due to sampling and analytical operations. The laboratory and method accuracy are calculated as a percentage using the following equation:

$$Accuracy = \frac{\text{Measured value}}{\text{True Value}} \times 100$$

Laboratory Accuracy - Laboratory Accuracy will be assessed through the analysis of MS/MSDs, laboratory control samples (LCSs), and surrogate spikes as appropriate to each method.

MS/MSDs are evaluated by analyzing a spiked and un-spiked portion of the same investigative sample. The objective is to equal or exceed the accuracy demonstrated for the analytical method

on samples of similar matrix, composition, and contaminant concentration. The level of recovery of an analyte and the resulting degree of accuracy expected for the analysis of QA samples and spiked samples are dependent on the sample matrix, method of analysis, and the contaminant. The concentration of the analyte relative to the detection limit of the method is also a factor.

The accuracy of the laboratory procedures is also evaluated by the analysis of LCS and laboratory control spike duplicate (LCSD) samples. The LCS/LCSD sample set consists of a clean matrix that is spiked with known constituents. The LCS/LCSD set is prepared and analyzed along with the investigative samples. The LCS/LCSD set is indicative of the accuracy of the laboratory techniques without possible sample matrix interferences. LCSD will not be used for CLP analyses, though may be used for non-CLP evaluation of laboratory techniques.

The accuracy of sample matrix data will be evaluated by determining the %R of matrix spike and surrogate spike samples, where applicable. The spike recovery is calculated using the following equation:

$$\%R = \frac{\text{Observed spike sample conc. - Unspiked sample conc.}}{\text{True concentration of spike}} \times 100$$

Sensitivity

Sensitivity is the ability of the method or instrument to detect the contaminant of concern and other target compounds at the level of interest. Sensitivity is typically expressed in the form of detection limits. The forms of detection or reporting limits listed below will be clarified by the geophysical and chemical laboratories and identified in the site database.

DL - detection limit
QL - quantitation limit
SSDL - sample specific detection limit (i.e., adjusted for percent moisture, dilutions, and sample size)
SSQL - sample specific quantitation limit (i.e., adjusted for percent moisture, dilutions, and sample size)

A.7.3 Completeness, Representativeness, and Comparability

Completeness

Completeness is a measure of the amount of valid data obtained compared to the amount of data that was planned to be collected under normal conditions. Field and laboratory completeness are a measure of the amount of valid measurements obtained from all measurements taken for the project. Valid data will be defined as all data and/or qualified data considered to meet the measurement quality objective (MQO) for this project. It is expected that the laboratories will provide data meeting quality control (QC) acceptance criteria for 90 percent or more of all samples tested (critical samples).

Representativeness

Representativeness is a measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. This is the degree to which samples represent the conditions for which they were taken.

Measures to Ensure Representativeness of Field Data - Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP is followed and that proper sampling techniques are used. The rationale for the sampling network and the sampling techniques are provided in the FSP.

Specific field procedures that will help ensure representativeness of specific samples include:

- Collect samples representative of the entire sample interval
- Use appropriate sampling methodology and equipment
- Use appropriate sampling procedures, including equipment decontamination
- Perform sample procedures consistently and methodically

Measures to Ensure Representativeness of Laboratory Data - Using the proper analytical procedures, meeting sample-holding times, and analyzing and assessing duplicate samples ensures representativeness in the laboratory.

Comparability

Comparability is a measure of the degree to which one data set can be compared to another. Conditions of comparability include standardized siting, standardized sampling and analysis, consistency of reporting units and standardized data format.

Measures to Ensure Comparability of Field Data - Comparability is dependent upon the proper design of the sampling program and will be satisfied by adhering to the standard sample collection, standard analytical procedures, and standard reporting methods described in the FSP.

Measures to Ensure Comparability of Laboratory Data - The analytical data to be obtained during the sampling activities will be comparable to existing data by using similar sampling methods, analytical methods, and QC objectives.

A.7.4 Levels of Quality Control Effort

To assess the quality of data resulting from the sampling program, field duplicates and MS/MSD samples will be collected and submitted to the analytical laboratory. QC samples will also be prepared and analyzed by the laboratory. Laboratory QC samples will include method blanks, laboratory duplicates, and laboratory control samples (as applicable).

Field Quality Control

Field duplicates and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field duplicate samples will be shipped to the analytical laboratory as blind samples. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are known as MS/MSD samples. Field blanks and field splits are not projected for this project. Region 5 U.S. EPA does not promote field blanks for sediment samples.

Field duplicates will be collected at the frequency defined in FSP Section 4.5. Field duplicates will receive a unique sample identification number and will be submitted to the laboratory as a "blind" duplicate to avoid laboratory bias.

MS/MSDs will be collected at the frequency defined in FSP Section 4.5. Additional MS/MSD information is provided in FSP Section 4.5.

Temperature blanks will be included in each cooler being shipped to ensure that the temperature in the cooler meets the specified requirements. Grain size analysis is not required to be shipped on ice.

Laboratory Quality Control

The level of QC for all parameters will be consistent with the CLP SOW and WESTON procured subcontractor laboratory SOPs. Grain size QC will be consistent with the applicable ASTM standard.

A.8 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

Training of field staff will be provided to ensure that technical, operational, and quality requirements are understood. All field team members should receive training including but not limited to, the following:

Logbook training- Training for the maintenance of field, equipment, and personal logbooks

Health and Safety Training - All field staff will maintain health and safety training to ensure compliance with Occupational Safety and Health Administration (OSHA) as established in 29 CFR 1910.120 and 29 CFR 1910.126 (as applicable). This training includes but is not limited to, 40-hour OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) training, 8-hour annual HAZWOPER refresher training, 8-hour supervisor training, cardiopulmonary resuscitation (CPR), first aid training, blood-borne pathogens training, and hazardous materials shipping training.

Data Validation Training - Team members who are responsible for an unbiased assessment of analytical data validation will be trained in accordance with the *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, June 2008; U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (June 2004); National Functional Guidelines for Superfund Organics Method Data Review, U.S. EPA,*

June 2008, and National Functional Guidelines for Superfund Inorganic Method Data Review, U.S. EPA January 2010. Review of data in conformance with these guidelines also lays the underlying foundation for non-CLP generated data review. Data validation/review utilizes information from the laboratory specific planning documents (QAPP/FSP), laboratory specific SOPs, CLP SOWs, and National Functional Guidelines for Data Review, as applicable. Personnel performing data validation have a minimum of 5 years of hands on experience with organic, inorganic, geotechnical, and miscellaneous parameters.

Other - The Site Manager will identify any other additional training for employees required to fulfill the requirement of this QAPP.

U.S. EPA will be responsible for providing training to their field staff as applicable and ensuring that personnel are competent to properly conduct the sampling and document all field activities.

A.9 DOCUMENTATION AND RECORDS

A.9.1 Project Documentation

Project information generated by WESTON and GLNPO will be documented in a format that is usable by all project personnel. Project data and information will be tracked and managed from its inception in the field to its final storage area. These evidentiary files (relevant records, reports, correspondence, logs, field notebooks, pictures, subcontractor reports, data, etc.) will be maintained by the GLNPO TM and the WESTON START Project Manager (and the WESTON QAO, as applicable) in a secured, limited access area. These files will be maintained for a minimum of three years after project closeout and will be offered to the U.S. EPA prior to disposal. Documents and records that will be managed include but are not limited to:

Sample Collection Records - Logbooks, field notes, data collection sheets, chain-of-custody records, custody seals, sample tags, phone conversation records, airbills, and corrective action reports.

Project Data Assessment Records - Field sampling audit checklists, field analytical audit checklists, fixed laboratory audit checklists, performance evaluation (PE) sample results, data validation reports, phone conversation records, and corrective action reports.

Laboratory Analytical Records - The analytical laboratory will be responsible for maintaining analytical logbooks, and laboratory data. Raw laboratory data files and electronic and hard copy data will be inventoried and maintained by the laboratory for the time period established by the U.S. EPA and the laboratory. Laboratory data packages will contain the following information at a minimum: case narrative, calibration summary and raw data, mass spec tuning data (as applicable), gas chromatogram (as applicable), quality control summary forms and raw data, blank results, and method and instrument detection limits.

Data submittals – The data review/validation reports prepared by WESTON for the WESTON procured chemical/geophysical laboratory will be submitted to the GLNPO TM in a PDF format. This submittal will include an overall summary report on the usability of the data, individual validation narrative/check lists for each individual data package, marked up data forms showing the data validation flags, and the GLNPO checklists. In addition, when SEDD/ADR exports are available, they will be included with the data review/validation reports. The overall site database will be exported in the Region 5 EDD format for chemical and geophysical data (field boring and field notes portion of the database) and provided to the GLNPO TM.

All incoming and outgoing correspondence or reports between WESTON and the U.S. EPA GLNPO will be assigned a unique Document Control Number (DCN). A DCN number is assigned to each individual document contained in the project file.

It will ultimately be the responsibility of the GLNPO TM to ensure that all applicable GLNPO, U.S.EPA, federal or state partners, or potentially responsible parties have the most current version of any planning documents or other submittals. It will be the responsibility of the WESTON PM to ensure that all WESTON team members, subcontractors, etc have the most current version of any planning documents or submittals.

SECTION B - DATA GENERATION AND ACQUISITION

B.1 SAMPLING PROCESS DESIGN

B.1.1 Sampling Network and Rationale

The project objectives described previously will be accomplished by collecting sediment samples from each area. Table 2 in the FSP (Appendix A) presents the sample matrices, analytical parameters, and frequencies of sample collection for the current sampling activities.

The sediment sampling locations were selected using VSP software. The VSP for Area 1 is based on detecting a "hot spot" (local areas of elevated concentration) with a 140 foot radius and a 95% confidence probability. The result of this method yielded a sampling grid of 31 sample points, based upon these inputs. The VSP for Area 2 is based on detecting a "hot spot" (local areas of elevated concentration) with a 192 foot radius and a 95% confidence probability. The result of this method yielded a sampling grid of 11 sample points, based upon these inputs. Based on review of the proposed sampling grid during the preliminary scoping meeting, two sample locations were relocated from Area 1 to Area 2 and three sample locations were added to Area 2. As a result, 29 sample locations are proposed for Area 1 and 16 sample locations are proposed for Area 2.

It is estimated that a total of 180 sediment samples (45 sampling locations) will be collected from Howard's Bay. All sediment samples will be analyzed for TAL metals, mercury, PAH list 17, tri-butyl tin, TCL pesticides, TOC, grain size, TPH as DRO, and TPH as ORO. In addition, approximately 30% of all samples collected will be analyzed for AVS/SEM, PAH list 34 (in lieu of PAH list 17), and PCBs as Aroclors. Field efforts will be carefully documented using a site logbook, field data collection sheets, sample COC forms, sample labels, and custody seals.

B.1.2 Parameters to be Tested and Frequency

It is estimated that a total of 180 sediment samples (45 sampling locations) will be collected from Howard's Bay. Samples will be collected from the following sampling intervals: 0 to 6 inches, 6 to 12 inches, 1 to 3 feet, 3 to 5 feet, etc. to "native material". All sediment samples will be analyzed for TAL metals, mercury, PAH list 17, tri-butyl tin, TCL pesticides, TOC, grain size, TPH as DRO, and TPH as ORO. In addition, approximately 30% of all samples collected will be

analyzed for AVS/SEM, PAH list 34 (in lieu of PAH list 17), and PCBs as Aroclors. The additional analysis will primarily be conducted on near shore samples from the 0 to 6 inch interval, as well as from all intervals of select cores.

B.2 SAMPLING METHODS REQUIREMENTS

Sample collection procedures for the various sampling activities are described in the FSP. Quality assurance during sample collection shall be achieved by following the procedures described in the FSP.

B.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

B.3.1 Sample Containers and Handling

Table 5 of the FSP presents the required sample containers, sample preservation methods, and maximum holding times for the proposed environmental sampling. All samples will be placed in appropriate sample containers and labeled.

Sample containers for the CLP parameters will be obtained from Environmental Sampling Supply (ESS). ESS will provide Pre-Cleaned Certified™ (PC Class) containers with Teflon®-lined closures attached. Certification is provided with each case. ESS PC Class containers are provided with certification that exceeds U.S. EPA standards for pre-cleaned containers. Sample containers for the other parameters will be obtained from the WESTON procured subcontractor laboratory. These containers will all also meet and/or exceed U.S. EPA standards for pre-cleaned containers.

The sample labels will include sample number, location, date, time of collection, and analyses to be performed. Samples will be cushioned inside the shipping coolers using bubble wrap or vermiculite. The temperature of the samples will be maintained at 4 degrees Celsius (C) with sealed plastic bags of ice.

Sediment samples will be collected using the U.S. EPA R/V Mudpuppy II and pontoon boat mounted with a vibracoring system. Cores will be collected with a vibracoring system through the sediment depth to native material. In shallower or hard to reach areas, surface sediment samples will be collected with a ponar/eckman dredge or Lexan tube. Core samples will be

measured and sectioned into the following intervals: 0 to 6 inches, 6 to 12 inches, 1 to 3 feet, 3 to 5 feet, etc. The vibracore tube will be cut open, photographed, described on the field collection sheet (Appendix A-B of the FSP) and then will be deposited into a stainless steel pan or disposable aluminum tins, homogenized, and placed in the appropriate sampling containers. Additional details are provided in FSP Sections 4 and 5. The U.S. EPA R/V Mudpuppy II SOPs for the vibracoring system and ponar sample collection are included in Appendix C-C of the FSP.

B.3.2 Documentation

Field efforts will be carefully documented using a site logbook, sample chain-of-custody forms, sample labels, and custody seals. In addition, field copies of this QAPP, the FSP and the HASP will be kept on-site. Field observation and other pertinent information will be recorded in the field. The information to be recorded for each sample will include date, time (24-hour military time reference), sample number, sample location, sample appearance, and the name of the person(s) collecting the sample. This information will be recorded on the Field Collection Sheet (Appendix A-B of the FSP). In addition, general information will be recorded in the site logbook daily, including personnel present at the site, sampling activities, and weather.

B.3.3 Field Log

A site logbook will be kept by the field team members to document site activities, field measurements, sample information, descriptions of photographs, and other relevant information. The logbook will be a bound document with consecutively numbered pages. All entries will be made in ink with no erasures. If an incorrect entry is made, the information will be crossed out with a single strike mark, which will be initialed and dated by the person making the correction. The following information will be recorded in the field logbook on a daily basis:

- Site location identification
- Start date and time (in military time format)
- Weather conditions
- Names of sampling team members
- Site visitors

- Level of personal protective equipment (PPE) used
- Signature

When collecting environmental samples, the following information will be recorded in the field logbooks, on the sample labels, and on the sample tags (if applicable):

- Unique sample identification number
- Date and time of sample collection
- Type of sample collected
- Samplers names
- Analyses to be performed on sample
- Preservatives used, especially any nonstandard types, and any other field preparation of the sample
- In addition to the above information, the logbook will contain a detailed description of the sample location, and the samples physical characteristics (i.e. color, odor, etc).

This information may also be collected on the Field Collection Sheet (Appendix A-B of the FSP).

B.3.4 Sample Custody – Overview

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

- Are in the possession of the individual
- Are in the view of the individual, after being in his/her possession
- Were in the possession of the individual before being placed in a secure location or are in a designated and identified secure area

B.3.5 Chain-of-Custody Form

Chain-of-custody forms will be used to track all samples from the time of sampling to the arrival of samples at the laboratory. Every sample container being shipped, hand delivered to, or picked

up by the laboratory will contain a chain-of-custody form. Field personnel will maintain their copy while the other copies are enclosed in a waterproof enclosure within the shipping container. The laboratory, upon receiving the samples, will sign the remaining copies and keep one copy for its records.

B.3.6 Sampling and Packaging Procedures

To ensure that samples will arrive at the laboratory without breakage and with the chain-of-custody intact, the following sampling and packaging procedures will be followed:

- The field sampler is personally responsible for the care and custody of the samples until they are transferred to another individual or properly dispatched to the laboratory. As few people as possible should handle the samples.
- All sample containers will be labeled with unique sample numbers and sample locations.
- Sample labels will be completed for each sample using waterproof ink unless precluded by weather conditions.

B.3.7 Sample Shipping Procedures

The following transfer of custody and shipment procedures will be followed:

- Samples will be accompanied by a properly completed chain-of-custody record. The sample numbers and locations will be listed on the chain-of-custody record. When transferring the possession of samples, the individuals relinquishing and receiving will sign and record the date and 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 laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. The cooler will be secured shut with strapping tape. U.S. EPA provided custody seals (orange) (or other laboratory custody seal), for evidence purposes, will be taped to the cooler in at least two locations.
- Whenever samples are split with an independent source or government agency, a separate chain-of-custody record will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency will request the representative's signature acknowledging sample receipt.
- All shipments will be accompanied by the chain-of-custody record identifying the contents.
- If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers

will not be required to sign off on the custody records as long as the custody records are sealed inside the sample cooler and the custody seals remain intact.

B.3.8 Laboratory Chain-Of-Custody Procedures

The laboratory custody procedures and document control will be carried out according to individual laboratory procedures detailed in their SOPs or laboratory specific Quality Management Plans.

B.4 ANALYTICAL METHODS REQUIREMENTS

B.4.1 Analytical Laboratory Procedures

The U.S. EPA CLP will be utilized to conduct TAL metals (ILM05.4), mercury (ILM05.4), PCBs as Aroclors (SOM01.2), PAH list 34 (SOM01.2), TCL pesticides (SOM01.2), and AVS/SEM (ILM05.4) analyses. The aforementioned WESTON procured subcontractor laboratories will perform the TOC (ASTM D4129-82/PSEP), grain size (ASTM D 2217 and D422-63), TPH as DRO (USEPA-8015B), TPH as ORO (USEPA-8015B), and tri-butyl tin (MOD-NOAA-NOS-ORCA 71) analyses.

The methods and reporting limits are provided in QAPP Table A-2 for the CLP procured subcontractor laboratory sediment parameters and QAPP Table A-3 for the WESTON-procured subcontractor laboratory sediment parameters. The actual detection limits obtainable for a specific sample depend upon sample characteristics and possible matrix interference. Departures from the detection limits will be consistent with applicable requirements including method adherence, deliverables, audit procedures, and a PE equivalent to the QA/QC procedures in the analytical methods.

B.5 QUALITY CONTROL REQUIREMENTS

B.5.1 Field Quality Control Checks

Field quality control checks are used to assess the representativeness of the sampling. They are designed to determine what effects activities such as sample collection, bottling, shipping, and storage have on sample integrity and to ensure that samples available for analysis in the laboratory are representative of actual conditions on Site. Field quality control checks, which

will be conducted in accordance with the applicable procedures and frequencies described in this QAPP and FSP, and include MS/MSDs, and field duplicates.

B.5.2 Laboratory Quality Control Checks

Internal laboratory QC procedures for the sample analyses are specified in the respective laboratory SOPs. These specifications include the types of QC checks required (method blanks, reagent/preparation blanks, MS/MSD, calibration standards, internal standards, surrogate standards, the frequency of each audit, the specific calibration check standards, laboratory duplicate/replicate analysis), compounds and concentrations to be used, and the QC acceptance criteria for these audits.

Laboratory analysis will be conducted in accordance with the appropriate laboratory SOPs. Internal laboratory quality control checks include: (1) standardization, (2) reagent or method blank generation, and (3) surrogate and matrix spike addition and analysis.

B.6 INSTRUMENT/EQUIPMENT PREVENTIVE MAINTENANCE

B.6.1 Laboratory Instruments

The primary objective of a preventive maintenance program is to help ensure the timely and effective completion of a measurement effort by minimizing the downtime of crucial sampling and/or analytical equipment due to expected or unexpected component failure. In implementing this program, efforts are focused in three primary areas: maintenance responsibilities, maintenance schedules, and adequate inventory of critical spare parts and equipment. Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment down time. This inventory emphasizes those parts (and supplies) which are subject to frequent failure, have limited useful lifetimes, or cannot be obtained in a timely manner should failure occur. The respective laboratory managers are responsible for maintaining an adequate inventory of spare part and backup instrumentation.

Maintenance responsibilities for laboratory equipment will be assigned to the respective laboratory managers. The laboratory managers will then establish maintenance procedures and schedules for each major equipment item. These will be contained in the maintenance logbooks

assigned to each instrument. Preventative maintenance procedures for the WESTON procured subcontractor laboratories CAS, Test America, and TriMatrix are presented in Appendix D.

Any deficiencies noted by the laboratory supervisor or designee will be promptly reported to the WESTON SMC and documented in the laboratory narrative or laboratory exception report which will accompany the final data report. WESTON will immediately relay any issues that affect the data usability to the U.S. EPA GLNPO TM. Preventative maintenance procedures for the CLP laboratories are in the CLP SOWs. Internal CLP preventative maintenance requirements or arrangements with U.S. EPA or the SMO from a contractual standpoint on preventative maintenance procedures, documentation, and corrective action are not the responsibility of WESTON. Any CLP preventative maintenance issues, deficiencies, re-inspection requirements, etc. will be relayed by SMO to the U.S. EPA GLNPO CLP Coordinator.

B.7 INSTRUMENT CALIBRATION AND FREQUENCY

Calibration procedures and frequency of laboratory instrumentation as specified by the CLP SOW, SW846, ASTM, or other approved methods will be strictly adhered to. Calibration of instruments and equipment will be performed at approved intervals as specified by the manufacturer or more frequently as conditions dictate. Calibrations may also be performed at the start and completion of each test run. However, such calibrations will be reinitiated as a result of delay due to work-time breaks, work shift change, or in the event that damage is incurred. Calibration standards used as reference standards will be traceable to the National Institute of Standards and Technology (NIST), when possible. All calibration activities and results will be recorded in the site logbook.

B.7.1 Laboratory Instruments

Records of calibration, repair, or replacement will be filed and maintained by the designated laboratory personnel performing QC activities. These records shall be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory shall maintain a factory trained repair staff with in-house spare parts or shall maintain service contracts with vendors.

B.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

Guidelines for sample container procurement are detailed in Section 7 of the FSP. All sample containers (bottles) will be prepared according to the procedures specified in U.S. EPA's *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers*, (U.S. EPA, 1992) or the most current revision. The WESTON procured subcontractor laboratory will be providing the sample containers for the parameters that they will be analyzing. WESTON will procure the sampling containers for the parameters being analyzed through the CLP. The containers ordered are Pre-Cleaned Certified containers with Teflon-lines closures. Certification is provided with each case. No reagents or blank water are being procured for the field investigation. The U.S. EPA R/V Mudpuppy II staff is supplying the core tubes and caps for sample core collection. WESTON will supply the core tubes and caps for sample core collection on the pontoon boat, and stainless steel pans and scoops for sample homogenizing.

The WESTON Field Team will inspect all materials provided by the laboratories and any field supplies required for sample collection prior to use. Any deficiencies will be noted and replacement consumables utilized, as appropriate. Any corrective actions will be immediately relayed to the U.S. EPA GLNPO TM.

B.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

Historical data/background information is presented in Section A.5 of the QAPP. Historical data was used to determine the parameters to analyze and the general locations that will be sampled. The current assessment will provide information on potential additional source areas.

B.10 DATA MANAGEMENT

B.10.1 Field Measurements and Sample Collection

Raw data from any field measurements and sample collection activities will be appropriately recorded in the site logbook and information will be captured on the field collection sheet (Appendix A-B of the FSP). If the data are to be used in the project reports, they will be reduced or summarized, and the method of reduction will be documented in the report.

B.10.2 Laboratory Reporting and Record-Keeping

The WESTON procured subcontractor laboratories will prepare and submit data packages including the analytical results as well as associated QC. The CLP assigned laboratories will submit their Electronic Data Deliverables (EDDs) and hardcopy in accordance to the appropriate SOW reporting and technical requirements. The laboratory deliverables will include the following (as applicable):

- Narrative, including statement of samples received, description of any deviations from standard procedures, explanation of qualifications regarding data quality, and any other significant problems encountered during analysis.
- A list of the sample numbers analyzed and a run chronology.
- Copies of all raw data including quantitation reports, strip charts, spectra, bench sheets and laboratory notebooks showing tare and sample weights, sample volumes, and other data that will allow the final results reported to be traced back to the analytical steps performed. Each data element should be clearly identified in the laboratory's data package.
- All QC data including Forms I to X (or similar) (e.g., surrogate spike results for each sample, matrix spike, and matrix spike duplicate results, and method blank results).
- All inorganic QC data, including Forms I to X (or similar) (e.g., spike and duplicate results, and method blank results).
- Field and laboratory COC documentation pertaining to each sample delivery group analyzed.
- Flagging descriptions
- Specifications of the number of significant figures for data reporting and analysis.

The Laboratory Project Manager will, as part of the data validation process, confirm that documentation is complete and legible; qualitative identifications are accurate; calculations are accurate; results are expressed in the appropriate units and number of significant figures; and the required quality control checks were run and met acceptance criteria. All pages in all data packages will be consecutively numbered. Review and approval of the data will be documented by the Laboratory Project Manager.

B.10.3 Electronic Records

Data deliverables for the project will comply with the requirements set forth in GLNPO's *Great*

Lakes Legacy Act Data Reporting Standard (March 2010 or latest revision). Analytical data results for the samples will be managed using EquIS. An electronic deliverable document (EDD) and a staged electronic data deliverable (SEDD) (stage 2a) is required for all parameters, except the geotechnical parameters. Only an EDD will be received for the geotechnical parameters.

The SOM01.2 data will be provided in a SEDD stage 3 and the ILM05.4 data will be provided in Agency Standard Format (ASF). SMO performs data assessment on laboratories' hardcopy and electronic deliverables based on contractual and technical requirements outlined in the SOW, and National Functional Guidelines (NFGs) for each analytical service. It includes:

1. Completeness - SMO ensures that all requested data are present and consistent (based on hardcopy and/or electronic reporting requirements).
2. Compliance - SMO compares the analytical Quality Control (QC) results with the SOW, method, contract, and regional validation requirements or guidelines.
3. Recalculation Checks - SMO confirms laboratory reported values (e.g., sample results) by recalculating them using the instrument output data reported by the laboratory in their EDD.
4. Instrument Output - SMO reviews the actual instrument outputs to ensure that the laboratory reported analytes have been correctly identified and quantified.
5. SMO will provide Region 5 and/or their designee with automated data assessment reports outlining discrepancies found and data summary spreadsheets outlining samples, analytes, concentration, lab qualifiers, and data validation qualifiers. The data assessment reports are provided within 24 to 48 hours of data receipt at SMO.
6. Region 5 will receive the data summary spreadsheet in the GLNPO modified format.

WESTON will receive hardcopies of the data from the WESTON procured subcontractor laboratory. Additional information on electronic and hardcopy data deliverables in located in the QAPP Section D.2.

All of the data will be integrated into the EQuIS database. Information collected in the field and SEDDs received from the laboratory are electronically uploaded to EQuIS for data reduction and interpretation purposes. Queries can be executed against the database and summarized in report-quality tables. In addition, analytical data can be compared to screening levels or site-specific clean-up levels. A chemical result that exceeds its respective screening level is displayed in

bold-faced text and the result cell is highlighted in the table for easy identification. Because the database is primarily populated with data from SEDDs, data reporting is typically free of typographical errors. However, qualifications determined by the data reviewers will be manually added to the database and will undergo a manual quality control check.

In addition to preparing data tables that summarize the chemicals identified in the media analyzed, maps can be prepared to display the extent of contamination at the site. WESTON has developed a standard set of geographic information system (GIS) tools for use with Arc View. The labeling tools display the sample location ID, the date and depth of sample collection, and the concentration of the chemical of concern. Because the GIS application is linked to the database, data reporting is "error free".

All chemistry data will be provided to U.S. EPA GLNPO in the Region 5 EDD format. This deliverable will incorporate any data validation flags imposed by the data reviewer.

SECTION C - ASSESSMENT/OVERSIGHT

C.1 ASSESSMENT AND RESPONSE ACTIONS

Assessment of performance of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with procedures established in the FSP and QAPP. Assessment will be performed in the form of audits. Audits of field and laboratory activities include internal and external audits.

Quality assurance system audits may be conducted during activities that may affect the integrity of the sampling program. The objectives of the system audits are:

- To verify that a system of quality control measures, procedures, reviews, and approvals is established for all activities that generates and process environmentally-related data.
- To verify that a system for project documentation (e.g., records, chain-of-custody forms, analytical tags, logbooks, worksheets, etc) is established.
- To verify documentation of the required quality control reviews, approvals, and activity records.
- To identify non-conformances with the established system of quality control measures, procedures, reviews, approvals, and documentation.
- To recommend corrective actions for identified nonconformance.
- To verify implementation of corrective action.
- To provide written reports of audits.

C.1.1 Field Performance Assessment

A field performance assessment (internal audit) may be performed by the U.S. EPA GLNPO TM, the WESTON Project Manager, or a designee. The audit would include examination of sample collection, handling and packaging procedures, chain-of-custody, etc., to ensure compliance with the established requirements. Follow-up audits would be conducted as deemed by the WESTON QAO and/or the U.S. EPA GLNPO TM or WESTON Project Manager, to correct deficiencies and to verify that QA procedures are maintained throughout the entire project.

External audits may also be conducted by the U.S. EPA GLNPO Quality Manager. These audits may be scheduled or unannounced. Since this project also has samples going through the CLP

program, the U.S. EPA GLNPO CLP Coordinator or U.S. EPA GLNPO QAM or their delegate could perform a field audit.

C.1.2 Laboratory Performance Audits

A laboratory system audit is a review of laboratory operations. It is conducted to verify that the laboratory has the necessary facilities, equipment, staff, and procedures in place to generate acceptable data. Each laboratory (CLP and WESTON procured) has laboratory audit procedures which are detailed in their respective laboratory QAPP or laboratory Quality Assurance Manual.. CLP laboratory audits could also be performed by U.S EPA Region 5 or other U.S. EPA entity, as applicable to the CLP program. WESTON will not be performing audits on the subcontractor laboratory.

C.1.3 Corrective Action

Corrective action can result from nonconformance to QAPP requirements. Corrective action may be required due to malfunctioning equipment systems and instruments, or equipment systems and instruments that fail calibration or generate data that exceed stated acceptance limits and may occur during sampling and handling, sample preparation, laboratory instrument analysis, and data review. It is the responsibility of the U.S. EPA GLNPO TM and the WESTON Project Manager to assure that corrective action is initiated as soon as possible. Corrective action taken by WESTON will be communicated to the U.S. EPA GLNPO TM. Any necessary field or sampling corrective action is likely to be identified by the U.S. EPA GLNPO representative onsite during field activities or WESTON's FTL.

For non-compliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the U.S. EPA GLNPO TM, the WESTON Project Manager, or his designee. Any nonconformance with the established QC procedures in the QAPP or FSP will be identified and corrected in accordance with the QAPP. All changes will be evaluated based on their potential to affect the quality of the data. Information on these problems will be promptly communicated to the WESTON QAO and the U.S. EPA GLNPO TM and the U.S. EPA GLNPO

QAM, as applicable. Implementation of corrective actions will be confirmed in writing through the same channels and documented in the site files.

Corrective actions will be implemented and documented in the site logbook. 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 a stop-work order issued by the U.S. EPA GLNPO TM or the WESTON QAO.

For the CLP laboratory and WESTON procured subcontractor laboratories corrective action is implemented at several different levels. The laboratories participating in the CLP are required to have a written SOP specifying corrective action to be taken when an analytical error is discovered or the analytical system is determined to be out of control. The SOP requires documentation of the corrective action and notification by the analyst about the errors and corrective procedures. In addition, the CLP SOWs identifies the appropriate corrective action that must be taken by the laboratory. The SMO, as part of the screening process, verifies that the laboratory has taken the appropriate corrective actions. The WESTON procured subcontractor laboratory also has corrective action procedures. These are detailed in the laboratories Quality Management Plans. This plan is available to the U.S. EPA GLNPO QAO, if requested.

If re-sampling is deemed necessary due to laboratory problems, the U.S. EPA GLNPO TM must identify the necessary approach including cost recovery from the CLP or WESTON procured subcontractor laboratories for the additional sampling effort. The WESTON QAO must be notified in writing of all decisions.

C.2 REPORTS TO MANAGEMENT

Project monthly status reports are issued to U.S. EPA GLNPO TM each month (to the U.S. EPA by the 20th day of each month). The WESTON Project Manager prepares the report for the U.S. EPA GLNPO TM. These reports will include projected delivery dates and schedule delays, results of performance or system audits, deviations from the QAPP or FSP and the associated corrective action and the usability of data. Additional quality assurance information will be included in the project final report.

SECTION D - DATA VALIDATION AND USABILITY

D.1 REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

All data generated in field and laboratory activities will be reduced, reviewed and validated prior to reporting. No data will be disseminated by the laboratory until they have been subjected to the procedures, which are summarized below.

D.1.1 Data Reduction and Review

Raw data from any field measurements and sample collection activities will be appropriately recorded in the site logbook. If the data are to be used in the project reports, they will be reduced and summarized, and the method of reduction will be documented in the report.

Laboratory data reduction procedures will be in accordance with the requirements of the CLP for TAL metals, mercury, PAHs list 17, PAHs list 34, TCL pesticides, AVS/SEM, and PCBs as Aroclors. Laboratory data reduction procedures will be in accordance with the requirements of the WESTON-procured subcontractor laboratory SOPs for tri-butyl tin (organotin), TOC, grain size, TPH as DRO, and TPH as ORO. All laboratory methods to be followed in this investigation are summarized in Tables A-2 and A-3. For each of the methods, the Laboratory Project Manager will complete a thorough inspection of all reports prior to release of the data. Following review and approval of the preliminary report by the Laboratory Project Manager, final reports will be generated and signed by the Laboratory Project Manager.

D.1.2 Data Validation

It is currently projected that the U.S. EPA Region 5 Environmental Services Assistance Team (ESAT) will complete the data validation for all of the analysis conducted by the CLP (TAL metals, mercury, PAHs list 17, PAHs list 34, TCL pesticides, AVS/SEM, and PCBs as Aroclors). However, if the GLNPO TM reassigns this tasking, WESTON will complete the validation of that data following the same procedures that follow. WESTON will complete the data validation for all of the analysis conducted by the WESTON-procured subcontractor laboratories. Completeness is evaluated by auditing the data package for:

- COC records

- Technical holding times
- Required analytical methods
- Reporting limits
- Reporting format
- Laboratory and field QC reporting forms (blanks, surrogates, laboratory control samples, duplicates, matrix spikes, etc., as appropriate)
- Appropriate supporting data
- Case narrative
- Completeness of results
- Data usability (compliance with project MQOs)

Details of any missing, incomplete or incorrect parts of the data packages will be stamped "Resubmitted on [date]", attached to the original data package, and returned to the analytical laboratory. All persons receiving data packages will receive copies of the resubmitted data from the laboratory.

D.2 VALIDATION AND VERIFICATION METHODS

Data may be received in one of several electronic formats: SEDD format (stage 2a – or stage 3 for CLP), U.S. EPA Region 5 GLNPO EDD, and/or the GLSED/NOAA Query template (not currently projected for this project but is a back up option). These formats are all acceptable formats for U.S. EPA GLNPO. The CLP laboratories will submit the SOM01.2, and ILM05.4 data in SEDD stage 3. In addition, a CLP-like data package (hardcopy or complete PDF) will be received with each electronic data set. Data that is received in a SEDD format will be run through an automated data review (ADR) program. The SEDD stage 2a data will also go through a manual compliance check because this level of SEDD deliverable is not all inclusive of the required quality control checks. Data that is received from a subcontracted laboratory in a CLP-like data package (complete package with raw data, narrative, and quality control data), with the Region 5 EDD will be manually validated by WESTON. Any chemical or geotechnical data validation/verification that is conducted by WESTON will be done independent of the WESTON Project Manager. WESTON will complete the QA/QC checklist (referenced in U.S. EPA GLNPO's QMP) for each geotechnical and chemical parameter. WESTON will also prepare an overall data narrative summary (by parameter) that describes any laboratory quality

control issues, data usability issues, completeness issues (overall for the project and by fraction), and any issues pertaining to meeting the project MQOs (precision and accuracy, etc). It is anticipated that U.S.EPA will perform a manual data review of the CLP parameters. Upon receipt of that data, WESTON will do a compliance check on that data to make sure that all quality control components (field quality control samples, etc) were properly evaluated and that the data meets the project DQOs. If any additional qualifications or clarifications are required, WESTON will put a summary report together for the U.S. EPA GLNPO TM and the project file.

Validation for data usability will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in the QAPP, the respective methods, and the laboratory SOPs.

General guidelines for data validation are presented in:

- National Functional Guidelines for Superfund Organics Method Data Review, U.S. EPA, June 2008
- National Functional Guidelines for Superfund Inorganics Method Data Review, U.S. EPA, January 2010
- National Functional Guidelines for Inorganic Data Review, U.S. EPA, October 2004
- Data that is not covered in the functional guidelines will be compared against the applicable analytical methods, the laboratory SOPs, and guidelines described in this QAPP

WESTON will perform a cursory review of the geotechnical parameter grain size. There is minimal documentation to review for quality control purposes. The data will be compared against the applicable ASTM methods. Findings or QC concerns will be included in the data narrative that WESTON will prepare. The WESTON Project Manager, after consultation with the WESTON data reviewers and/or WESTON QAO will be responsible for any corrective action identified during the data validation process for the data generated by the WESTON procured subcontractor laboratory. WESTON will relay any data quality issues associated with any of the data to the U.S. EPA GLNPO TM and the U.S. EPA GLNPO Quality Assurance Manager (as applicable). Any severe corrective actions regarding the CLP data will be the responsibility of the U.S. EPA GLNPO CLP Coordinator. Any other laboratory corrective action

will be the responsibility of the WESTON SMC and possibly the START Program Manager, pending the severity of any issues.

D.3 USABILITY/RECONCILIATION WITH DATA QUALITY OBJECTIVES

Laboratory results will be assessed for compliance with required precision, accuracy, completeness, and sensitivity requirements as described in Section A.7 of this QAPP. Data which does not meet the requirements specified in Section A.7 and QA requirements in the analytical methods will be discussed in the data validation summaries and incorporated into the data assessment report for this project. Sample qualifiers will be identified on all tables in the data assessment report and focused feasibility study. Any sources of sampling or analytical error will be identified as early as possible during the sample collection activities so that corrective action can quickly be implemented. Data which is not deemed usable to support or address the project decision making process will be identified and the potential need for additional sampling will be discussed with all project parties.

FIGURES

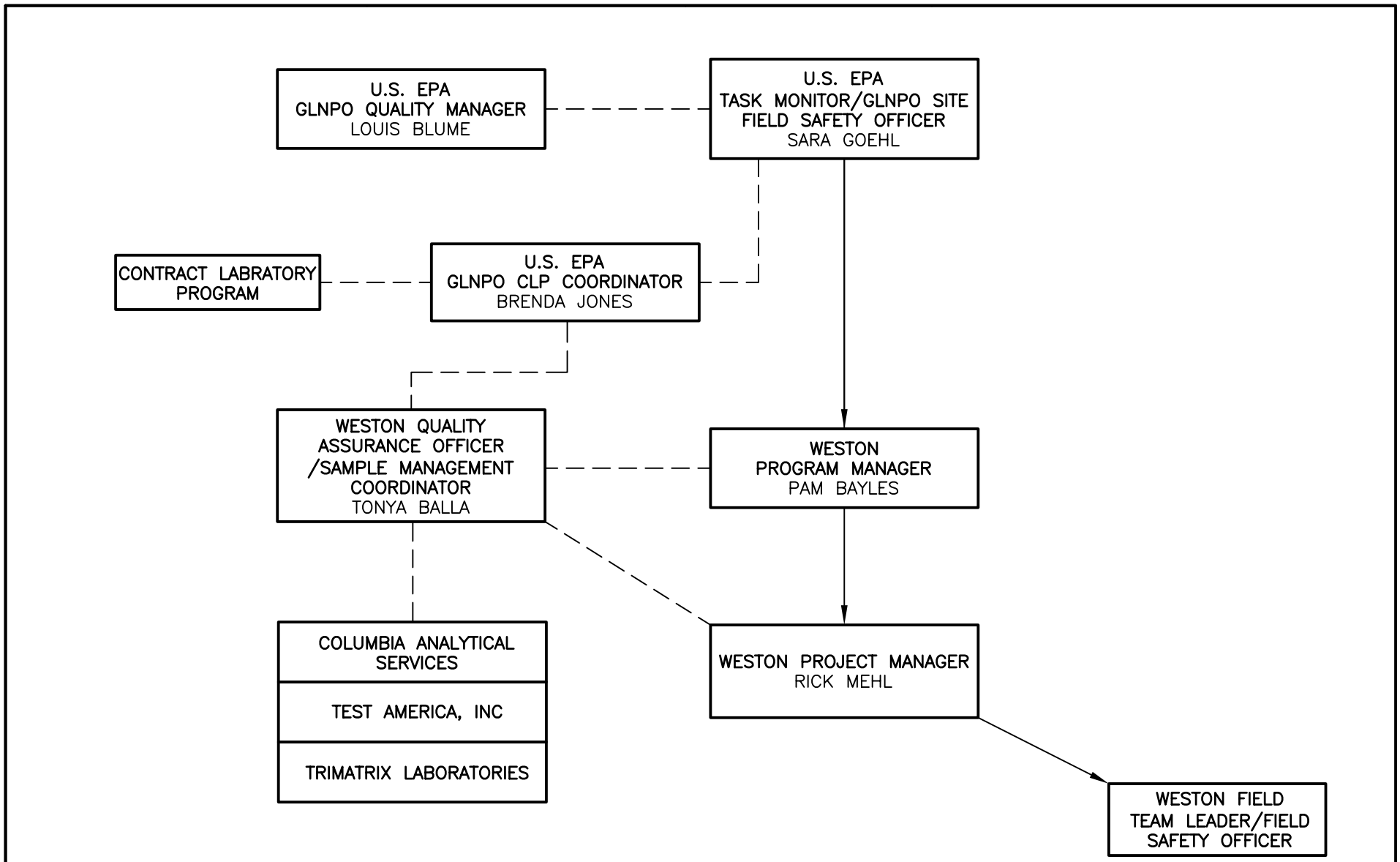

Figure A-1

Prepared for:
U.S. EPA. REGION V
Contract No: EP-S5-06-04
TDD NO: S05-0008-1004-032
DCN: 1023-2E-AHTH

Prepared by:
WESTON SOLUTIONS, INC.
750 East Bunker Court
Vernon Hills, IL 60061

Project Organization Chart

Howard's Bay

Superior, Douglas County, Wisconsin

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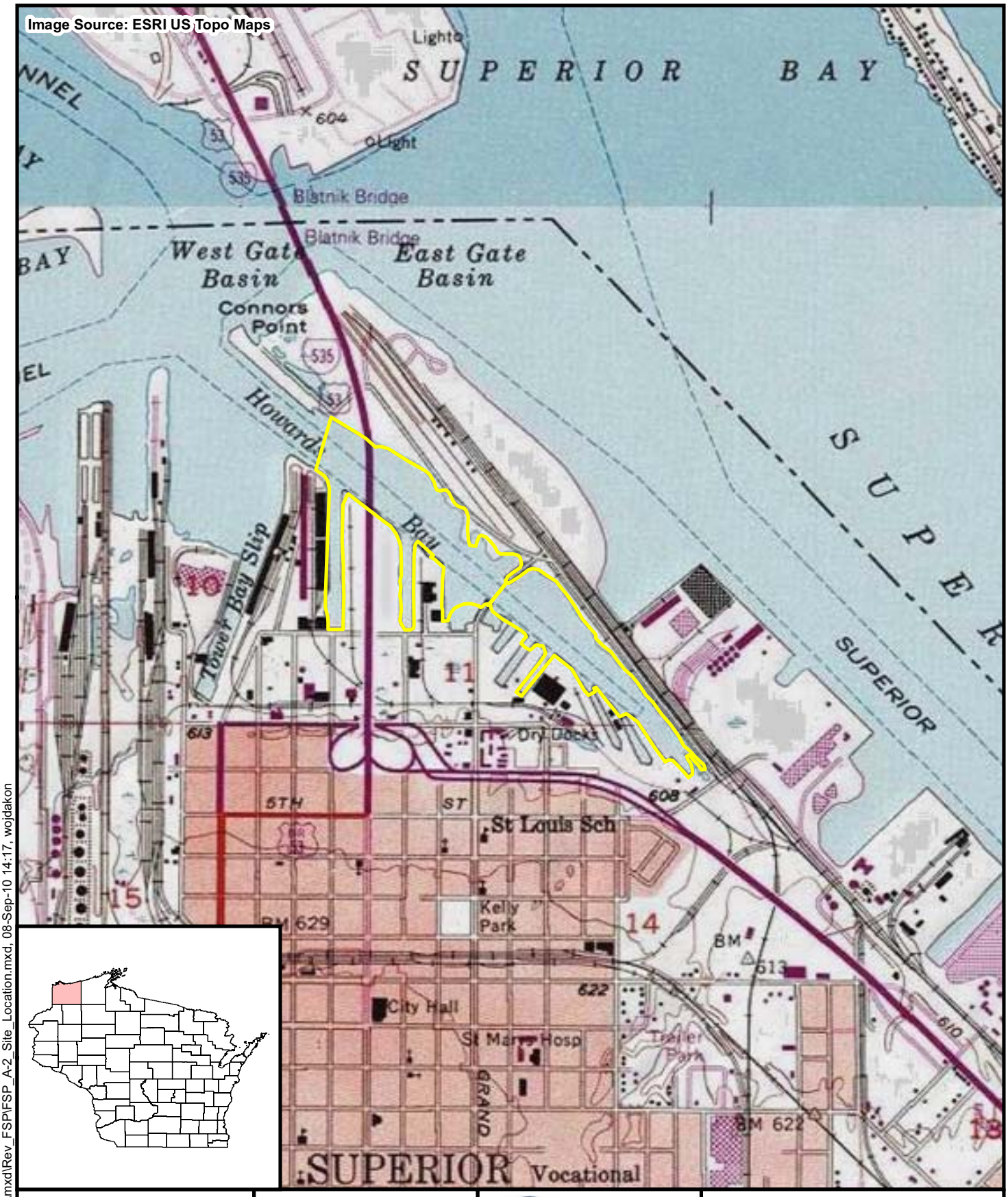

Legend

Project Area

0 2,000 Feet

N

Prepared for:
U.S. EPA REGION V

Contract No.: EP-S5-06-04
TDD: S05-0008-1004-032
DCN: 1023-2E-AHTH

Prepared By:
WESTON SOLUTIONS, INC

750 E. Bunker Court
Suite 500
Vernon Hills, Illinois 60061

Figure A-2
Site Location Map
Howard's Bay
Superior, Douglas County, Wisconsin

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Legend

- Focus Area 1
- Focus Area 2

0 750 Feet

N

Prepared for:
U.S. EPA REGION V

Contract No.: EP-S5-06-04
TDD: S05-0008-1004-032
DCN: 1023-2E-AHTH

Prepared By:
WESTON SOLUTIONS, INC

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Suite 500
Vernon Hills, Illinois 60061

Figure A-3
Site Features Map
Howard's Bay
Superior, Douglas County, Wisconsin

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File: D:\Howards_Bay\mxd\Rev_FSP\FSP_A-4_Prop_Locs.mxd, 08-Sep-10 14:19, wojdakon

Prepared for:
U.S. EPA REGION V
Contract No.: EP-S5-06-04
TDD: S05-0008-1004-032
DCN: 1023-2E-AHTH

Prepared By:
WESTON SOLUTIONS, INC
750 E. Bunker Court
Suite 500
Vernon Hills, Illinois 60061

Figure A-4
Proposed Sampling Locations
Howard's Bay
Superior, Douglas County, Wisconsin

TABLES

Table A-1
QAPP Distribution List
Howard's Bay
Superior, Douglas County, Wisconsin

QAPP Recipients	Title	Organization	Telephone	Email Address
Sara Goehl	U.S. EPA Task Monitor	U.S. EPA Great Lakes National Program Office (GLNPO)	312-886-0270	goehl.sara@epa.gov
Louis Blume	GLNPO Quality Assurance Manager	U.S. EPA GLNPO	312 353-2317	Blume.louis@epa.gov
Howard Holmes	Project Manager	Columbia Analytical Services Laboratory	360 577-7222	hholmes@caslab.com
Jim Madison	Project Manager	Test America, Inc.	802 660-1990	jim.madison@testamericainc.com
Lisa Harvey	Project Manager	TriMatrix Labs	616 975-4500	harveylm@trimatrixlabs.com
Rick Mehl	WESTON Project Manager	Weston Solutions, Inc.	312 424-3312	rick.mehl@westonsolutions.com
Tonya Balla	WESTON Quality Assurance Officer	Weston Solutions, Inc.	847 918-4094	t.balla@westonsolutions.com

Table A-2
U.S. EPA CLP Reporting Limits and Analytical Methods
Howard's Bay Site
Superior, Douglas County, Wisconsin

Analyte	[1]SQTs		[2]SQGs						U.S. EPA CLP	
	Level I	Level II	PEL	SEL	TET	ERM	PEL-HA28	PEC	Reporting Limits	Analytical Reference Method
PAH 17 (µg/kg)										
2-Methylnaphthalene	20	200	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Naphthalene	180	560	NG	NG	600	2100	140	561	3.3	SOM01.2
Acenaphthene	6.7	89	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Acenaphthylene	5.9	130	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Fluorene	77	540	NG	1600	NG	640	150	536	3.3	SOM01.2
Anthracene	57	850	NG	3700	NG	960	170	845	3.3	SOM01.2
Phenanthrene	200	1200	515	9500	800	1380	410	1170	3.3	SOM01.2
Fluoranthene	420	2200	2355	10200	2000	3600	320	2230	3.3	SOM01.2
Pyrene	200	1500	875	8500	1000	2200	490	1520	3.3	SOM01.2
Benzo(a)anthracene	110	1100	385	14800	500	1600	280	1050	3.3	SOM01.2
Benzo(a)pyrene	150	1500	782	14400	700	2500	320	1450	3.3	SOM01.2
Benzo(b)fluoranthene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Benzo(g,h,i)perylene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Benzo(k)fluoranthene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Chrysene	170	1300	862	4600	800	2800	410	1290	3.3	SOM01.2
Dibenz(a,h)anthracene	33	140	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Fluoranthene	420	2200	2355	10200	2000	3600	320	2230	3.3	SOM01.2
Indeno(1,2,3-cd)pyrene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Pyrene	200	1500	875	8500	1000	2200	490	1520	3.3	SOM01.2
Total PAHs	1600	23000	NG	100000	NG	35000	3400	22800	3.3	SOM01.2
PAH 34 (µg/kg)										
2-Methylnaphthalene	20	200	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Naphthalene	180	560	NG	NG	600	2100	140	561	3.3	SOM01.2
Acenaphthene	6.7	89	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Acenaphthylene	5.9	130	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Fluorene	77	540	NG	1600	NG	640	150	536	3.3	SOM01.2
Anthracene	57	850	NG	3700	NG	960	170	845	3.3	SOM01.2
Phenanthrene	200	1200	515	9500	800	1380	410	1170	3.3	SOM01.2
Fluoranthene	420	2200	2355	10200	2000	3600	320	2230	3.3	SOM01.2
Pyrene	200	1500	875	8500	1000	2200	490	1520	3.3	SOM01.2
Benzo(a)anthracene	110	1100	385	14800	500	1600	280	1050	3.3	SOM01.2
Benzo(a)pyrene	150	1500	782	14400	700	2500	320	1450	3.3	SOM01.2
Benzo(b)fluoranthene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Benzo(g,h,i)perylene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Benzo(k)fluoranthene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Chrysene	170	1300	862	4600	800	2800	410	1290	3.3	SOM01.2
Dibenz(a,h)anthracene	33	140	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Fluoranthene	420	2200	2355	10200	2000	3600	320	2230	3.3	SOM01.2
Indeno(1,2,3-cd)pyrene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Pyrene	200	1500	875	8500	1000	2200	490	1520	3.3	SOM01.2
Total PAHs	1600	23000	NG	100000	NG	35000	3400	22800	3.3	SOM01.2
1-Methylnaphthalene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C1-Naphthalenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C2-Naphthalenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C3-Naphthalenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C4-Naphthalenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C1 Fluorenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C2 Fluorenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2

Table A-2
U.S. EPA CLP Reporting Limits and Analytical Methods
Howard's Bay Site
Superior, Douglas County, Wisconsin

Analyte	[1]SQTs		[2]SQGs						U.S. EPA CLP	
	Level I	Level II	PEL	SEL	TET	ERM	PEL-HA28	PEC	Reporting Limits	Analytical Reference Method
C3 Fluorenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C1-Phenanthrenes/Anthracenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C2-Phenanthrenes/Anthracenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C3-Phenanthrenes/Anthracenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C4-Phenanthrenes/Anthracenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C1-Fluoranthenes/Pyrenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C2-Fluoranthenes/Pyrenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C3-Fluoranthenes/Pyrenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C1 Chrysenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C2 Chrysenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C3 Chrysenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
C4 Chrysenes	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Benzo(e)pyrene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Perylene	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2

Table A-2
U.S. EPA CLP Reporting Limits and Analytical Methods
Howard's Bay Site
Superior, Douglas County, Wisconsin

Analyte	[1]SQTs		[2]SQGs						U.S. EPA CLP	
	Level I	Level II	PEL	SEL	TET	ERM	PEL-HA28	PEC	Reporting Limits	Analytical Reference Method
PCB Arocolors (µg/kg)										
Aroclor 1016	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
Aroclor 1221	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
Aroclor 1232	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
Aroclor 1242	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
Aroclor 1248	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
Aroclor 1254	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
Aroclor 1260	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
Aroclor 1262	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
Aroclor 1268	60	680	277	5,300	1,000	400	240	676	33	SOM01.2
TAL Metals (mg/kg)										
Aluminum	NG	NG	NG	NG	NG	NG	NG	NG	20	ILM05.4
Antimony	NG	NG	NG	NG	NG	NG	NG	NG	6	ILM05.4
Arsenic	9.8	33	17	33	17	85	48	33	1	ILM05.4
Barium	NG	NG	NG	NG	NG	NG	NG	NG	20	ILM05.4
Beryllium	NG	NG	NG	NG	NG	NG	NG	NG	0.5	ILM05.4
Cadmium	0.99	5	3.53	10	3	9	3.2	4.98	0.5	ILM05.4
Calcium	NG	NG	NG	NG	NG	NG	NG	NG	500	ILM05.4
Chromium	43	110	90	110	100	145	120	111	1	ILM05.4
Cobalt	NG	NG	NG	NG	NG	NG	NG	NG	5	ILM05.4
Copper	32	150	197	110	86	390	100	149	2.5	ILM05.4
Iron	NG	NG	NG	NG	NG	NG	NG	NG	10	ILM05.4
Lead	36	130	91.3	250	170	110	82	128	1	ILM05.4
Magnesium	NG	NG	NG	NG	NG	NG	NG	NG	500	ILM05.4
Manganese	NG	NG	NG	NG	NG	NG	NG	NG	1.5	ILM05.4
Mercury	0.18	1.1	0.486	2	1	1.3	NG	1.06	0.1	ILM05.4
Nickel	23	49	36	75	61	50	33	48.6	4	ILM05.4
Potassium	NG	NG	NG	NG	NG	NG	NG	NG	500	ILM05.4
Selenium	NG	NG	NG	NG	NG	NG	NG	NG	3.5	ILM05.4
Silver	NG	NG	NG	NG	NG	NG	NG	NG	1	ILM05.4
Sodium	NG	NG	NG	NG	NG	NG	NG	NG	500	ILM05.4
Thallium	NG	NG	NG	NG	NG	NG	NG	NG	2.5	ILM05.4
Vanadium	NG	NG	NG	NG	NG	NG	NG	NG	5	ILM05.4
Zinc	120	460	315	820	540	270	540	459	6	ILM05.4
AVS/SEM (mg/kg)										
Cadmium	NG	NG	NG	NG	NG	NG	NG	NG	0.2	ILM05.4
Copper	NG	NG	NG	NG	NG	NG	NG	NG	0.4	ILM05.4
Lead	NG	NG	NG	NG	NG	NG	NG	NG	3	ILM05.4
Nickel	NG	NG	NG	NG	NG	NG	NG	NG	0.5	ILM05.4
Silver	NG	NG	NG	NG	NG	NG	NG	NG	1	ILM05.4
Zinc	NG	NG	NG	NG	NG	NG	NG	NG	0.4	ILM05.4
TCL Pesticides (µg/kg)										
4,4'-DDD	4.9	28	8.51	60	60	20	NG	28	3.3	SOM01.2
4,4'-DDE	3.2	31	6.75	190	50	15	NG	31.3	3.3	SOM01.2
4,4'-DDT	4.3	63	NG	710	50	7	NG	62.9	3.3	SOM01.2
Aldrin	NG	NG	NG	NG	NG	NG	NG	NG	1.7	SOM01.2
alpha-BHC	NG	NG	NG	NG	NG	NG	NG	NG	1.7	SOM01.2
alpha-Chlordane	NG	NG	8.9	60	30	6	NG	17.6	1.7	SOM01.2
beta-BHC	NG	NG	NG	NG	NG	NG	NG	NG	1.7	SOM01.2

Table A-2
U.S. EPA CLP Reporting Limits and Analytical Methods
Howard's Bay Site
Superior, Douglas County, Wisconsin

Analyte	[1]SQTs		[2]SQGs						U.S. EPA CLP	
	Level I	Level II	PEL	SEL	TET	ERM	PEL-HA28	PEC	Reporting Limits	Analytical Reference Method
delta-BHC	NG	NG	NG	NG	NG	NG	NG	NG	1.7	SOM01.2
Dieldrin	NG	NG	6.67	910	300	8	NG	61.8	3.3	SOM01.2
Endosulfan I	NG	NG	NG	NG	NG	NG	NG	NG	1.7	SOM01.2
Endosulfan II	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Endosulfan sulfate	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Endrin	2.2	210	62.4	1,300	500	45	NG	207	3.3	SOM01.2
Endrin aldehyde	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
Endrin ketone	NG	NG	NG	NG	NG	NG	NG	NG	3.3	SOM01.2
gamma-BHC (Lindane)	2.4	5	1.38	10	9	NG	NG	4.99	1.7	SOM01.2
gamma-Chlordane	NG	NG	NG	NG	NG	NG	NG	NG	1.7	SOM01.2
Heptachlor	NG	NG	NG	NG	NG	NG	NG	NG	1.7	SOM01.2
Heptachlor epoxide	2.5	16	2.74	50	30	NG	NG	16	1.7	SOM01.2
Methoxychlor	NG	NG	NG	NG	NG	NG	NG	NG	17	SOM01.2
Toxaphene	0.1	32	NG	NG	NG	NG	NG	NG	170	SOM01.2

Notes:
AVS/SEM - Acid Volatile Sulfide/Simultaneously Extracted Metal
CLP - Contract Laboratory Program
ERM - Effects Range Median
mg/kg - milligram per kilogram
NG - No Guideline
PAH - Polycylic Aromatic Hydrocarbon
PCB - Polychlorinated Biphenyls
PEC - Probable Effect Concentration
PEL - Probable Effect Level
PELHA28 - Probable Effect Level for Hyalella Azteca (28 day test)
SEL - Severe Effect Level
TAL - Target Analyte List
TCL - Target Compound List
TET - Toxic Effect Threshold
µg/kg - microgram per kilogram
U.S. EPA CLP - United States Environmental Protection Agency

[1] *Evaluation of Numerical SQTs for the St Louis River AOC* (DOI: 10.1007/s00244-002-1155-x)

[2] *Prediction of Sediment Toxicity using Consensus-Based Freshwater SQGs* (EPA 905/R-00/007)

Table A-3
WESTON Procured Subcontractor Laboratory Reporting Limits, Method Detection Limits, and Analytical Methods
Howard's Bay Site
Superior, Douglas County, Wisconsin

Analyte	WESTON Procured Subcontract Laboratory						
	Surrogate (%Rec.)	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)	Reporting Limits (mg/kg)	Method Detection Limits (mg/kg)	Analytical Method Reference
Total Petroleum Hydrocarbon							
DRO	NA	44-135	30-141	20	6.7	1.3	USEPA-8015B
ORO	NA	50-150	50-150	20	10	4.4	USEPA-8015B
o-Terphenyl (Surr.)	44-137	NA	NA	NA	NA	NA	USEPA-8015B
Miscellaneous							
Tributyltin	NA	30-160	30-160	30	1500	370	MOD-NOAA-NOS-ORCA 71 (GC/FPD)
TOC	NA	82-119	77-155	20	0.05	0.02	ASTM D4129-82/PSEP
Grain Size	NA	NA	NA	NA	NA	NA	ASTM D 2217 and D422-63

Notes:
% - percent
DRO - Diesel Range Organic
LCS - Laboratory Control Samples
NA - Not Applicable
ORO - Oil Range Organic
Rec. - Recovery
RPD - Relative Percent Difference
Surr. - Surrogate
TOC - Total Organic Carbon

APPENDIX A

Field Sampling Plan

APPENDIX A

FIELD SAMPLING PLAN

FOR

THE HOWARD'S BAY SITE

SUPERIOR, DOUGLAS COUNTY, WISCONSIN

NPL STATUS: NON-NPL

Prepared for:

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Great Lakes National Program Office

77 West Jackson Boulevard

Chicago, Illinois 60604

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APPENDIX A

FIELD SAMPLING PLAN

FOR

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September 15, 2010

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ACRONYM LIST

%	Percent
°C	Celsius
AOC	Area of Concern
AVS/SEM	Acid Volatile Sulfide/Simultaneously Extracted Metal
CLP	Contract Laboratory Program
COC	Contaminants of Concern
DRO	Diesel Range Organics
FS	Feasibility Study
FSP	Field Sampling Plan
FTL	Field Team Leader
GLLA	Great Lakes Legacy Act
GLNPO	Great Lakes National Program Office
GPS	Global Positioning System
HASP	Health and Safety Plan
IATA	International Air Transport Association
IDW	Investigative-derived Wastes
MS/MSD	Matrix spikes/matrix spike duplicates
ORO	Oil Range Organics
OSWER	Office of Solid Waste and Emergency Response
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PPE	Personal Protective Equipment
PVC	Polyvinyl Chloride
QA	Quality Assurance

QAPP	Quality Assurance Project Plan
QC	Quality Control
RAP	Remedial Action Plan
RI	Remedial Investigation
RSCC	Region V Sample Control Coordinator
R/V Mudpuppy II	Research Vessel Mudpuppy II
SMC	Sample Management Coordinator
SOP	Standard Operating Procedure
START	Superfund Technical Assessment and Response Team
TAL	Target Analyte List
TCL	Target Compound List
TPH	Total Petroleum Hydrocarbon
U.S. DOT	United States Department of Transportation
U.S. EPA	United States Environmental Protection Agency
WDNR	Wisconsin Department of Natural Resources
WESTON	Weston Solutions, Inc.

SECTION 1

INTRODUCTION

This Field Sampling Plan (FSP) identifies the data collection activities and associated quality assurance/quality control (QA/QC) measures specific to the Howard's Bay Site (the Site) located in Superior, Douglas County, Wisconsin (Figure 1), as part of the St. Louis River Area of Concern (AOC) Great Lakes Legacy Act (GLLA) Project. This FSP has been prepared by Weston Solutions, Inc. (WESTON®) under the Superfund Technical Assessment and Response (START) III contract EP-S5-06-04 on behalf of the United States Environmental Protection Agency (U.S. EPA) Great Lakes Nation Project Office (GLNPO).

The purpose of this FSP is to describe site-specific tasks that will be performed in support of the stated objectives. The FSP will reference the Quality Assurance Project Plan (QAPP) for tasks common to all data collection activities including routine procedures for sampling and analysis, sample documentation, equipment decontamination, sample handling, data management, assessment, and data review.

1.1 REPORT ORGANIZATION

The FSP is organized as follows and addresses the following:

- Section 1 – Introduction
- Section 2 – Planning and Problem Definition
- Section 3 – Sample Network Design and Rationale
- Section 4 – Field Investigation Protocols
- Section 5 – General Sampling Protocols
- Section 6 – Sample Team Organization
- Section 7 – Sample Container Procurement

SECTION 2

PLANNING AND PROBLEM DEFINITION

The purpose of the Howard's Bay site characterization is to further define the extent of chemical contaminants in the sediment, locate contaminated areas of focus for further evaluation, and identify priority areas for remediation and habitat restoration. The characterization will build on data from 2007 to identify areas to move into the Remedial Investigation/Feasibility Study (RI/FS) phase. Data collected during this investigation will also help in determining contamination levels that may contribute to beneficial use impairments of this area.

A preliminary scoping event was conducted in June prior to the sampling event to determine the accessibility of the proposed sampling locations, identify access issues, identify necessary sampling equipment, and determine the accessibility of the U.S. EPA Research Vessel (R/V) Mudpuppy II to proposed sampling locations.

2.1 SITE HISTORY AND BACKGROUND

Howard's Bay is located in the St. Louis River AOC Superior, Douglas County, Wisconsin (Figure 1). The investigation area for Howard's Bay has been segregated into Area 1 and Area 2 (Figure 2).

Contaminated sediments in the study area contribute to beneficial use impairments in the AOC. The subsequent investigation of the Howard's Bay area will provide insight into contaminated sediments and what affects that may have on beneficial use impairments. Impairment of beneficial use is defined as a change in the chemical, physical, or biological integrity of the Great Lakes ecosystem. The Remedial Action Plan (RAP) identifies the following beneficial use impairments for the St. Louis River AOC:

- Restrictions on fish and wildlife consumption
- Excessive loading of sediment and nutrients
- Degradation of fish and wildlife populations
- Beach closings
- Fish tumors or other deformities
- Degradation of aesthetics
- Degradation of benthos
- Restriction on dredging activities
- Loss of fish and wildlife habitat

In 2004, the St. Louis River Remedial Action Committee identified "clean-up all hotspot contaminated sediments sites by 2020" a goal as part of the AOC's desisting strategy and named Howard's Bay as a target for sediment remediation.

The land surrounding Howard's Bay is primarily industrial and commercial and includes an active ship yard. In general, sediments in the area are suspected to have elevated concentrations of oil and grease, mercury, and heavy metals.

2.2 CONTAMINANTS OF CONCERN/TARGET ANALYTES

All sediment samples will be analyzed for target analyte list (TAL) metals, mercury, polycyclic aromatic hydrocarbons (PAH) list 17, tri-butyl tin, target compound list (TCL) pesticides, total organic carbon (TOC), grain size, and total petroleum hydrocarbons (TPH) as diesel range organics (DRO) corresponding to an alkaline range of C_{10} through C_{28},, and oil range organics (ORO) corresponding to an alkaline range of C_{28} through C_{36}. In addition, approximately 30

percent (%) of all samples collected will be analyzed for acid volatile sulfide/simultaneously extracted metal (AVS/SEM), PAH list 34 (in lieu of PAH list 17 at surface sediment samples 0-6 inches), and polychlorinated biphenyls (PCB) as Aroclors.

SECTION 3

SAMPLE NETWORK DESIGN AND RATIONALE

The purpose of the Howard's Bay site characterization is to further define chemical contaminants in the sediment, locate contaminated areas of focus for additional evaluation, and identify priority areas for remediation and habitat restoration. The characterization will build on data from 2007 to identify areas to move into the RI/FS phase..

Sampling will focus on known and suspected areas of deposition and contamination. The approximate area of the Howard's Bay project is 300 acres. Water depth is expected to range from 2-30 feet and sediment depth is expected to be approximately 4-5 feet. The data collected during this study will be used by U.S. EPA GLNPO to evaluate the locations of the most heavily contaminated sediments, and focus on areas for further evaluation and/or remediation.

The sediment sampling locations were selected using Visual Sampling Plan (VSP) software. The sample design for Area 1 is based on detecting a "hot spot" (local areas of elevated concentration) with a 140 foot radius and a 95% confidence probability. The result of this method yielded a sampling grid of 31 sample points, based upon these inputs (Figure 3). The sample design for Area 2 is based on detecting a "hot spot" (local areas of elevated concentration) with a 192 foot radius and a 95% confidence probability. The result of this method yielded a sampling grid of 11 sample points, based upon these inputs (Figure 3). Based on review of the proposed sampling grid during the preliminary scoping meeting held on June 8, 2010, two sample locations were relocated from Area 1 to Area 2 and three sample locations were added to Area 2. As a result, 29 sample locations are proposed for Area 1 and 16 sample locations are proposed for Area 2. The samples were relocated based on project specific requirements not satisfied by VSP. The relocated sample points serve to cover particular areas of

interest. The areas of interest not taken into account by the sample design, are particular slips and the creek outfall at the southeast end of Area 2. The points were manually placed in these areas of interest and serve to supplement the VSP sample points.

Based on previous sampling events conducted in Howard's Bay, the sample design was given a higher density in Area 1 where little data has been acquired. Area 2, which is better defined by historic data, was given a lower sample density. The circular pattern was chosen based on the highest probability of encountering a hot spot. The hot spot radii varied in each area based on sample density per near shore and off shore regions. The sample design produced radii for each area with 95% confidence probability of finding a hot spot. This sampling approach requires systematic grid sampling with a random start. The algorithm used to calculate the grid size (and hence, the number of samples) is based on work by Singer and Wickman for locating geologic deposits (see Singer and Wickman [1969] and Hassig et al. [2004] for details). Inputs to the algorithm include the size, shape, and orientation of a hot spot of interest, an acceptable probability of finding a hot spot. The VSP Reports are presented in Appendix A-A.

It is estimated that a total of 180 sediment samples (45 sampling locations) will be collected from Howard's Bay. Area 1 will be comprised of 29 sample locations and Area 2 will be comprised of 16 sample locations. Samples will be collected from the following sampling intervals: 0 to 6 inches, 6 to 12 inches, 1 to 3 feet, 3 to 5 feet, etc. to "native material".

SECTION 4

FIELD INVESTIGATION PROTOCOLS

The sediment sampling activities in Howard's Bay are tentatively planned to begin the week of August 30, 2010, and is projected to last 5 days. U.S. EPA GLNPO and WESTON START will perform the activities detailed in the following subsections.

4.1 SAMPLE NAVIGATION

A Global Positioning System (GPS) unit will be used to identify all sample locations. A minimum of two GPS reference points will be located, referenced, photographed, and recorded on the Field Collection Sheet (Appendix A-B). These GPS reference points will be easily visible, stationary, and will facilitate verification of the locational data for any future activities. In addition to the GPS reference points, the GPS instrument and calibration information, accuracy, and the coordinate system will be recorded. This information will be provided to the project partners for inclusion into their site files. All sample locations shall be uploaded as way-points or point files into a real-time differential GPS with sub-meter accuracy capabilities. Using the navigation capabilities of the GPS, the U.S. EPA R/V Mudpuppy II and any other boat used for sample collection, will be navigated to the pre-determined sample locations shown on Figure 3. Table 1 presents the sample location coordinates.

4.2 SAMPLE LOCATION AND FREQUENCY

Each location is anticipated to be sampled on a one time basis. Due to sample volume requirements, additional cores may be collected adjacent to original locations. In these cases, the cores will be homogenized together and then the sample collected. The number of samples, frequency, and analysis are presented in Tables 2 and 3.

Due to the various nature of industry in the project area, subsurface obstructions may inhibit sampling efforts. Additionally, soft sediment may not allow for representative sample collection. In areas where obstruction occurs and a representative sample is cannot be collected, the locations may be offset to obtain the appropriate material. Each off-set location will be documented on the sample collection form and new GPS data will be acquired for future reference.

4.3 SAMPLING EQUIPMENT AND PROCEDURES

Cores will be completed with a vibracoring system through the sediment depth to native material. In shallower or hard to reach areas, surface sediment samples may be collected with a vibracoring system mounted to a pontoon boat, ponar/eckman dredge, or Lexan core tube.

Table 2 presents the anticipated sampling locations and associated chemical analyses, Table 3 presents a summary of the anticipated sample quantities, Table 4 presents the analytical methods, specific laboratory information, and laboratory specific standard operating procedures (SOPs), Table 5 presents the required sample containers, sample preservation methods, and maximum holding times for the proposed environmental sampling, and Table 6 presents the target compounds of PAH list 17 and 34.

Vibracoring System Sampling Procedures

Subsurface samples will be collected using the vibracorer sampler in accordance with the U.S. EPA R/V Mudpuppy II SOP (Appendix A-C). The vibracoring system consists of the vibrohead, core tube, underwater electrical cable coming from surface support platform to the vibrohead, and control box located between the underwater cable and the power source. The vibracorer applies thousands of vibrations per minute to help penetrate the sediment. When the

core tube is inserted in the core tube clamp, the vibracorer is lowered to one foot above the water body and then turned on. As soon as the core tube touches the sediment, the sediment and water interface create a slurry due to the vibrations between the core tube and sediment.

If locations are inaccessible with the U.S. EPA R/V Mudpuppy II, then a pontoon boat mounted with a vibracoring system may be used. The subsurface samples will be collected using the vibracorer sampler in accordance with the pontoon boat SOP outlined in Appendix A-D.

Samples will be processed on shore at the sample processing area. The samples will be cut open, photographed, and visual observations logged. Samples will be homogenized in an aluminum pan or stainless steel bowl from the following sampling intervals: 0 to 6 inches, 6 to 12 inches, 1 to 3 feet, 3 to 5 feet, etc. to native material. If a partial interval (minimum 6 inches) is collected, the sample interval will be processed for analysis. Samples will be placed in the required sample containers using a disposable poly scoop or stainless steel spoon and maintained at 4 degrees Celsius (°C) with ice after sample collection. All information will be recorded on the Field Collection Sheet, which includes sampling location, sampling date, sample description and sampling time.

Ponar/Eckman Dredge Sampling Procedures

Sediment samples requiring the use of ponar/eckman equipment will be collected in accordance with the U.S. EPA R/V Mudpuppy II SOP (Appendix A-C). The ponar dredge consists of a center pivot, tapered scooped edges, heavy-duty hinges, scoop, underlip, stainless steel screen, and a pinch-pin. It has a scoop volume of 8,200 milliliters and a sampling area of 229 millimeters by 229 millimeters or 9 inches by 9 inches.

Samples will be processed on shore at the sample processing area. The samples will be photographed and visual observations logged. Samples will be homogenized in an aluminum pan or stainless steel bowl from the sampling interval. Samples will be placed in the required sample containers using a disposable poly scoop or stainless steel spoon and maintained at 4°C with ice (except grain size) after sample collection. All information will be recorded on the Field Collection Sheet, which includes sampling location, sampling date, sample description and sampling time.

Lexan Procedures

Sediment samples requiring the use of the Lexan will be collected in accordance with the following procedures. This apparatus consists of a polyvinyl chloride (PVC) holder attached to a length of Lexan tubing. The Lexan is driven manually into the subsurface sediment and a plunger system is retracted as the apparatus is driven to depth. The plunger provides adequate vacuum to retain sediment in the Lexan tube. The Lexan will be used either from a pontoon boat or by wading into the water.

Samples will be processed on shore at the sample processing area. The samples will be photographed and visual observations logged. Samples will be homogenized in an aluminum pan or stainless steel bowl from the sampling interval. Samples will be placed in the required sample containers using a disposable poly scoop or stainless steel spoon and maintained at 4°C with ice after sample collection. All information will be recorded on the Field Collection Sheet, which includes sampling location, sampling date, sample description and sampling time.

4.4 SAMPLE HANDLING AND ANALYSIS

Sediment samples will be collected at the proposed intervals, transferred to the appropriate sample containers outlined in Table 5, and maintained at 4°C with ice (except grain size) after sample collection.

A U.S. EPA Contract Laboratory Program (CLP) lab will perform the TAL metals, mercury, PAHs, TCL pesticides, AVS/SEM, and PCBs as Aroclors analyses. A Weston Procured Subcontractor laboratory will perform the tri-butyl tin, TOC, grain size, and TPH as DRO and ORO analyses. Table 4 presents the analytical methods, specific laboratory information, and laboratory SOPs.

The sample results for TAL metals, mercury, PAHs, TCL pesticides, and PCBs as Aroclors will be compared to the *Evaluation of Numerical Sediment Quality Targets for the St Louis River AOC* and the *Prediction of Sediment Toxicity using Consensus-Based Freshwater Sediment Quality Guidelines*. The sample results for AVS/SEM, tri-butyl tin, TPH DRO, and TPH ORO will be compared to guidelines set forth at a later date. The sample results for TOC and grain size will not be compared to screening criteria.

4.5 QUALITY CONTROL SAMPLES

The sampling effort will include the following types of field QC samples. Table 3 presents the specific level of QC effort for field activities.

- Field duplicates
- Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Field duplicates, for this project, are defined as a duplicate sample collected adjacent to the investigative sample. Field duplicate samples will be collected on a 1:10 frequency and will use

procedures identical to those used for the investigative samples. Sample containers and handling and shipment procedures that will be used are identical to those used for the investigative samples.

MS/MSD are samples or sample volume designated for spiking by the laboratory running the analysis. MS/MSD samples will be collected on a 1:20 frequency.

Each field QC sample will be documented on a chain-of-custody (COC) form.

SECTION 5

SAMPLE NUMBERING SYSTEM

5.1 SAMPLE NUMBERING SYSTEM

All samples collected for analysis, including QC samples, will be given a unique sample number. The sample numbers will be recorded in the site logbook, Field Collection Sheet, COC, and shipping documents.

The field team will assign each sample a project sample number. The project sample number highlights the suspected contaminated area and location, and will be used for documentation purposes in field logbooks, as well as for presentation of the analytical data in memoranda and reports. The project sample numbering system will be composed of the components below.

Project Identifier - The first part of the project sample numbering system will be the two character designation HB. HB corresponds to Howard's Bay.

Year Identifier - The second part will designate the year the sample was collected. The identifier will be a two character designation; 10 for 2010.

Location Identifier - This shall consist of a number or letter representing the locations within each reach starting with 01 for the first core in an area and following through the remaining locations.

- Howard's Bay 2010 Location 01 = HB10-01
- Howard's Bay 2010 Location 32 = HB10-32

Depth Identifier - This shall consist of a two-digit number. The number designation will represent depth in feet below sediment surface of the top of the sample interval. Depth numbers will be rounded to the nearest whole number. Exact depths for a specific sample may be also determined by consulting field log notes.

- Howard's Bay 2010 Location 1 Depth 0-0.5' = HB10-1-005
- Howard's Bay 2010 Location 32 Depth 0.5-1' = HB10-32-051
- Howard's Bay 2010 Location 32 Depth 1-3' = HB10-32-03

Sequence Identifier - This shall consist of the following:

- If the sample is a field duplicate sample, the above will be combined with DP.
- Field duplicate samples will be submitted to the laboratory without reference (i.e.; the laboratory will not be informed that the sample is duplicate).

5.2 SAMPLE DOCUMENTATION AND TRACKING

Field efforts will be carefully documented using a site logbook, Field Collection Sheets, sample COC forms, sample labels, and custody seals. In addition, field copies of the QAPP, the FSP, and the Health and Safety Plan (HASP) will be available on-site.

Documentation

Field observation and other pertinent information will be recorded in the field. The information to be recorded for each sample will include date, time (24-hour military time reference), sample number, sample location, sample appearance, and the name of the person(s) collecting the sample. This information will be recorded on the Field Collection Sheet (Appendix C). In addition, general information will be recorded in the site logbook daily, including personnel present at the site, sampling activities, and weather.

Field Log

A site logbook will be kept by the field team members to document site activities, field measurements, sample information, descriptions of photographs, and other relevant information. The logbook will be a bound document with consecutively numbered pages. All entries will be

made in ink with no erasures. If an incorrect entry is made, the information will be crossed out with a single strike mark, which will be initialed and dated by the person making the correction.

The following information will be recorded in the field logbook on a daily basis:

- Site location identification
- Start date and time (in military time format)
- Weather conditions
- Names of sampling team members
- Site visitors
- Level of personal protective equipment (PPE) used
- Signature

When collecting environmental samples, the following information will be recorded in the field logbooks, on the sample labels, and on the sample tags:

- Unique sample identification number
- Date and time of sample collection
- Type of sample collected
- Samplers names
- Analyses to be performed on sample
- Preservatives used, especially any nonstandard types, and any other field preparation of the sample
- In addition to the above information, the logbook will contain a detailed description of the sample location, and the samples physical characteristics (i.e. color, odor, etc).

This information may also be collected on the Field Collection Sheet (Appendix A-B).

Field Chain-of-Custody Procedures

Table 5 of the FSP presents the required sample containers, sample preservation methods, and maximum holding times for the proposed environmental sampling. All samples will be placed in appropriate sample containers and labeled. The sample labels will include sample number,

location, date, and time of collection, and analyses to be performed. The labels will be created using the Forms II Lite software. The label information will be completed in permanent, non-erasable blue or black ink. Samples will be cushioned inside the shipping coolers using bubble wrap, vermiculite, or other suitable packing material. The temperature of the chemical samples will be maintained at 4°C with sealed plastic bags of ice.

Samples will be shipped via commercial air courier on a daily basis (as feasible) to the respective analytical laboratories. The exception to this procedure will be for samples that are collected on a Sunday or a holiday. For samples collected on a Sunday or holiday, additional ice will be placed in the coolers or samples will be placed in a secure refrigerator. The coolers or refrigerator will be sealed and kept in a designated secure area until they are picked up by the courier or delivered to the laboratory on the next business day.

Prior to shipment, two custody seals will be fastened to the right and left sides of each shipping cooler to secure the lid and provide evidence that the samples have not been tampered with en route to the laboratory. Upon receipt of the cooler at the laboratory, the cooler will be inspected by the laboratory's sample custodian.

5.3 LABORATORY DOCUMENTATION FORMS

Required paperwork for laboratory samples includes COC, and COC seals. All sample documentation forms will be prepared by WESTON personnel in accordance with the requirements outlined in the *CLP Guidance for Field Samplers* (U.S. EPA, 2007) or the most recent version. The U.S. EPA is currently using the Forms II Lite Software (Version 5.1.47). The WESTON Sample Management Coordinator (SMC) will train all field personnel on any new documentation requirements before field activities begin.

All paperwork accompanying the samples being shipped to the laboratory will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies will be made of all sample documentation and retained for in-house files.

Chain-of-Custody Form

To maintain custody in accordance with the U.S. EPA requirements, the following sample documentation protocol must be implemented:

- Each sample shipment container must have at least one COC form enclosed with the samples.
- Each sample in a shipment container must be identified and documented on the accompanying COC form.
- The COC seal numbers on seals assigned to a particular cooler must be documented on the COC form in that cooler.
- The carrier service person does not have to sign the COC form if the custody seals remain intact. The airbill number (if applicable) must be written on the COC form.
- Forms II Lite will be utilized to generate the COC for any CLP samples as well as samples going to any WESTON-procured laboratory with the exception of the toxicity laboratory and the dewatering laboratories. Those laboratory standard COC forms will be utilized.

The laboratory custody procedures and document control will be carried out as specified in the individual laboratory SOPs. Laboratory custody procedures are further defined in the QAPP.

Chain-of-Custody Seals

COC seals are provided by the U.S. EPA.

- Two seals will be used per shipping container to secure the lid and provide evidence that the samples have not been tampered with.

- The COC seals must be covered with clean tape to avoid accidental damage during shipment.
- The COC seal numbers must be documented on the COC form.
- All sample shipment containers require COC seals.

5.4 SAMPLE SHIPPING PROCEDURES

The following transfer of custody and shipment procedures will be followed:

- Samples will be accompanied by a properly completed COC record. The sample numbers and locations will be listed on the COC record. When transferring the possession of samples, the individuals relinquishing and receiving will sign and record the date and 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 laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be secured with custody tape for shipment to the laboratory. The cooler will be secured shut with strapping tape. The laboratory-provided custody seals will be taped to the cooler in at least two locations.
- Whenever samples are split with an independent source or government agency, a separate COC record will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency will request the representative's signature acknowledging sample receipt.
- All shipments will be accompanied by the COC record identifying the contents.
- If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers will not be required to sign off on the custody records as long as the custody records are sealed inside the sample cooler and the custody seals remain intact.

5.5 SAMPLE HANDLING

Sample Containers and Sample Preservation

The required sample containers, sample volumes, sample preservation requirements, and holding times associated with all parameters and media applicable to the Howard's Bay sampling activities are presented in Table 5. Sample containers will be obtained according to U.S. EPA specifications as described in Section 7.

Sample Packaging and Shipment

All samples shipped from the site must be shipped in accordance with current United States Department of Transportation (U.S. DOT) regulations and International Air Transport Association (IATA).

All samples will be processed on shore at the sample processing area. Following sampling, all sample caps will be tightened and the exterior of all sample bottles will be initially decontaminated by spraying off with water and wiping with a moist cloth. In preparation for shipment to the laboratories, all samples will be packaged in accordance with the following general procedures:

- Check to make sure container cap is securely tightened.
- Make sure the sample labels are securely attached to the sample containers. Place each container in a zip-lock bag, ensuring that the sample tags can be read.
- Samples will be placed in a shipment container lined with a large plastic bag. Enough absorbent material will be packed around the samples to minimize the possibility of sample container breakage. The temperature will be maintained at $4^{o}C$ with ice sealed in plastic bags as appropriate to the sample. The remaining space in the container will be filled with additional packing material and the large bag sealed.
- Place COC packing lists in a zip-lock bag and tape to inside of shipment container lid.
- Close shipment container and seal it shut with strapping tape. If shipment container has a drain port, seal it shut with tape. Place custody seals across seam between the container lid and base so that custody seal would be broken if shipment container was opened. Cover custody seals with clear tape.

- Affix airbill with shipper's and recipient's names and addresses to top of shipment container or to a cooler tag provided by the air courier. Affix a second mailing label with the same information to the top of container in case airbill becomes detached from container during shipment. Place "This End Up" labels on container.

5.6 DECONTAMINATION PROCEDURES

Prior to commencing work, sampling equipment (i.e.; vibracoring system, ponar/eckman dredge, and Lexan) will be thoroughly cleaned. Any stainless steel pans/buckets or spoons will be thoroughly rinsed between sample locations at the sample processing area. Whenever possible, disposable sampling supplies such as plastic scoops and aluminum trays will be used. No fluids or sediment is anticipated to be containerized. However, if conditions warrant, the management of water generated during decontamination will be in accordance with the requirements outlined below.

In general, decontamination procedures will include the following sequential steps:

- Sampling equipment on the boats will be rinsed with river water and a brush used to remove any particulates
- Equipment used at the sample processing area will be rinsed with river water and as necessary, scrubbed with detergent (Alconox) using a brush to remove particulates
- Rinse with river water
- Rinse with distilled water, as necessary
- Air dry for as long as possible

In the event of oily material that is not easily removed from any piece of sampling equipment, a methanol spray will be applied and the area scrubbed using a brush. If the use of methanol is required, that water will be collected for offsite disposal.

5.7 WASTE HANDLING

For purposes of this FSP, IDW are defined as any by-product or disposable equipment from the field activities that is suspected or known to be contaminated with any hazardous substances. The performance of field activities will produce waste products such as decontamination wastewater, disposable sampling equipment, and expendable personnel protective equipment.

Decontamination water will consist of Alconox and surface water only. It is the intention of the Mudpuppy 2 crew and pontoon boat crew to wash the sampling equipment over the edge of the boat. No fluids will be containerized. Excess sediment from the sample cores will be containerized and disposed of in accordance with applicable regulations. Any PPE, spent core tubes will be bagged and properly disposed of in an onsite dumpster.

The field team will refer to the U.S. EPA's *Management of Investigation-Derived Wastes During Site Inspections* (U.S. EPA, 1991) for guidance on off-site disposal policy, if this action is deemed necessary.

SECTION 6

SAMPLE TEAM ORGANIZATION

This project is a federal-lead project which is being coordinated by a U.S. EPA GLNPO Task Monitor. They have overall responsibility for all phases of this project. Key personnel responsibilities are detailed in the QAPP Section A.4.

SECTION 7

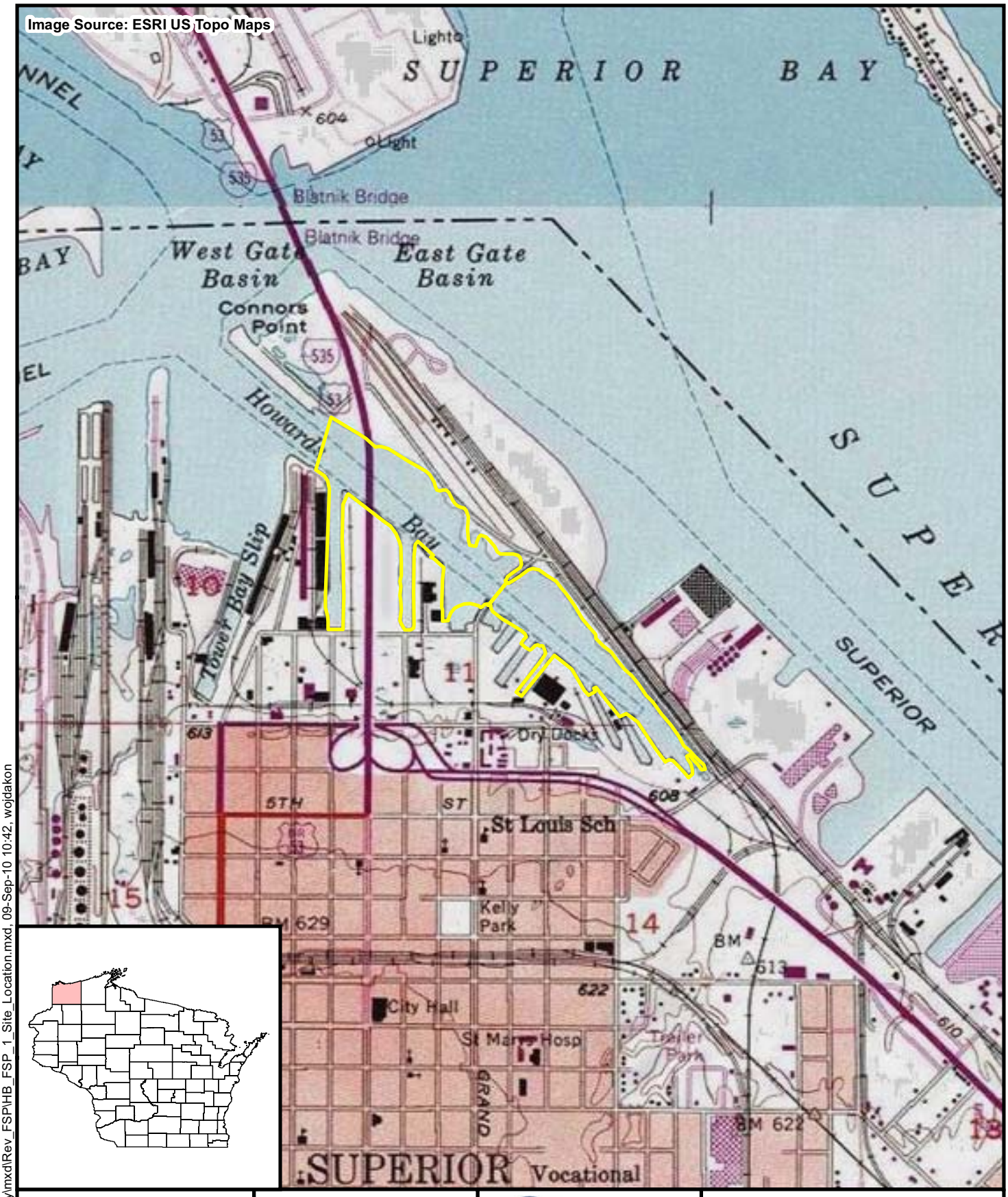
SAMPLE CONTAINER PROCUREMENT

Sample containers being used for chemical analysis will meet or exceed the strict quality control requirements set forth by the U.S. EPA in the Office of Solid Waste and Emergency Response (OSWER) Directive No. 9240.0-05A. WESTON will procure the sampling containers for the analyses that are being conducted by the CLP. Sample containers will be provided by the WESTON Procured Subcontractor Laboratory for each of the parameters being conducted by their laboratory.

All sample containers will be prepared according to the procedures specified in U.S. EPA's *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers*, (U.S. EPA, 1992) or the most current revision. It will be ensured that the bottles used for the sampling activity do not contain target organic and inorganic contaminants exceeding the level specified in the above-mentioned document.

Corrective actions will be conducted comprehensively to avoid the use of identified contaminated lots from other projects, and to ensure that if the bottle suppliers are deemed unresponsive or unable to provide cleaned bottles as specified, then other U.S. EPA-related projects are not negatively affected by the use of the noncompliant bottles.

FIGURES



Legend

Project Area

0 2,000 Feet

N

Prepared for:
U.S. EPA REGION V

Contract No.: EP-S5-06-04
TDD: S05-0008-1004-032
DCN: 1023-2E-AHTH

Prepared By:
WESTON SOLUTIONS, INC

750 E. Bunker Court
Suite 500
Vernon Hills, Illinois 60061

Figure 1
Site Location Map
Howard's Bay
Superior, Douglas County, Wisconsin

File: D:\Howards_Bay\mxd\Rev_FSP\HB_FSP_1_Site_Location.mxd, 09-Sep-10 10:42, wojdakon

Legend

- Focus Area 1
- Focus Area 2

0 750 Feet

N

Prepared for:
U.S. EPA REGION V

Contract No.: EP-S5-06-04
TDD: S05-0008-1004-032
DCN: 1023-2E-AHTH

Prepared By:
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750 E. Bunker Court
Suite 500
Vernon Hills, Illinois 60061

Figure 2
Site Features Map
Howard's Bay
Superior, Douglas County, Wisconsin

File: D:\Howards_Bay\mxd\Rev_FSP\HB_FSP_2_Site_Features.mxd, 09-Sep-10 10:43, wojdakon

File: D:\Howards_Bay\mxd\Rev_FSP\HB_FSP_3_Prop_Locs.mxd, 09-Sep-10 10:45, wojdakon

Prepared for:
U.S. EPA REGION V

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750 E. Bunker Court
Suite 500
Vernon Hills, Illinois 60061

Figure 3
Proposed Sampling Locations
Howard's Bay
Superior, Douglas County, Wisconsin

TABLES

Table 1
Sample Location Coordinates
Howard's Bay Site
Superior, Douglas County, Wisconsin

Location ID	UTM NAD83 Zone 17 - X	UTM NAD83 Zone 17 - Y	Latitude	Longitude	Latitude (D M S)	Longitude (D M S)	Latitude (D M.m)	Longitude (D M.m)
Area 1								
HB-01	-347515.9626	5236800.0977	46.743384	-92.099295	46 44' 36.18" N	92 5' 57.46" W	46 44.603' N	92 5.957' W
HB-02	-347557.1914	5236728.6871	46.742701	-92.099693	46 44' 33.72" N	92 5' 58.89" W	46 44.562' N	92 5.981' W
HB-03	-347474.7338	5236728.6871	46.742805	-92.098634	46 44' 34.10" N	92 5' 55.08" W	46 44.568' N	92 5.918' W
HB-04	-347392.2760	5236728.6871	46.742909	-92.097575	46 44' 34.47" N	92 5' 51.27" W	46 44.574' N	92 5.854' W
HB-05	-347515.9626	5236657.2765	46.742122	-92.099033	46 44' 31.64" N	92 5' 56.51" W	46 44.527' N	92 5.942' W
HB-06	-347433.5049	5236657.2765	46.742226	-92.097974	46 44' 32.02" N	92 5' 52.70" W	46 44.534' N	92 5.878' W
HB-07	-347351.0472	5236657.2765	46.742330	-92.096914	46 44' 32.39" N	92 5' 48.89" W	46 44.540' N	92 5.815' W
HB-08	-347268.5895	5236657.2765	46.742434	-92.095855	46 44' 32.76" N	92 5' 45.07" W	46 44.546' N	92 5.751' W
HB-09	-347557.1914	5236585.8658	46.741440	-92.099431	46 44' 29.18" N	92 5' 57.95" W	46 44.486' N	92 5.966' W
HB-10	-347392.2761	5236585.8658	46.741648	-92.097313	46 44' 29.93" N	92 5' 50.32" W	46 44.499' N	92 5.839' W
HB-11	-347309.8183	5236585.8658	46.741752	-92.096254	46 44' 30.31" N	92 5' 46.51" W	46 44.505' N	92 5.775' W
HB-12	-347227.3606	5236585.8658	46.741856	-92.095195	46 44' 30.68" N	92 5' 42.70" W	46 44.511' N	92 5.712' W
HB-13	-347351.0473	5236514.4552	46.741069	-92.096652	46 44' 27.85" N	92 5' 47.94" W	46 44.464' N	92 5.799' W
HB-14	-347268.6830	5236514.4620	46.741173	-92.095594	46 44' 28.22" N	92 5' 44.14" W	46 44.470' N	92 5.736' W
HB-15	-347186.2251	5236514.4620	46.741277	-92.094535	46 44' 28.60" N	92 5' 40.32" W	46 44.477' N	92 5.672' W
HB-16	-347536.6847	5236512.9061	46.740821	-92.099034	46 44' 26.96" N	92 5' 56.52" W	46 44.449' N	92 5.942' W
HB-17	-347309.9118	5236443.0514	46.740491	-92.095993	46 44' 25.77" N	92 5' 45.57" W	46 44.429' N	92 5.760' W
HB-19	-347144.9963	5236443.0514	46.740699	-92.093875	46 44' 26.52" N	92 5' 37.94" W	46 44.442' N	92 5.632' W
HB-20	-347598.4204	5236371.6340	46.739496	-92.099568	46 44' 22.19" N	92 5' 58.44" W	46 44.370' N	92 5.974' W
HB-21	-347351.1408	5236371.6408	46.739808	-92.096391	46 44' 23.31" N	92 5' 47.00" W	46 44.389' N	92 5.783' W
HB-22	-347186.2252	5236371.6408	46.740016	-92.094273	46 44' 24.06" N	92 5' 39.38" W	46 44.401' N	92 5.656' W
HB-23	-347103.7674	5236371.6408	46.740120	-92.093214	46 44' 24.43" N	92 5' 35.57" W	46 44.407' N	92 5.593' W
HB-24	-347358.1379	5236296.4903	46.739136	-92.096343	46 44' 20.89" N	92 5' 46.83" W	46 44.348' N	92 5.781' W
HB-25	-347227.4540	5236300.2302	46.739333	-92.094672	46 44' 21.60" N	92 5' 40.81" W	46 44.360' N	92 5.680' W
HB-27	-347062.5385	5236300.2302	46.739541	-92.092553	46 44' 22.35" N	92 5' 33.19" W	46 44.372' N	92 5.553' W
HB-28	-347598.4205	5236228.8128	46.738235	-92.099306	46 44' 17.65" N	92 5' 57.50" W	46 44.294' N	92 5.958' W
HB-29	-347352.4857	5236182.2267	46.738134	-92.096061	46 44' 17.28" N	92 5' 45.82" W	46 44.288' N	92 5.764' W
HB-30	-347186.2252	5236228.8196	46.738755	-92.094011	46 44' 19.52" N	92 5' 38.44" W	46 44.325' N	92 5.641' W
HB-31	-347103.7674	5236228.8196	46.738859	-92.092952	46 44' 19.89" N	92 5' 34.62" W	46 44.331' N	92 5.577' W
Area 2								
HB-18	-346727.0139	5235804.3067	46.735585	-92.087334	46 44' 8.11" N	92 5' 14.40" W	46 44.135' N	92 5.240' W
HB-26	-346502.5485	5235604.6043	46.734104	-92.084085	46 44' 2.77" N	92 5' 2.70" W	46 44.046' N	92 5.045' W
HB-32	-347006.9334	5236223.6516	46.738935	-92.091699	46 44' 20.17" N	92 5' 30.11" W	46 44.336' N	92 5.502' W
HB-33	-346893.7277	5236223.6449	46.739078	-92.090245	46 44' 20.68" N	92 5' 24.88" W	46 44.345' N	92 5.415' W
HB-34	-346950.2840	5236125.6863	46.738141	-92.090791	46 44' 17.31" N	92 5' 26.84" W	46 44.288' N	92 5.447' W
HB-35	-346837.2644	5236125.6930	46.738284	-92.089340	46 44' 17.82" N	92 5' 21.62" W	46 44.297' N	92 5.360' W
HB-36	-346893.7277	5236027.7278	46.737348	-92.089885	46 44' 14.45" N	92 5' 23.58" W	46 44.241' N	92 5.393' W
HB-37	-346780.7080	5236027.7345	46.737490	-92.088434	46 44' 14.97" N	92 5' 18.36" W	46 44.249' N	92 5.306' W
HB-38	-346817.1575	5235886.6260	46.736198	-92.088643	46 44' 10.31" N	92 5' 19.11" W	46 44.172' N	92 5.319' W
HB-39	-346724.1517	5235929.7759	46.736696	-92.087528	46 44' 12.11" N	92 5' 15.09" W	46 44.202' N	92 5.251' W
HB-40	-346667.5026	5235831.8107	46.735903	-92.086620	46 44' 9.25" N	92 5' 11.83" W	46 44.154' N	92 5.197' W
HB-41	-346611.0390	5235733.8588	46.735109	-92.085716	46 44' 6.39" N	92 5' 8.57" W	46 44.106' N	92 5.143' W
HB-42	-346554.4827	5235635.9003	46.734315	-92.084810	46 44' 3.53" N	92 5' 5.31" W	46 44.059' N	92 5.088' W
HB-43	-346479.1533	5235558.3728	46.733725	-92.083700	46 44' 1.41" N	92 5' 1.32" W	46 44.023' N	92 5.022' W
HB-44	-346636.0529	5235710.7392	46.734873	-92.085995	46 44' 5.54" N	92 5' 9.58" W	46 44.092' N	92 5.159' W
HB-45	-346587.9986	5235636.2926	46.734276	-92.085241	46 44' 3.39" N	92 5' 6.86" W	46 44.057' N	92 5.114' W

Notes:
D.d - Decimal Degrees
D M S - Degree, Minute, Second
D M.m - Degree, Decimal Minutes
ID - Identification
NAD - North American Datum
UTM - Universal Transverse Mercator

Table 2
List of Anticipated Sample Locations
Howard's Bay Site
Superior, Douglas County, Wisconsin

Location ID	Sample Interval	Sample ID	Analysis
Area 1			
HB-01	0-6"	HB10-1-1-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-1-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-1-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-1-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-02	0-6"	HB10-1-2-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-2-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-2-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-2-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-03	0-6"	HB10-1-3-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-3-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-3-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-3-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-04	0-6"	HB10-1-4-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-4-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-4-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-4-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-05	0-6"	HB10-11-5-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-5-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-5-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-5-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-06	0-6"	HB10-1-6-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-6-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-6-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-6-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-07	0-6"	HB10-1-7-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-7-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-7-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-7-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-08	0-6"	HB10-1-8-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-8-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-8-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-8-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-09	0-6"	HB10-1-9-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-9-051	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-9-03	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-9-05	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
HB-10	0-6"	HB10-1-10-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-10-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-10-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-10-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-11	0-6"	HB10-1-11-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-11-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-11-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-11-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO

Table 2
List of Anticipated Sample Locations
Howard's Bay Site
Superior, Douglas County, Wisconsin

Location ID	Sample Interval	Sample ID	Analysis
HB-12	0-6"	HB10-1-12-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-12-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-12-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-12-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-13	0-6"	HB10-1-13-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-13-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-13-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-13-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-14	0-6"	HB10-1-14-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-14-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-14-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-14-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-15	0-6"	HB10-1-15-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-15-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-15-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-15-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-16	0-6"	HB10-1-16-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-16-051	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-16-03	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-16-05	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
HB-17	0-6"	HB10-1-17-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-17-051	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-17-03	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-17-05	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
HB-19	0-6"	HB10-1-19-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-19-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-19-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-19-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-20	0-6"	HB10-1-20-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-20-051	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-20-03	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-20-05	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO

Table 2
List of Anticipated Sample Locations
Howard's Bay Site
Superior, Douglas County, Wisconsin

Location ID	Sample Interval	Sample ID	Analysis
HB-21	0-6"	HB10-1-21-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-21-051	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-21-03	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-21-05	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
HB-22	0-6"	HB10-1-22-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-22-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-22-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-22-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-23	0-6"	HB10-1-23-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-23-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-23-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-23-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-24	0-6"	HB10-1-24-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-24-051	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-24-03	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-24-05	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
HB-25	0-6"	HB10-1-25-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-25-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-25-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-25-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-27	0-6"	HB10-1-27-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-27-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-27-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-27-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-28	0-6"	HB10-1-28-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-28-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-28-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-28-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-29	0-6"	HB10-1-29-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-29-051	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-29-03	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-29-05	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
HB-30	0-6"	HB10-1-30-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-30-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-30-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-30-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-31	0-6"	HB10-1-31-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-31-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-31-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-31-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO

Table 2
List of Anticipated Sample Locations
Howard's Bay Site
Superior, Douglas County, Wisconsin

Location ID	Sample Interval	Sample ID	Analysis
Area 2			
HB-18	0-6"	HB10-1-18-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-18-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-18-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-18-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-26	0-6"	HB10-1-26-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-1-26-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-1-26-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-1-26-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-32	0-6"	HB10-2-32-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-32-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-32-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-32-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-33	0-6"	HB10-2-33-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-33-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-33-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-33-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-34	0-6"	HB10-2-34-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-34-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-34-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-34-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-35	0-6"	HB10-2-35-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-35-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-35-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-35-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-36	0-6"	HB10-2-35-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-36-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-36-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-36-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-37	0-6"	HB10-2-37-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-37-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-37-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-37-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-38	0-6"	HB10-2-38-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-38-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-38-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-38-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-39	0-6"	HB10-2-39-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-39-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-39-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-39-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-40	0-6"	HB10-2-40-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-40-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-40-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-40-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-41	0-6"	HB10-2-41-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-41-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-41-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-41-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO

Table 2
List of Anticipated Sample Locations
Howard's Bay Site
Superior, Douglas County, Wisconsin

Location ID	Sample Interval	Sample ID	Analysis
HB-42	0-6"	HB10-2-42-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-42-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-42-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-42-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-43	0-6"	HB10-2-43-005	TAL Metals, Mercury, PAH (34 list), Tri-butyl tin, TCL Pesticides, PCB Aroclor, AVS/SEM, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-43-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-43-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-43-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-44	0-6"	HB10-2-44-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-44-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-44-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-44-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
HB-45	0-6"	HB10-2-45-005	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	6-12"	HB10-2-45-051	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	1-3'	HB10-2-45-03	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO
	3-5'	HB10-2-45-05	TAL Metals, Mercury, PAH (17 list), Tri-butyl tin, TCL Pesticides, TOC, Grain Size, TPH: DRO/ORO

Notes:
AVS/SEM - Acid Volatile Sulfide/Simultaneously Extracted Metal
DRO - Diesel Range Organic
ORO - Oil Range Organic
PAH - Polycyclic Aromatic Hydrocarbon
PCB - Polychlorinated Biphenyl
TAL - Target Analyte List
TCL - Target Compound List
TOC - Total Organic Carbon
TPH - Total Petroleum Hydrocarbon

Table 3
List of Anticipated Sample Quantities and Analytical Parameters for Laboratory Samples Collected
Howard's Bay Site
Superior, Douglas County, Wisconsin

Sample Matrix	Laboratory Parameters	Investigative			Duplicate			MS/MSD			Sample Total
		No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Area 1											
Sediment	TAL Metals	124	1	124	13	1	13	25	1	25	137
	Mercury	124	1	124	13	1	13	25	1	25	137
	PAH (34 List)	40	1	40	5	1	5	8	1	8	45
	PAH (17 List)	84	1	84	14	1	14	17	1	17	98
	TPH DRO	124	1	124	13	1	13	25	1	25	137
	TPH ORO	124	1	124	13	1	13	25	1	25	137
	TOC	124	1	124	13	1	13	25	1	25	137
	Grain Size	124	1	124	13	1	13	0	0	0	137
	TCL Pesticide	124	1	124	13	1	13	25	1	25	137
	Tri-butyl tin	124	1	124	13	1	13	25	1	25	137
	PCB Aroclor	40	1	40	5	1	5	8	1	8	45
	AVS/SEM	40	1	40	5	1	5	8	1	8	45
Area 2											
Sediment	TAL Metals	56	1	56	6	1	6	12	1	12	62
	Mercury	56	1	56	6	1	6	12	1	12	62
	PAH (34 List)	8	1	8	5	1	5	2	1	2	13
	PAH (17 List)	48	1	48	14	1	14	10	1	10	62
	TPH DRO	56	1	56	6	1	6	12	1	12	62
	TPH ORO	56	1	56	6	1	6	12	1	12	62
	TOC	56	1	56	6	1	6	12	1	12	62
	Grain Size	56	1	56	6	1	6	0	0	0	62
	TCL Pesticide	8	1	8	1	1	1	12	1	12	9
	Tri-butyl tin	8	1	8	1	1	1	12	1	12	9
	PCB Aroclor	8	1	8	5	1	5	2	1	2	13
	AVS/SEM	8	1	8	5	1	5	2	1	2	13

Notes:
% - percent
AVS/SEM - Acid Volatile Sulfide/Simultaneously Extracted Metal
DRO - Diesel Range Organic
Freq. - Frequency
MS/MSD - Matrix Spike/Matrix Spike Duplicate
No. - Number
ORO - Oil Range Organics
PAH - Polycyclic Aromatic Hydrocarbon
PCB - Polychlorinated Biphenyl
TAL - Target Analyte List
TCL - Target Compound List
TOC - Total Organic Carbon
TPH - Total Petroleum Hydrocarbon

Sample total does not include MS/MSD samples

Table 4
Laboratory Sampling Information
Howard's Bay Site
Superior, Douglas County, Wisconsin

No. of Samples	Matrix	Analytical Parameter	Applicable Screening Criteria	Laboratory	Lab SOP Number
199	Sediment	TAL Metals	[1]Level I/II SQTs-St Louis River AOC [2]U.S. EPA/USGS/GLNPO SQGs	U.S. EPA CLP	ILM 05.4
199	Sediment	Mercury			ILM 05.4
58	Sediment	PAH (34 List)			SOM 01.2
160	Sediment	PAH (17 List)			SOM 01.2
198	Sediment	TCL Pesticide			SOM 01.2
58	Sediment	PCB Aroclor			SOM 01.2
58	Sediment	AVS/SEM	Not Applicable		ILM 05.4
199	Sediment	TPH DRO	Not Applicable	TriMatrix	GR-09-123
199	Sediment	TPH ORO	Not Applicable		GR-03-122
199	Sediment	TOC	Not Applicable	CAS	GEN-ASTM
199	Sediment	Grain Size	Not Applicable	Test America	BR-GT-006
199	Sediment	Tri-butyl tin	Not Applicable		BR-EX-001

Notes:
% - percent
AOC - Area of Concern
AVS/SEM - Acid Volatile Sulfide/Simultaneously Extracted Metal
CLP - Contract Laboratory Program
DRO - Diesel Range Organic
GLNPO - Great Lakes National Program Office
MS/MSD - Matrix Spike/Matrix Spike Duplicate
No. - Number
ORO - Oil Range Organics
PAH - Polycyclic Aromatic Hydrocarbon
PCB - Polychlorinated Biphenyl
SOP - Standard Operating Procedure
SQG - Sediment Quality Guideline
SQT - Sediment Quality Target
TAL - Target Analyte List
TCL - Target Compound List
TOC - Total Organic Carbon
TPH - Total Petroleum Hydrocarbon
U.S. EPA - United States Environmental Protection Agency
USGS - United States Geological Survey
WESTON - Weston Solutions, Inc

Sample total does not include MS/MSD samples

[1] *Evaluation of Numerical SQTs for the St Louis River AOC* (DOI: 10.1007/s00244-002-1155-x)

[2] *Prediction of Sediment Toxicity using Consensus-Based Freshwater SQGs* (EPA 905/R-00/007)

Table 5
Containers, Preservatives, and Holding Times
Howard's Bay
Superior, Douglas County, WI

Sample Matrix	Analysis	Number of Containers	Type of Containers	Preservatives	Technical Holding Time[1]
Sediment	TAL Metals[2]	1	8 oz glass jar	Cool to 4°C	6 months
	Mercury[2]	1	8 oz glass jar	Cool to 4°C	6 months
	PAH (34 List)	1	8 oz glass jar	Cool to 4°C	14 day extraction 40 day analysis
	PAH (17 List)	1	8 oz glass jar	Cool to 4°C	14 day extraction 40 day analysis
	TPH DRO[3]	1	8 oz glass jar	Cool to 4°C	14 day extraction 40 day analysis
	TPH ORO[3]	1	8 oz glass jar	Cool to 4°C	14 day extraction 40 day analysis
	TOC	1	4 oz glass jar	Cool to 4°C	28 days
	Grain Size	1	16 oz glass jar or zip lock bag	Cool to 4°C	Not Applicable
	TCL Pesticide	1	8 oz glass jar	Cool to 4°C	6 months
	Tri-butyl tin	1	4 oz glass jar	Cool to 4°C	14 day extraction 40 day analysis
	PCB Aroclor	1	8 oz glass jar	Cool to 4°C	14 day extraction 40 day analysis
	AVS/SEM	1	8 oz glass jar	Cool to 4°C	14 days

Notes:
NA - Not Applicable
oz - Ounce
PAH - Polyaromatic Hydrocarbons
PCB - Polychlorinated Biphenyls
AVS/SEM - Acid Volatile Sulfide/Simultaneously Extracted Metal
DRO - Diesel Range Organic
ORO - Oil Range Organics
PAH - Polycyclic Aromatic Hydrocarbon
PCB - Polychlorinated Biphenyl
TAL - Target Analyte List
TCL - Target Compound List
TOC - Total Organic Carbon
TPH - Total Petroleum Hydrocarbon

[1] All holding times are from the date of sample collection.

[2, 3] Soil for these analyses may be collected in the same 8-oz glass jar.

Table 6
PAH List 17 and 34 Target Compounds
Howard's Bay
Superior, Douglas County, WI

Compound	SIM CRQL (µg/kg)	Modified Analysis
1-Methylnaphthalene	3.3	PAH 34 SOM01.2
2-Methiylnaphthalene	3.3	PAH 17 and 34 SOM01.2
Naphthalene	3.3	PAH 17 and 34 SOM01.2
C1-Napthalenes	3.3	PAH 34 SOM01.2
C2-Naphthalenes	3.3	PAH 34 SOM01.2
C3-Naphthalenes	3.3	PAH 34 SOM01.2
C4-Naphthalenes	3.3	PAH 34 SOM01.2
Acenaphthylene	3.3	PAH 17 and 34 SOM01.2
Acenapthene	3.3	PAH 17 and 34 SOM01.2
Fluorene	3.3	PAH 17 and 34 SOM01.2
C1 Fluorenes	3.3	PAH 34 SOM01.2
C2 Fluorenes	3.3	PAH 34 SOM01.2
C3 Fluorenes	3.3	PAH 34 SOM01.2
Phenanthrene	3.3	PAH 17 and 34 SOM01.2
Anthracene	3.3	PAH 17 and 34 SOM01.2
C1-Phenanthrenes/Anthracenes	3.3	PAH 34 SOM01.2
C2-Phenanthrenes/Anthracenes	3.3	PAH 34 SOM01.2
C3-Phenanthrenes/Anthracenes	3.3	PAH 34 SOM01.2
C4-Phenanthrenes/Anthracenes	3.3	PAH 34 SOM01.2
Fluoranthene	3.3	PAH 17 and 34 SOM01.2
Pyrene	3.3	PAH 17 and 34 SOM01.2
C1-Fluoranthenes/Pyrenes	3.3	PAH 34 SOM01.2
C2-Fluoranthenes/Pyrenes	3.3	PAH 34 SOM01.2
C3-Fluoranthenes/Pyrenes	3.3	PAH 34 SOM01.2
Benzo (a) anthracene	3.3	PAH 17 and 34 SOM01.2
Chrysene	3.3	PAH 17 and 34 SOM01.2
C1 Chrysenes	3.3	PAH 34 SOM01.2
C2 Chrysenes	3.3	PAH 34 SOM01.2
C3 Chrysenes	3.3	PAH 34 SOM01.2
C4 Chrysenes	3.3	PAH 34 SOM01.2
Benzo (b) fluoranthene	3.3	PAH 17 and 34 SOM01.2
Benzo (k) fluoranthene	3.3	PAH 17 and 34 SOM01.2
Benzo (e) pyrene	3.3	PAH 34 SOM01.2
Benzo (a) pyrene	3.3	PAH 17 and 34 SOM01.2
Perylene	3.3	PAH 34 SOM01.2
Indeno (1,2,3-cd) pyrene	3.3	PAH 17 and 34 SOM01.2
Dibenzo (a,h) anthracene	3.3	PAH 17 and 34 SOM01.2
Benzo (g,h,i) perylene	3.3	PAH 17 and 34 SOM01.2
Total PAHs	3.3	PAH 17 and 34 SOM01.2

Notes:
PAH - Polycylic Aromatic Hydrocarbon
SIM CRQL- Select Ion Monitoring Contract Required Quantitation Limits
ug/kg - micro gram per kilogram

APPENDIX A-A

Visual Sampling Plan Reports

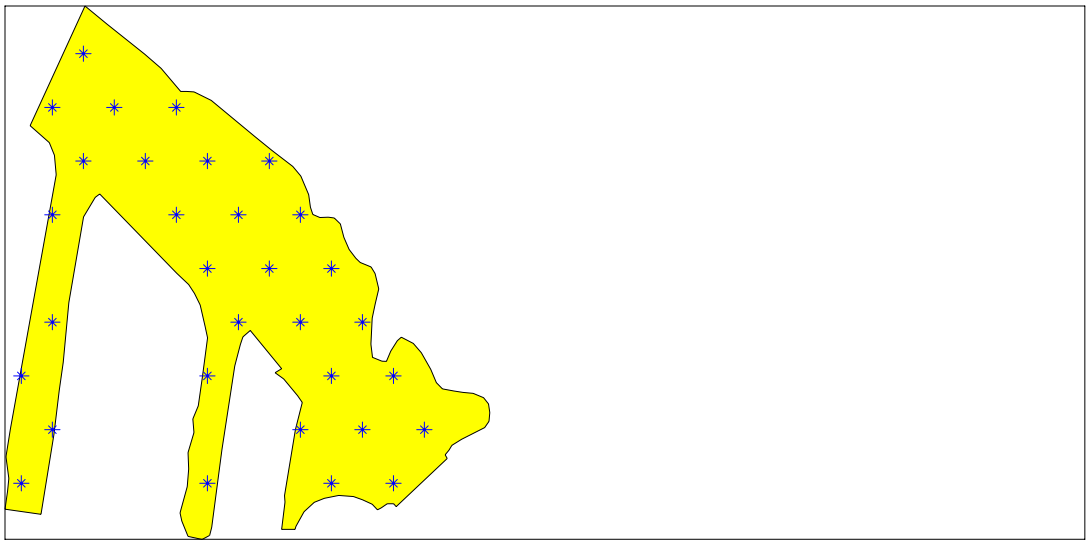
Systematic sampling locations for detecting an area of elevated values (hot spot)

This report summarizes the sampling design used, associated statistical assumptions, as well as general guidelines for conducting post-sampling data analysis. Sampling plan components presented here include how many sampling locations to choose and where within the sampling area to collect those samples. The type of medium to sample (i.e., soil, groundwater, etc.) and how to analyze the samples (in-situ, fixed laboratory, etc.) are addressed in other sections of the sampling plan.

The following table summarizes the sampling design developed. A figure that shows sampling locations in the field and a table that lists sampling location coordinates are also provided below.

SUMMARY OF SAMPLING DESIGN	
Primary Objective of Design	Detect the presence of a hot spot that has a specified size and shape
Type of Sampling Design	Hot spot
Sample Placement (Location) in the Field	Systematic (Hot Spot) with a random start location
Formula for calculating number of sampling locations	Singer and Wickman algorithm
Calculated total number of samples	31
Type of samples	Point Samples
Number of samples on map [a]	31
Number of selected sample areas [b]	1
Specified sampling area [c]	176651.01 m^2
Grid pattern	Triangular
Size of grid / Area of grid [d]	270.531 feet / 63381.9 ft^2
Total cost of sampling [e]	$16,500.00

[a] This number may differ from the calculated number because of 1) grid edge effects, 2) adding judgment samples, or 3) selecting or unselecting sample areas.
[b] The number of selected sample areas is the number of colored areas on the map of the site. These sample areas contain the locations where samples are collected.
[c] The sampling area is the total surface area of the selected colored sample areas on the map of the site.
[d] Size of grid / Area of grid gives the linear and square dimensions of the grid spacing used to systematically place samples.
[e] Including measurement analyses and fixed overhead costs. See the Cost of Sampling section for an explanation of the costs presented here.



Area: Area 1					
X Coord	**Y Coord**	**Label**	**Value**	**Type**	**Historical**
-347598.6079	5236228.8263			Hotspot	
-347351.2343	5236228.8263			Hotspot	
-347186.3185	5236228.8263			Hotspot	
-347103.8606	5236228.8263			Hotspot	
-347557.3789	5236300.2369			Hotspot	
-347227.5474	5236300.2369			Hotspot	
-347145.0896	5236300.2369			Hotspot	
-347062.6317	5236300.2369			Hotspot	
-347598.6079	5236371.6475			Hotspot	
-347351.2343	5236371.6475			Hotspot	
-347186.3185	5236371.6475			Hotspot	
-347103.8606	5236371.6475			Hotspot	
-347557.3789	5236443.0581			Hotspot	
-347310.0053	5236443.0581			Hotspot	
-347227.5474	5236443.0581			Hotspot	
-347145.0896	5236443.0581			Hotspot	
-347351.2343	5236514.4687			Hotspot	
-347268.7764	5236514.4687			Hotspot	
-347186.3185	5236514.4687			Hotspot	
-347557.3789	5236585.8793			Hotspot	
-347392.4632	5236585.8793			Hotspot	
-347310.0053	5236585.8793			Hotspot	
-347227.5474	5236585.8793			Hotspot	
-347516.1500	5236657.2900			Hotspot	
-347433.6921	5236657.2900			Hotspot	

-347351.2343	5236657.2900			Hotspot	
-347268.7764	5236657.2900			Hotspot	
-347557.3789	5236728.7006			Hotspot	
-347474.9211	5236728.7006			Hotspot	
-347392.4632	5236728.7006			Hotspot	
-347516.1500	5236800.1112			Hotspot	

Primary Sampling Objective
The primary purpose of sampling at this site is to detect "hot spots" (local areas of elevated concentration) of a given size and shape with a specified probability, 1-β.

Selected Sampling Approach
This sampling approach requires systematic grid sampling with a random start. If a systematic grid is not used, the probability of detecting a hot spot of a given size and shape will be different than desired or calculated.

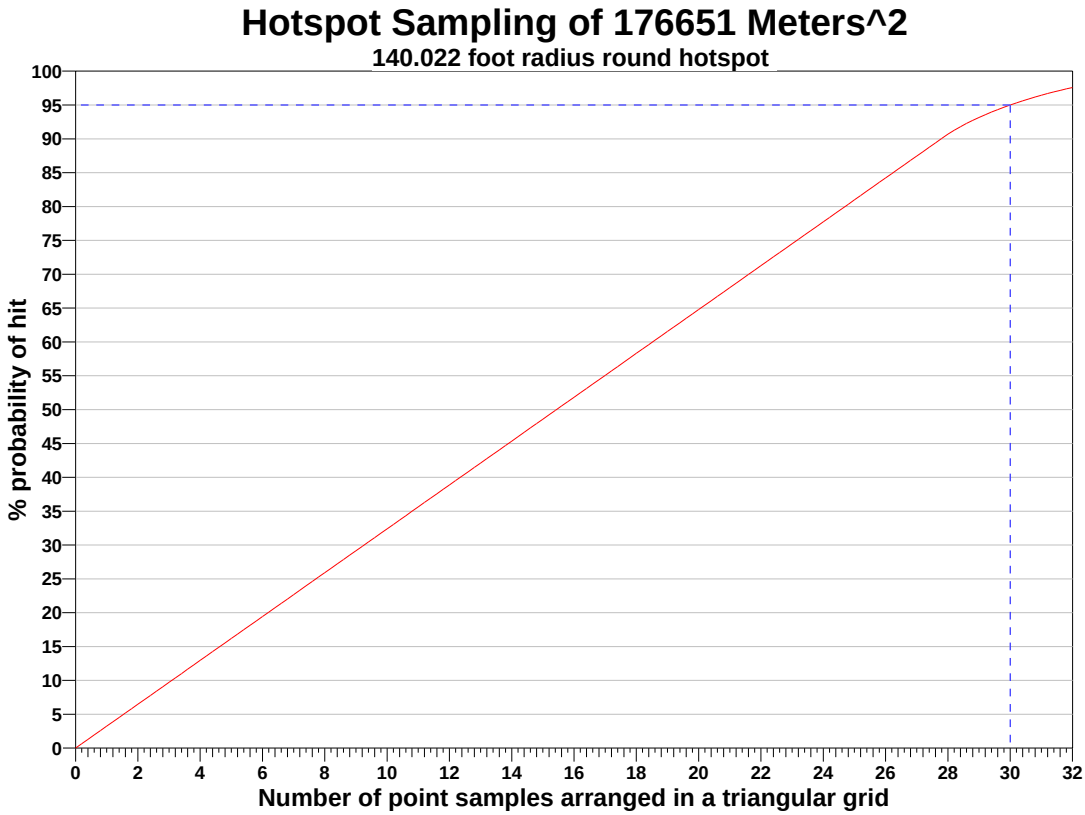
Number of Total Samples: Calculation Equation and Inputs
The algorithm used to calculate the grid size (and hence, the number of samples) is based on work by Singer and Wickman for locating geologic deposits [see Singer and Wickman (1969) and Hassig et al. (2004) for details]. Inputs to the algorithm include the size, shape, and orientation of a hot spot of interest, an acceptable probability of finding a hot spot, the desired type of sampling grid, and the sampling budget. For this design, the smallest hot spot that could be detected was calculated based on the given grid size and other parameters.

The inputs to the algorithm that result in the smallest hot spot that could be detected are:

Parameter	Description	Value
Inputs		
Samples	Number of samples specified by user	30
1-β	Probability of detection	95%
Grid Type	Grid pattern (Square, Triangular or Rectangular)	Triangular
Grid Size	Spacing between samples	270.531 feet
Grid Area	Area represented by one grid	63381.9 ft^2
Sample Type	Point samples or square cells	Points
Hot Spot Shape	Hot spot height to width ratio	1
Angle	Angle of orientation between hot spot and grid	Random
Sampling Area	Total area to sample	176651.01 m^2
Outputs		
Hot Spot Size	Length of hot spot semi-major axis	140.022 feet
Hot Spot Area [a]	Area of hot spot (Length^2 * Shape * π)	61594.3 ft^2

[a] Length of semi-major axis is used by Singer-Wickman algorithm. Hot spot area is provided for informational purposes.

The following graph shows the relationship between the number of samples and the probability of finding the hot spot. The dashed blue line shows the actual number of samples for this design (which may differ from the optimum number of samples because of edge effects).



Assumptions that Underlie the VSP Locating a Hot Spot Design Method

1. The shape of the hot spot of concern is circular or elliptical.
2. The level of contamination that defines a hot spot is well defined.
3. The location of the hot spot is unknown, and if a hot spot is present, all locations within the sampling area are equally likely to contain the hot spot.
4. Samples are taken on a square, rectangular or triangular grid pattern.
5. Each sample is collected, handled, measured or inspected using approved methods that yield unbiased and sufficiently precise measurements.
6. A very small proportion of the surface being studied will be sampled (the sample is much smaller than the hot spot of interest).
7. Sample locations are independent of the measurement process.
8. The systematic grid is placed at a randomly determined starting place to cover the surface area of interest.
9. There are no classification errors (if a hot spot is sampled, it is not mistakenly overlooked or an area is not mistakenly identified as a hot spot).

Sensitivity Analysis

The sensitivity of the calculation of number of samples was explored by varying the probability of hit (%), hot spot shape (height to width ratio) and hot spot size (length of semi-major axis). The following table shows the results of this analysis.

Number of Samples				
		Size=70.0108	**Size=140.022**	**Size=210.033**
1-β=90	**Shp=0.8**	146	37	17
	Shp=0.9	126	32	14
	Shp=1	112	28	13
1-β=95	**Shp=0.8**	160	40	18
	Shp=0.9	137	35	16
	Shp=1	121	31	14

1-β=100	**Shp=0.8**	194	49	22
	Shp=0.9	167	42	19
	Shp=1	149	38	17

1-β = Probability of Hit (%)
Shp = Hot Spot Shape (Height to Width Ratio)
Size = Hot Spot Size (Length of Semi-major Axis)

Cost of Sampling
The total cost of the completed sampling program depends on several cost inputs, some of which are fixed, and others that are based on the number of samples collected and measured. Based on the numbers of samples determined above, the estimated total cost of sampling and analysis at this site is $16,500.00, which averages out to a per sample cost of $532.26. The following table summarizes the inputs and resulting cost estimates.

COST INFORMATION			
Cost Details	**Per Analysis**	**Per Sample**	**31 Samples**
Field collection costs		$100.00	$3,100.00
Analytical costs	$400.00	$400.00	$12,400.00
Sum of Field & Analytical costs		**$500.00**	**$15,500.00**
Fixed planning and validation costs			$1,000.00
Total cost			**$16,500.00**

Recommended Data Analysis Activities
Post data collection activities generally follow those outlined in EPA's Guidance for Data Quality Assessment (EPA, 2006). The data analysts will become familiar with the context of the problem and goals for data collection and assessment. The data will be verified and validated before being subjected to statistical or other analyses. Graphical and analytical tools will be used to verify to the extent possible the assumptions of any statistical analyses that are performed as well as to achieve a general understanding of the data. The data will be assessed to determine whether they are adequate in both quality and quantity to support the primary objective of sampling.

A map of the actual sample locations will be generated so that the sampling plan and the field implementation may be compared. Deviations from planned sample locations due to topographic, vegetative, or other features will be noted. Their impacts will be qualitatively assessed. If a hot spot is discovered, additional sampling may be performed to determine its size and shape, in which case, the initial assumptions of the sampling design may then be assessed and/or reconsidered.

References
EPA 2006. *Data Quality Assessment: Statistical Methods for Practitioners EPA QA/G-9S*, EPA/240/B-06/003, U.S. Environmental Protection Agency, Office of Environmental Information, Washington DC.

Gilbert, R.O. 1987. *Statistical Methods for Environmental Pollution Monitoring*. Wiley & Sons, Inc., New York, NY.

Hassig, N.L., J.E. Wilson, R.O. Gilbert and B.A. Pulsipher. 2004. *Visual Sample Plan Version 3.0 User's Guide*. PNNL-14970. Pacific Northwest National Laboratory, Richland, WA, December 2004.

Singer, D.A. and J.E. Wickman. 1969. *Probability Tables for Locating Elliptical Targets with Square, Rectangular, and Hexagonal Point Nets*. Pennsylvania State University, University Park, Pennsylvania. Special Publication 1-69.

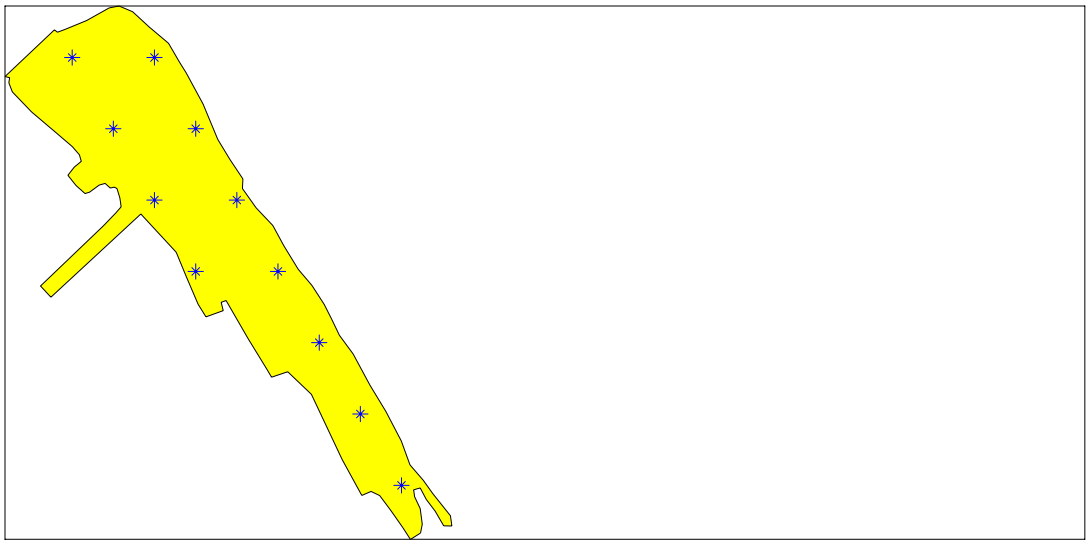
Systematic sampling locations for detecting an area of elevated values (hot spot)

This report summarizes the sampling design used, associated statistical assumptions, as well as general guidelines for conducting post-sampling data analysis. Sampling plan components presented here include how many sampling locations to choose and where within the sampling area to collect those samples. The type of medium to sample (i.e., soil, groundwater, etc.) and how to analyze the samples (in-situ, fixed laboratory, etc.) are addressed in other sections of the sampling plan.

The following table summarizes the sampling design developed. A figure that shows sampling locations in the field and a table that lists sampling location coordinates are also provided below.

SUMMARY OF SAMPLING DESIGN	
Primary Objective of Design	Detect the presence of a hot spot that has a specified size and shape
Type of Sampling Design	Hot spot
Sample Placement (Location) in the Field	Systematic (Hot Spot) with a random start location
Formula for calculating number of sampling locations	Singer and Wickman algorithm
Calculated total number of samples	11
Type of samples	Point Samples
Number of samples on map [a]	11
Number of selected sample areas [b]	1
Specified sampling area [c]	110803.65 m^2
Grid pattern	Triangular
Size of grid / Area of grid [d]	371.105 feet / 119268 ft^2
Total cost of sampling [e]	$6,500.00

[a] This number may differ from the calculated number because of 1) grid edge effects, 2) adding judgment samples, or 3) selecting or unselecting sample areas.
[b] The number of selected sample areas is the number of colored areas on the map of the site. These sample areas contain the locations where samples are collected.
[c] The sampling area is the total surface area of the selected colored sample areas on the map of the site.
[d] Size of grid / Area of grid gives the linear and square dimensions of the grid spacing used to systematically place samples.
[e] Including measurement analyses and fixed overhead costs. See the Cost of Sampling section for an explanation of the costs presented here.



Area: Area 1					
X Coord	**Y Coord**	**Label**	**Value**	**Type**	**Historical**
-346554.5753	5235635.9070			Hotspot	
-346611.1317	5235733.8655			Hotspot	
-346667.6881	5235831.8241			Hotspot	
-346837.3573	5235929.7826			Hotspot	
-346724.2445	5235929.7826			Hotspot	
-346893.9137	5236027.7412			Hotspot	
-346780.8009	5236027.7412			Hotspot	
-346950.4701	5236125.6997			Hotspot	
-346837.3573	5236125.6997			Hotspot	
-347007.0265	5236223.6583			Hotspot	
-346893.9137	5236223.6583			Hotspot	

Primary Sampling Objective
The primary purpose of sampling at this site is to detect "hot spots" (local areas of elevated concentration) of a given size and shape with a specified probability, 1-β.

Selected Sampling Approach
This sampling approach requires systematic grid sampling with a random start. If a systematic grid is not used, the probability of detecting a hot spot of a given size and shape will be different than desired or calculated.

Number of Total Samples: Calculation Equation and Inputs
The algorithm used to calculate the grid size (and hence, the number of samples) is based on work by Singer and Wickman for locating geologic deposits [see Singer and Wickman (1969) and Hassig et al. (2004) for details]. Inputs to the algorithm include the size, shape, and orientation of a hot spot of interest, an acceptable probability of finding a hot spot, the desired type of sampling grid, and the sampling budget. For this design, the smallest hot spot that could be detected was calculated based on the given grid size and other parameters.

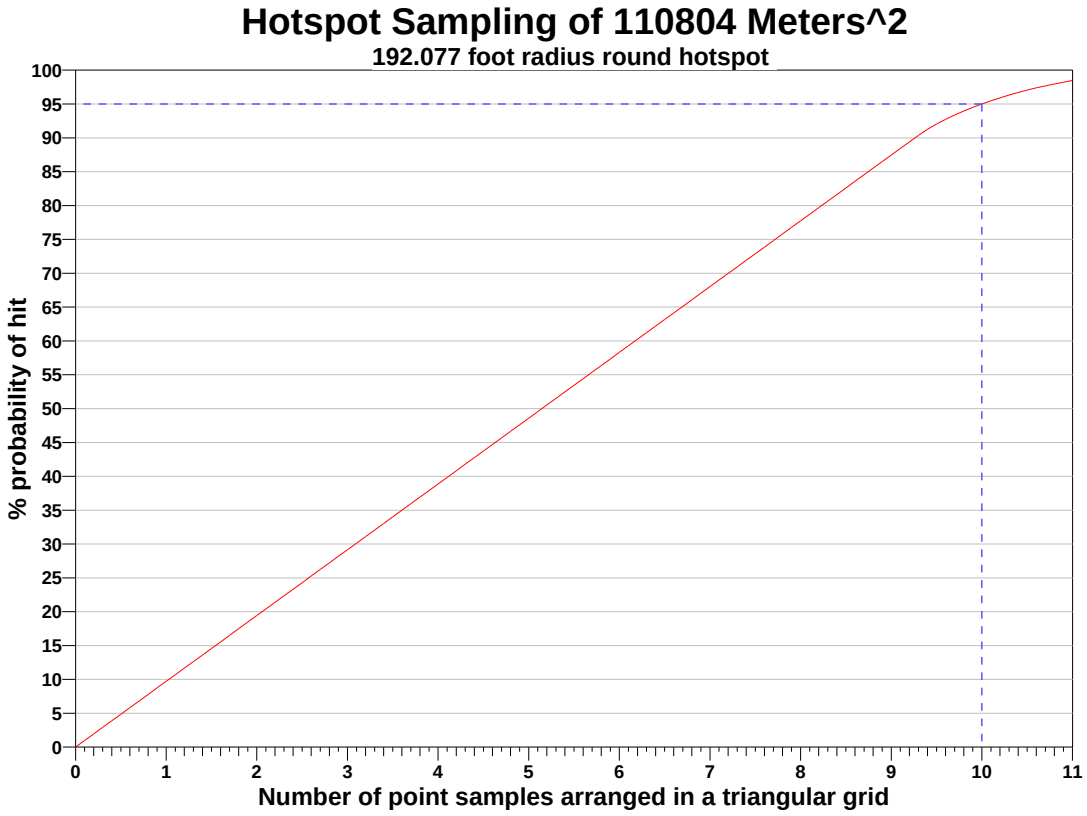
The inputs to the algorithm that result in the smallest hot spot that could be detected are:

Parameter	Description	Value
Inputs		

Samples	Number of samples specified by user	10
1-β	Probability of detection	95%
Grid Type	Grid pattern (Square, Triangular or Rectangular)	Triangular
Grid Size	Spacing between samples	371.105 feet
Grid Area	Area represented by one grid	119268 ft^2
Sample Type	Point samples or square cells	Points
Hot Spot Shape	Hot spot height to width ratio	1
Angle	Angle of orientation between hot spot and grid	Random
Sampling Area	Total area to sample	110803.65 m^2
Outputs		
Hot Spot Size	Length of hot spot semi-major axis	192.077 feet
Hot Spot Area [a]	Area of hot spot (Length^2 * Shape * π)	115904 ft^2

[a] Length of semi-major axis is used by Singer-Wickman algorithm. Hot spot area is provided for informational purposes.

The following graph shows the relationship between the number of samples and the probability of finding the hot spot. The dashed blue line shows the actual number of samples for this design (which may differ from the optimum number of samples because of edge effects).



Assumptions that Underlie the VSP Locating a Hot Spot Design Method

1. The shape of the hot spot of concern is circular or elliptical.
2. The level of contamination that defines a hot spot is well defined.
3. The location of the hot spot is unknown, and if a hot spot is present, all locations within the sampling area are equally likely to contain the hot spot.
4. Samples are taken on a square, rectangular or triangular grid pattern.
5. Each sample is collected, handled, measured or inspected using approved methods that yield unbiased and

sufficiently precise measurements.

6. A very small proportion of the surface being studied will be sampled (the sample is much smaller than the hot spot of interest).
7. Sample locations are independent of the measurement process.
8. The systematic grid is placed at a randomly determined starting place to cover the surface area of interest.
9. There are no classification errors (if a hot spot is sampled, it is not mistakenly overlooked or an area is not mistakenly identified as a hot spot).

Sensitivity Analysis

The sensitivity of the calculation of number of samples was explored by varying the probability of hit (%), hot spot shape (height to width ratio) and hot spot size (length of semi-major axis). The following table shows the results of this analysis.

Number of Samples				
		Size=96.0384	**Size=192.077**	**Size=288.115**
1-β=90	**Shp=0.8**	49	13	6
	Shp=0.9	42	11	5
	Shp=1	38	10	5
1-β=95	**Shp=0.8**	54	14	6
	Shp=0.9	46	12	6
	Shp=1	41	11	5
1-β=100	**Shp=0.8**	65	17	8
	Shp=0.9	56	14	7
	Shp=1	50	13	6

1-β = Probability of Hit (%)
Shp = Hot Spot Shape (Height to Width Ratio)
Size = Hot Spot Size (Length of Semi-major Axis)

Cost of Sampling

The total cost of the completed sampling program depends on several cost inputs, some of which are fixed, and others that are based on the number of samples collected and measured. Based on the numbers of samples determined above, the estimated total cost of sampling and analysis at this site is $6,500.00, which averages out to a per sample cost of $590.91. The following table summarizes the inputs and resulting cost estimates.

COST INFORMATION			
Cost Details	**Per Analysis**	**Per Sample**	**11 Samples**
Field collection costs		$100.00	$1,100.00
Analytical costs	$400.00	$400.00	$4,400.00
Sum of Field & Analytical costs		**$500.00**	**$5,500.00**
Fixed planning and validation costs			$1,000.00
Total cost			**$6,500.00**

Recommended Data Analysis Activities

Post data collection activities generally follow those outlined in EPA's Guidance for Data Quality Assessment (EPA, 2006). The data analysts will become familiar with the context of the problem and goals for data collection and assessment. The data will be verified and validated before being subjected to statistical or other analyses. Graphical and analytical tools will be used to verify to the extent possible the assumptions of any statistical analyses that are performed as well as to achieve a general understanding of the data. The data will be assessed to determine whether they are adequate in both quality and quantity to support the primary objective of sampling.

A map of the actual sample locations will be generated so that the sampling plan and the field implementation may be

compared. Deviations from planned sample locations due to topographic, vegetative, or other features will be noted. Their impacts will be qualitatively assessed. If a hot spot is discovered, additional sampling may be performed to determine its size and shape, in which case, the initial assumptions of the sampling design may then be assessed and/or reconsidered.

References

EPA 2006. *Data Quality Assessment: Statistical Methods for Practitioners EPA QA/G-9S*, EPA/240/B-06/003, U.S. Environmental Protection Agency, Office of Environmental Information, Washington DC.

Gilbert, R.O. 1987. *Statistical Methods for Environmental Pollution Monitoring*. Wiley & Sons, Inc., New York, NY.

Hassig, N.L., J.E. Wilson, R.O. Gilbert and B.A. Pulsipher. 2004. *Visual Sample Plan Version 3.0 User's Guide*. PNNL-14970. Pacific Northwest National Laboratory, Richland, WA, December 2004.

Singer, D.A. and J.E. Wickman. 1969. *Probability Tables for Locating Elliptical Targets with Square, Rectangular, and Hexagonal Point Nets*. Pennsylvania State University, University Park, Pennsylvania. Special Publication 1-69.

APPENDIX A-B

Field Collection Sheet

Field Data Collection Form
Howard's Bay
Superior, Douglas County, Wisconsin

Sample Location ID: _____

Water Depth: _____ **Total Core Recovery (sediment depth)**: _____

Sample Date: _____ **Sample Time**: _____

Sample Collected By: _____

Sample Observations (color, texture, odor, etc)

Overall: _____

0 to 0.5 foot: _____

0.5 to 1 feet: _____

1 to 3 feet: _____

3 to 5 feet: _____

5 to 7 feet: _____

Sample Type:	PONAR	VIBRACORE	OTHER	
Analysis (all):	TAL Metals[1]	PAH (34 List) [1]	PCB Aroclor[1]	TPH DRO[2]
	TPH ORO[2]	Mercury[1]	Tri-butyl tin[2]	Grain Size[2]
	TCL Pesticides[1]	PAH (17 List)	AVS/SEM[1]	TOC[2]

[1]*CLP Lab* [2]*WESTON Procured Lab*

Field duplicate/replicate: YES / NO _____

Photos: YES / NO _____

Coordinates same as projected: YES / NO _____

If no – new coordinates: _____

Other Comments: _____

_____

APPENDIX A-C

U.S. EPA R/V Mudpuppy II Standard Operating Procedures

Standard Operating Procedure for Using the Standard & Petite Ponar and Peterson Grabs On Board the Research Vessel *Mudpuppy II*

MPXXX

Revision 0, July 2010

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
GREAT LAKES NATIONAL PROGRAM OFFICE
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

APPROVED BY:

USEPA Region V Health & Safety Manager

_____
(Signature and Date) – *as necessary*

GLNPO Health and Safety Team Leader

_____
(Signature and Date)

GLNPO Sediment Team Member

_____
(Signature and Date)

TABLE OF CONTENTS

Standard Operating Procedure for Using the Standard & Petite Ponar and Peterson Grabs On Board the Research Vessel (R/V) *Mudpuppy II*

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes procedures in using the Standard & Petite Ponar and Peterson grabs to obtain sediment grab samples on board the R/V *Mudpuppy II*.

This SOP should be used in conjunction with the Great Lakes National Program Office's Safety, Health, and Environmental Compliance, Appendix N, for the health and safety requirements as this takes precedence over the SOP requirements. Project participants also should refer to the project-specific quality assurance project plan (QAPP) for detailed sampling requirements.

2.0 INTRODUCTION

The Standard Ponar consists of a center pivot, tapered scooped edges, heavy-duty hinges, scoop, underlip, stainless steel screen, and a pinch-pin (Figure 1). It has a scoop volume of 8.2 liters, can hold 400 ounces, and a sampling area of 229 mm by 229 mm (9 inches by 9 inches). The maximum depth of collection is 3.2 inches.

Please reference the 1725-F10 Standard Ponar user manual for specifications on the unit.

Figure 1. Standard Ponar

The Peterson grab consists of a clamshell pivot, tapered scoop edges, and a safety pin lock (Figure 2). It has a scoop volume of 9.89 liters and a sampling area of 305 mm x 305 mm (12 inches by 12 inches). The maximum depth of collection is 5.5 inches. Operation requires winch and crane due to the working weight. The Petersen grab is used for sand, gravel, and/or clay sediments or for collecting large-volume samples.

Figure 2. Peterson Grab

The Petite Ponar consists of a center pivot for low-bottom disturbance with removable top screens, self-releasing pinch-pin, heavy duty hinges, and is designed for hand line operation especially since it is half the weight of a Standard Ponar (Figure 3). The maximum depth of collection is 2.75 inches.

Please reference the 1728-G30/G40 Petite Ponar user manual for specifications on the unit.

Figure 3. Petite Ponar

An image of all grabs on board the R/V *Mudpuppy II* is shown in Figure 4.

Figure 4. Petite Ponar, Standard Ponar, and Peterson Grabs *(left to right)*

3.0 EQUIPMENT AND SUPPLIES FOR OPERATION OF THE GRABS

- Winch (not needed for Petite Ponar)
- Winch mount (not needed for Petite Ponar)
- Stainless steel cable for Standard and Peterson grabs; standard line for Petite Ponar grab

4.0 STEPS TO OBTAIN SAMPLES USING THE GRABS

1. Record sample location using global positioning system (GPS).
2. Measure and record water depth.
3. Securely fasten the cable or rope to the grab.
4. Insert the pinch-pin (tripping device) into ponar. Pinch-pin has a spring wrapped around bolt. Hold ponar grab at the shackle to avoid getting fingers and hands pinched.
5. After inserting the pinch-pin into grab, lift the grab with the winch. Lifting the ponar will secure the pinch-pin in place.
6. Winch the grab into the water body until grab has reached sediment bottom. Pull in the line to trip the grab.
7. Record the latitude and longitude.

8. Trip the grab shut by allowing slack to the line. The pinch-pin will be released allowing for the jaws clamp shut, grabbing a sediment sample.
9. Winch the grab to the deck. Decant water from grab before placing grab into pan.
10. Empty sediment from grab by grabbing weight to open and let sediments out in pan. Insert the safety pin into the grab. Winch the grab and put the grab back into its place on the deck.
11. Follow sample collection, sample handling and preservation, safety and waste handling per QAPP and site safety plan.
12. Rinse the grabs after each use to avoid cross-contamination in samples.

5.0 QUALITY CONTROL AND QUALITY ASSURANCE

If the first attempt at sample collection is not successful then do not dump the sample overboard at the same exact location where collection occurred. Instead, dump the sample away from the original sample location.

6.0 PERSONAL PROTECTIVE EQUIPMENT FOR OPERATION OF GRABS

At a minimum, wear a life jacket, steel-toed boots, safety goggles, and rubber gloves or leather gloves to operate the grabs.

7.0 PERSONNEL QUALIFICATIONS

Personnel have an opportunity to learn how to use any of the ponar grabs at a sampling event during the sampling season. Training involves shadowing a trained sampler and taking samples under supervision of the trainer.

8.0 PREVENTATIVE MAINTENANCE

Please see the section entitled Maintenance in the 1725-F10 Standard Ponar and 1728-G30/G40 Petite Ponar user manuals for steps in maintaining ponars on board the R/V *Mudpuppy II*.

9.0 REFERENCES

http://www.wildco.com

Standard Operating Procedure for Using the Vibracoring System On Board the Research Vessel *Mudpuppy II*

MP103

Revision 0, July 2010

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

GREAT LAKES NATIONAL PROGRAM OFFICE
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

APPROVED BY:

USEPA Region V Health & Safety Manager

_____
(Signature and Date) – *as necessary*

GLNPO Health and Safety Team Leader

_____
(Signature and Date)

GLNPO Sediment Team Member

_____
(Signature and Date)

TABLE OF CONTENTS

Standard Operating Procedure for Using the Vibracoring System On Board the Research Vessel (R/V) *Mudpuppy II*

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes procedures in using the vibracoring system to obtain sediment cores on board the Research Vessel (R/V) *Mudpuppy II*.

This SOP should be used in conjunction with the Great Lakes National Program Office's Safety, Health, and Environmental Compliance, Appendix N, for the health and safety requirements as this takes precedence over the SOP requirements. Project participants also should refer to the project-specific quality assurance project plan for detailed sampling requirements.

2.0 INTRODUCTION

The vibracoring system consists of the vibracore head, core tube, underwater electrical cable coming from surface support platform to the vibracore head, and control box located between the underwater cable and the power source. The vibracore head has a core tube clamp and an internal vibrator motor. The vibracorer applies thousands of vibrations per minute to help penetrate the sediment. When the core tube is inserted in the core tube clamp, the vibracorer is lowered to one foot above the water body and then turned on. As soon as the core tube touches the sediment, the sediment and water interface to create a slurry due to the vibrations between the core tube and sediment. This eases the entry of the core tube into the sediment. The vibracorer for the R/V *Mudpuppy II*, Rossfelder P3C Vibracore (P3C) (Figure 1), operates at the following specifications:

Weight of vibracore head:	150 lbs
Power setting:	Medium = 5.0 kW, 8.0 amps
Force:	Centrifugal force at 60 Hz, medium power setting, produces a force of 20 kilonewtons
Vibrations per minute:	3450 vibrations per minute at 60 Hz
Water depth capability:	500 feet
Core tube type:	• 4-inch diameter core tubes • Metal or polycarbonate core tubes NOTE: the Sediment team uses polycarbonate core tubes for sediment sampling

3.0 EQUIPMENT AND SUPPLIES

The following equipment and supplies are required for the collection of a long core sediment sample at a typical sampling location, and are available on the R/V *Mudpuppy II* unless otherwise stated.

- Rossfelder P3C Vibracore
- Polycarbonate core tubes
- Underwater electrical cable
- Control box/power source
- Wrench
- Hydraulic articulating crane with winch
- Eggshell core catcher (also referred to as the "nose cone" in this SOP)

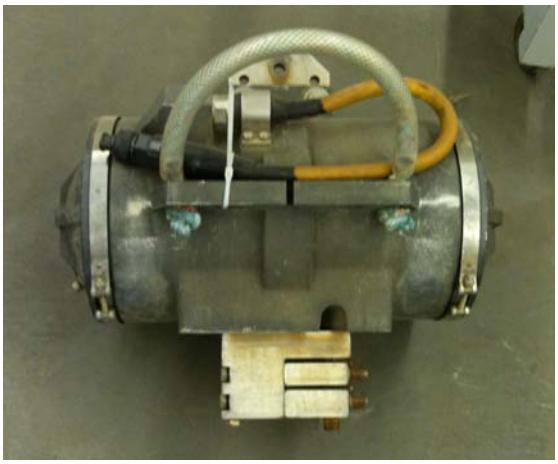


Figure 1. Vibracore sampler

4.0 STEPS TO OBTAIN SEDIMENT CORES USING THE VIBRACORER

Before coring, the nose cone should be installed in the core tube with at least three rivets and core tubes should be cut to accommodate sediment depth.

1. Record sample location using global positioning system (GPS).
2. Measure and record water depth.
3. Using the winch, vertically lift the vibracore head so that the vibracore head is suspended just off of the bow of the sampling vessel.
4. Insert the core tube into the core tube clamp, making sure that the tube slides into the check valve.
5. Hold core tube in place while tightening the clamp around the core tube using a wrench.
6. Counting the markings on the cable prevents the vibracore head from becoming imbedded into the sediment. With the winch, lower the entire assembly until the core nose is just above the sediment surface, as indicated by the markings on the cable. Turn on the power to the vibracore head.
7. Slowly lower the vibracorer by keeping 6-10 inches slack of cable at a time. The cable is marked in 1-ft increments. Monitor the core tube penetration by feeling for slack in the cable. Keep track of penetration depth by counting the markings on the cable.
8. Once the vibracorer ceases to penetrate the sediment (i.e., the unit stops lowering, cable starts to slack, when the end of the core tube length is reached, or when maximum core length is reached when vibracore stops or tube is buried), turn off power to the vibracore head.
   NOTE: Care must be taken to ensure the vibracore head is not embedded in the sediment.
9. Using the winch, remove the core from the sediment surface.

10. Lift the entire assembly so that the sediment/water interface in the core tube is visible. Rinse off the sediment from the core tube with the hose. Drill holes through the core tube at the sediment/water interface to decant water from the tube.
11. Tie a clove hitch around the core tube.
12. Remove and lower the core tube onto the processing table.

5.0 QUALITY CONTROL AND QUALITY ASSURANCE

If the first attempt at sample collection is not successful then do not dump the sample overboard at the same exact location where collection occurred. Instead, dump the sample away from the original sample location.

6.0 PERSONAL PROTECTIVE EQUIPMENT FOR OPERATION OF VIBRACORING SYSTEM

At a minimum, a hard hat with face shield, steel-toed boots, safety goggles, thicker rubber gloves or leather gloves, and a life jacket needs to be worn to operate the vibracoring system.

7.0 PERSONNEL QUALIFICATIONS

Personnel have an opportunity to learn how to use the Rossfelder P3C Vibracore at a sampling event during the sampling season. Training involves shadowing a trained sampler and taking samples under supervision of the trainer.

8.0 REFERENCES

http://www.epa.gov/quality/qs-docs/g6-final.pdf

Rossfelder P3C Vibracore Manual, Oct 1999.

APPENDIX A-D

Pontoon Boat Standard Operating Procedures

AFFILIATED RESEARCHERS
Environmental Research, Mapping, and Reporting

GSA Contract Holder

STANDARD OPERATING PROCEDURES
IN USING THE
SHALLOW-WATER VIBRACORE BOAT SYSTEM

July 2010

(989) 739-5471 • 3585 North US Highway 23, Oscoda, MI 48750 • www.affiliatedresearchers.com
Service-disabled Veteran-owned small business • Certified HUBZone business consideration

1.0 Scope and Application

This Standard Operating Procedure describes procedures in using Affiliated Researchers' shallow-water vibracore boat system.

2.0 Personnel Qualifications

The vibracore boat operator-in-charge (OIC) will ensure all personnel participating with the vibracore operation have received training by a qualified instructor, and are proficient with all aspects of the vibracore system and the boat.

3.0 Review of Project Details

The vibracore boat OIC will review with all members the scope, objectives, data, and other details of the project to ensure all members understand equipment operations, roles, positions, and expected times of project accomplishments.

4.0 General Safety

The vibracore boat OIC will review applicable safety plans and requirements (to include Affiliated Researchers' SOP for Boating Safety, and GLNPO's Safety, Health, and Environmental Compliance documents) and explain general safety precautions and procedures to ensure all members understand the safe and correct manner of accomplishing their respective duties.

The vibracore boat OIC will observe and ensure throughout the vibracore operation that safety requirements are complied with.

5.0 System Overview

Affiliated Researchers' shallow-water vibracore boat system consists of 20 foot long pontoon boat, a vibracorer, a vertical A-frame hoist, a 40hp *Honda* 4-stoke outboard engine, a removable hatch, a freshwater wash down system, and a 2.5KW GFCI-protected power source. The vibracorer consists of a high frequency electric vibrator, vibracore head, polycarbonate *Lexan* core tube, and an electrical power cable.

When a core tube is rigidly attached to a vibracore head, the vibrational energy is propagated along the tube and into the sediments. The vibrational energy re-orients the sediment particles, allowing for the advancing core tube into the sediment, and allowing a more efficient sediment sample (i.e. equivalent sediment profile) to be obtained. Because the sediments are saturated, the interstitial water pressure in the region around the core tube increases with the vibration. As

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interstitial pressure increases, the effective confining stress in the sediment decreases, and the strength of the sediments diminishes. With the penetration resistance of the sediment reduced from the vibration, the core tube penetrates the soil under the static weight of the vibracorer.

5.1 Shallow-water Vibracore Head

The shallow-water vibracore head consists of a *Vibco* industrial electric vibrator that weighs 9lbs; operates with 115V (AC or DC) at 0.65 amps; vibrates at 10,000VPM at 100lbs of force (445 Newtons); and produces a noise level of 70 dB (less than that of traffic noise). The *Vibco* electric vibrator is bolted within a stainless steel, submersible portion of the vibracore head.

The vibracore head contains a core tube clamp to hold a 4" diameter (O.D.) *Lexan* core tube. The vibracore head also contains a check valve to allow air from the core tube to escape during descent of the core tube into the sediment, and creates suction during retrieval of the core tube to help contain the sediment sample. The total weight of the vibracore head is 37lbs.

Affiliated Researchers' shallow-water vibracorer has been tested to a depth of 50ft, and has an operating duty cycle of 20 minutes.

5.2 Deep-water Vibracore Head

Affiliated Researchers' deep-water vibracore head is a submersible *PVL Technologies VC-3.5 Vibracore Power Head* that weighs 80lbs, operates with 115VAC at 15 amps at 60Hz, and vibrates at 9,000VPM.

The *VC-3.5 Vibracore Power Head* contains core tube clamps to hold either a 3" or 4" diameter (O.D.) *Lexan* core tube. The *VC-3.5 Vibracore Power Head* also contains a check valve to allow air from the core tube to escape during descent of the core tube into the sediment, and creates suction during retrieval of the core tube to help contain the sediment sample.

The *VC-3.5 Vibracore Power Head* includes the following optional equipment for deeper waters:

- Bottom Cap Clamp Accessory;
- Stabilizer Accessory; and,
- Clamp-on Weight Accessory.

Affiliated Researchers' deep-water *VC-3.5 Vibracore Power Head* has been tested to a depth of 500ft, although the power cable is only 75ft in length. The *VC-3.5 Vibracore Power Head* has and operating duty cycle of 6 minutes. More information regarding the *VC-3.5 Vibracore Power Head* can be found in the VC-3.5.2 VIBRACORE SYSTEM OPERATING & MAINTENANCE MANUAL (Version 1.0, Updated April 11, 2010).

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6.0 EQUIPMENT AND SUPPLIES

- Vibracore head(s)
- Polycarbonate core tubes and caps
- Core tube nose piece and core catchers
- Underwater electrical cable
- Miter box and saw
- Core tube rack
- Power source
- Freshwater washdown
- GPS with preloaded coordinates of sampling sites
- Tool box with wrenches
- PDD and safety equipment and supplies
- Boat operation equipment

7.0 OBTAINING SEDIMENT CORES USING THE VIBRACORER

1. Ensure GPS locations of the sediment sampling sites are uploaded onto the vibracore boat GPS.
2. If no overhead obstructions are to be encountered enroute to the sediment sampling site, erect the A-frame hoist while at the dock prior to departure.
3. Using the GPS chart display on the vibracore boat, position vibracore boat at the predetermined coring site.
4. Determine water depth using echo sounder.
5. Secure the vibracore boat at the coring site using at least 2 spuds (in waters less than 10ft deep) or using 2 anchors (in waters greater than 10ft deep).
6. Re-determine and record water depth using echo sounder and tape-disk.
7. Measure and record the depth of the accumulated sediments using a sediment probe-pole.
8. If not previously accomplished, erect the A-frame hoist.
9. Select or cut the core tube to proper length to accommodate water and sediment depth.
10. Lay the core tube upon the boat deck and insert it into the core tube clamp on the vibracore head. Tighten the core tube clamp around the core tube using a torque wrench to 20lbs of torque.
11. Install the nose cone and/or core catcher at the other end of the core tube with 4 rivets.
12. Connect the vibracore head to the hoist cable and raise the vibracorer to a height sufficient to allow the bottom of core tube to be placed within the opened hatch located amidships the boat.
13. Ensure check valve is operational.
14. Lower the vibracorer to one foot above the water surface and then turn on the electrical power to the system.
15. Record the latitude and longitude of the sediment sampling site on the GPS and on a data sheet. Record the time and date on the data sheet as well as any noted conditions.
16. Slowly lower the vibracorer; keeping note not to exceed about 6” of slack in the cable.

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17. The cable is marked in 1 ft increments. Keep track of penetration depth by counting the markings on the cable. Counting the markings on the cable prevents the vibracore head from becoming imbedded in the sediment.
18. Once the establish sediment depth has been established, or the vibracorer has reached refusal, turn off electrical power to the vibracore head
19. Raise the vibracorer from the sediments using the A-frame hoist.
20. Lift the entire assembly so that the sediment/water interface in the core tube is visible. Rinse off the sediment from the core tube with freshwater from the washdown.
21. Place a cap at the bottom of the core tube, and drill ¼" diameter holes through the core tube at the sediment-water interface to decant water from the tube.
22. Tie a safety line (using a "clove-hitch" knot) around the core tube.
23. Remove the core tube from the core tube clamp.
24. Place the core tube on the miter box for cutting to the described sampling lengths, in accordance with described sample handling procedures (e.g. Project QAPP procedures).
25. Cap the ends of the sampling lengths and place cut and capped core tube lengths in core tube holding rack.
26. Lower the vibrohead back onto the boat deck.

8.0 QUALITY CONTROL AND QUALITY ASSURANCE

Affiliated Researchers' GENERAL QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES, as well as any additional project QAQC requirements should be briefed and followed by the OIC prior to operations.

Equipment should be ops-checked at the beginning of each operational day. The equipment should be inventoried with all damage noted, at the end of each operational day.

Operational checklist should be followed during all phases of the operation.

Data sheets should be completed during operations and transcribed to electronic format at the end of each field day.

Clean-sampling protocol should be followed to prevent cross-contamination of sediment samples.

The onboard freshwater washdown system should be used to rinse sediments from vibracore system and system handlers following the retrieval of each core tube. Gloves should be disposed of after each sediment sample has been collected.

Chain-of-custody protocol should be followed when required.

9.0 PPE FOR OPERATION OF VIBROCORE SYSTEM

At a minimum, a hard hat, safety goggles, rubber gloves, and a USCG approve life preserver need to

be worn when operating the vibracore boat system.

Refer to the other project related documents for additional requirements (e.g. Affiliated Researchers' SOP for Boating Safety, and GLNPO's Safety, Health, and Environmental Compliance, Appendix N).

10.0 PREVENTATIVE MAINTENANCE

The vibracore system should be check before and after each day's use to determine maintenance requirements. Refer to the VC-3.5.2 VIBRACORE SYSTEM OPERATING & MAINTENANCE MANUAL (Version 1.0, Updated April 11, 2010).

The connectors and o-rings should be inspected, cleaned and/or lubricated, and replaced as necessary, each day during operations.

11.0 APPROVALS

20 July 2010

_____ _____

Chris Martin Date
Technician, Vibracore Boat System OIC

20 July 2010

_____ _____

Rollin C. Reineck, Jr. Date
Director

APPENDIX B

CLP SOWs, Reporting, Accuracy, and Precision Limits

Request for Quote (RFQ) for Modified Analysis

Date: April 12, 2010

Subject: Modification Reference Number: 1734.2
Title: PAH with Three Additional Compounds and Cx Alkylated Series Analysis with Special Reporting Requirements
Sample Matrix: Sediment and Water
Fraction Affected: SVOA SIM
Statement of Work: SOM01.2

Purpose:

The Contractor Laboratory is requested to perform the following modified analyses under the Organic Statement of Work (SOW) SOM01.2, based on the additional specifications listed below. Unless specifically modified by this modification, all analyses, Quality Control (QC), and reporting requirements specified in SOW SOM01.2 remain unchanged and in full force and effect. The number of samples requested in this modification is not guaranteed.

Please note that accepting a modified analysis request is voluntary, and that the Laboratory is not required to accept the modified analysis. There will be no adverse effect to the Laboratory for not accepting the modified analysis request. However, once the Laboratory accepts the request for modified analysis, it shall perform the analysis in accordance with this modification and as specified in SOW SOM01.2.

The Laboratory is requested to review the modification described herein, determine whether or not it shall accept the requested modified analyses, and complete the attached response form. The Laboratory shall provide comments in response to the required changes in the designated area, in order to ensure that the modified analysis can be completed in accordance with the specifications described herein.

Modification to the SOW Specifications:

SOW SOM01.2 requires contract Laboratories to prepare and analyze samples for Semivolatile target compounds through the protocol outlined in Exhibit D, Analytical Method for the Analysis of Semivolatile Organic Compounds, at the Contract Required Quantitation Limits (CRQLs) specified in Exhibit C, Section 2.0. ***Refer to the special extraction instructions for soil/sediment samples and Special Reporting for all matrices***.

The proposed modification will require the Laboratory to extract and analyze samples for the list of Semivolatile target compounds following the calibration and analytical requirements at the CRQLs specified in Table 1, by Selected Ion Monitoring (SIM) protocol. The Laboratory shall note that Table 1 contains the 17 SOW PAH target compounds, additional target compounds, and the several Cx alkylated PAH homologue series, for SIM analyses. ***A SVOA extraction procedure for SIM analysis shall be performed spiked with the two (2) SOW SIM Deuterated Monitoring Compounds (DMCs) specified below.***

Table 1 - Polynuclear Aromatic Hydrocarbons Target Compounds including Alkylated PAH Homologue List

Compound	CAS Number OR Analyte Code	Quantitation Ion	Conf. Ions	SIM CRQL (µg/kg)
*1-Methylnaphthalene	90-12-0	142	141	3.3
2-Methylnaphthalene	91-57-6	142	141	3.3
******Naphthalene***	***91-20-3***	***128***	***127***	3.3
**C1-Naphthalenes	C1NAPH	142	141	3.3
**C2-Naphthalenes	C2NAPH	156	141	3.3
**C3-Naphthalenes	C3NAPH	170	155	3.3
**C4-Naphthalenes	C4NAPH	184	169	3.3
******Acenaphthylene***	***208-96-8***	***152***	***153***	3.3
******Acenapthene***	***83-32-9***	***154***	***153***	3.3
******Fluorene***	***86-73-7***	***166***	***165***	3.3
**C1 Fluorenes	C1FLUOR	180	165	3.3
**C2Fluorenes	C2FLUOR	194	179	3.3
**C3 Fluorenes	C3FLUOR	208	193	3.3
******Phenanthrene***	***85-01-8***	***178***	***176***	3.3
******Anthracene***	***120-12-7***	***178***	***176***	3.3
**C1-Phenanthrenes/Anthracenes	C1PHAN	192	191	3.3
**C2-Phenanthrenes/Anthracenes	C2PHAN	206	191	3.3
**C3-Phenanthrenes/Anthracenes	C3PHAN	220	205	3.3
**C4-Phenanthrenes/Anthracenes	C4PHAN	234	219	3.3
******Fluoranthene***	***206-44-0***	***202***	***101***	3.3
******Pyrene***	***129-00-0***	***202***	***101***	3.3
**C1-Fluoranthenes/Pyrenes	C1FLPY	216	215	3.3
**C2-Fluoranthenes/Pyrenes	C2FLPY	230	215	3.3
**C3-Fluoranthenes/Pyrenes	C3FLPY	244	229	3.3

Compound	CAS Number OR Analyte Code	Quantitation Ion	Conf. Ions	SIM CRQL (µg/kg)
***Benzo (a) anthracene	56-55-3	228	226	3.3
***Chrysene	218-01-9	228	226	3.3
**C1 Chrysenes	[1]C1CHRYS	242	241	3.3
**C2 Chrysenes	[1]C2CHRYS	256	241	3.3
**C3 Chrysenes	[1]C3CHRYS	270	255	3.3
**C4 Chrysenes	[1]C4CHRYS	284	269	3.3
***Benzo (b) fluoranthene	205-99-2	252	253, 125	3.3
***Benzo (k) fluoranthene	207-08-9	252	253, 125	3.3
*Benzo (e) pyrene	192-97-2	252	253	3.3
***Benzo (a) pyrene	50-32-8	252	253, 125	3.3
*Perylene	198-55-0	252	253	3.3
***Indeno (1,2,3-cd) pyrene	193-39-5	276	277, 138	3.3
***Dibenzo (a,h) anthracene	53-70-3	278	279, 139	3.3
***Benzo (g,h,i) perylene	191-24-2	276	277, 138	3.3
Total 16 PPAH	**TPPAH**	**N/A**	**N/A**	3.3
Total PAH	**TPAH**	**N/A**	**N/A**	3.3

*Indicates an additional non-SOW target compound.
** Indicates a Cx alkylated PAH homologue.
***Indicates compound included in the Total 16 PPAH
Total PAH shall be the sum of all analyte concentrations in Table 1 except 1-Methylnaphthalene and 2- Methylnaphthalene
[1]This code is used to report data for this modified analysis request only.

Calibration and Standardization: The Laboratory shall prepare at least five calibration standards containing all 17 SOW PAH target compounds, 3 additional target compounds, internal standards and Deuterated Monitoring Compounds (DMCs) at the concentrations described in the table below:

Compound Class	Cal Level 1 (ng/µL)	Cal Level 2 (ng/µL)	Cal Level 3 (ng/µL)	Cal Level 4 (ng/µL)	Cal Level 5 (ng/µL)
PAHs	0.10	0.20	0.40	0.80	1.0
Internal Standards	0.40	0.40	0.40	0.40	0.40
DMCs	0.10	0.20	0.40	0.80	1.0

The Laboratory shall analyze each calibration standard and extract by injecting 1.0 µL of standard or injecting 2.0 µL of each calibration standard and extract with half the concentrations listed in the table above. When the laboratory chooses 2.0 µL injections DMC and Internal Standards concentrations must also be half the concentration in sample extracts.

Internal Standard Solution: The internal standard solution for the modified analysis of PAH and alkylated PAH shall consist of Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-$_{d12}$ and Pyrelene-d_{12}. Just prior to SIM analysis the Laboratory shall add sufficient amount of this internal standard solution to an aliquot of sample extract to result in a 0.40 ng/µL concentration of each internal standard.

Deuterated Monitoring Compounds (DMC): The DMC spiking solution for the modified analysis of PAH and alkylated PAH shall contain Fluoranthene-d_{10} and 2-Methylnaphthalene-d_{10} as DMC compounds at concentrations specified in the SOW.

After sample preparation the Laboratory shall perform SIM analyses for all the Table 1 target compounds. The Laboratory shall analyze the DMCs mentioned in this document, in addition to all the internal standards. The Laboratory will perform a dilution for any of the 17 SOW PAH target compounds and the 3 additional target compounds exceeding the calibration range (***this does not apply to alkylated PAH homologue series***).

Standard Reference Oil: An alkylated PAH homologue Retention Time (RT) source material must be analyzed after the opening CCV prior to sample analysis, in each analytical batch. The Standard Reference Oil shall be called SRO## (where ## can be alpha numeric characters). This standard is not billable, but must be submitted as part of the deliverable.

A one (1) percent solution of Alaskan North Slope Crude Oil in Methylene chloride was used as the Standard Reference Oil to develop this Modified Analysis. As an alternative a one (1) percent solution of standard reference Coal Tar in methylene chloride may be substituted for Alaskan North Slope Crude Oil. The solution should be analyzed initially using GC/MS full scan to establish the retention time windows for the alkylated PAH homologues to create the SIM descriptors. The standard reference oil must be analyzed after the opening CCV standard and prior to sample analysis using GC/MS SIM to verify that the SIM descriptors are appropriate for the detection of the alkylated PAH homologues. The Alaskan North Slope Crude Oil can be cleaned-up using Gel Permeation Chromatography (GPC) to remove the "residual" crude oil fraction, provided that the GPC sample is concentrated to represent a 1% dilution of the original crude oil sample.

Refer to the **Special Reporting Requirements for the Electronic Data Deliverable (EDD) and Hardcopy** section below for details on reporting requirements of the Standard Reference Oil.

PAH Alkyl Homologues: No separate calibration is required for PAH alkyl homologues. Once a homologue pattern has been identified using the Standard Reference Oil, the multi-peak area representing the homologue series is quantified using the Response Factor (RF) of the parent compound. For example, the C2-Phenanthrene series is quantified using the RF of Phenanthrene. Because quantitative standards are not available for every isomer, the concentration of each homologue group must be reported as estimated concentration using a "J" qualifier.

Tentatively Identified Compounds (TICs): TICs are not to be reported for this Modified Analysis request.

GC Column: A 30-m x 0.25-mm (5% Phenyl)-methylpolysiloxane capillary column with 0.33 or 0.50 µm film thickness such as an Agilent DB-5MS, Ultra-2, Rtx-5MS, HP-5MS, or a PTE-5 which is certified for GC/MS analysis with improved inertness, signal to noise ratio and sensitivity shall be used for this analysis. Substitution of a different gas chromatography column will result in data that will not be comparable to those data that this modified analytical protocol will achieve.

Gas Chromatography: The SOW recommended gas chromatographic conditions are not sufficient to achieve the chromatography required for this modification, therefore they have been modified for this analytical request in order to separate the 17 SOW PAH target compounds, 3 additional target compounds and establish the retention time ranges necessary to identify the Cx alkylated PAH homologue series for GC/MS SIM analyses. The following gas chromatographic conditions are recommended in order to achieve the separation necessary to perform this request for Modified Analysis.

Injection Volume: Use a 1-µL injection volume into a 2 or 4mm ID glass liner.

Carrier Gas: Helium

Electronic Pressure Control (EPC) Gas Program:
Initial Pressure: 30 psi
Initial Time: 1 min
Rate1: 99 psi/min
Final Pressure: Constant Flow (1 mL/min)
Vacuum Compensation: On

Gas Chromatography Conditions:
Injection Port: 300°C
Transfer Liner: 280°C
Initial Temp: 40°C
Initial Hold: 1 minute
Ramp Rate: 6°C/min
Final Temp: 320°C
Final Hold: 20 minutes or until Benzo (g,h,i) perylene elutes
Carrier gas: Helium

Note: Other GC conditions may be used, as appropriate, to achieve the chromatographic separation and response requirements of the Modified Analysis request.

Mass Spectrometer Conditions: Follow SOW SOM01.2 protocol.

SIM Analyte and Window Identification: Determine the elution order and co-elution characteristics of analytes through the qualitative analysis of individual compounds and solution of multiple compounds and comparison of sample mass spectra with reference mass spectra under the gas chromatography conditions presented above.

The Laboratory shall define the SIM data acquisition and SIM window retention times (RTs) of selected PAH and alkylated PAH found in the Standard Reference Oil prior to the first analysis

of analytical standards and/or samples in the SIM mode. The Standard Reference Oil should be analyzed in full-scan mode under the chromatography conditions presented above using the full-scan total ion chromatograms from these analyses to determine the proper Cx alkylated PAH homologue series start and stop times for this GC/MS SIM analyses. Start and stop times should be based on mass-chromatographic profiles of alkylated PAHs in an RT-range of 4 to 6 minutes with the characteristic peak in the approximate center of the cluster profile of the quantitation ion.

SIM Windows: Ensure that a minimum of 5 SIM scans will be acquired during the elution of each compound. ***The Laboratory must document the chromatographic time references it establishes* to identify the SIM acquisition ion groups and SIM acquisition windows for the PAH target analytes and the alkylated PAH homologue series:***

Time Step	Chromatographic Time Reference	Chromatographic Time in Minutes (Laboratory Determined)
1	End of Solvent Delay	
2	Start of Naphthalene-d8	
3	Start of Biphenyl	
4	Start of Acenaphthene-d10	
5	Start of Fluorene-d10	
6	End of Phenanthrene-d10	
7	Start of Fluoranthene	
8	Between Fluoranthene and Benz (a) anthracene	
9	Start of Benz (a) anthracene	
10	Start of Benzo (b) fluoranthene	
11	Start of Benzo (a) pyrene-d12	
12	Start of Indeno (1,2,3-cd) pyrene	

NOTE:** ***The Laboratory may modify chromatographic conditions if necessary, however the chromatographic conditions used in the analysis of samples must be identical to those established for the initial calibration.

Verify that the GC and SIM MS conditions and the SIM windows have been identified as required. The length of the time required to perform each sample and standard analysis is about one hour; ***therefore, the Laboratory shall verify that the instrument meets the SOW SOM01.2 tuning (DFTPP) criteria every 12 hours (Refer to Exhibit D/SVOA, Section 9.2.2).*** The Laboratory is required to demonstrate and document an acceptable initial calibration before any samples are analyzed. Response factors (RF) from a linear ICAL are used to calculate analyte concentrations in samples. The only exceptions are alkylated PAH homologues (multi-component) analytes not available commercially. The ICAL must be verified by analysis of an opening CCV immediately following the ICAL. A CCV is required at ***the beginning and end of each 12-hour period during which analyses are performed***.

The ICAL acceptance criteria are:

- Linearity of calibration curve assessed using the mean RRF.
- Individual %RSD $\leq$ 25%.
- Minimum RRF shall be according to the SOM01.2.

The CCV criteria are:

- After every 12 hours or less.
- Individual % diff ≤ 25%.
- The instrumental response (EICP area) for each of the internal standards in the sample must be within the range of 50.0% and 200% of the response of the internal standard in the most recent opening CCV standard analysis.

Corrective action for I/CAL and Opening or Closing CCV failure of the technical acceptance criteria outlined above, shall be according to the SOW specifications.

NOTE: ***Because of the low concentrations of compounds in the CCV solutions, column adsorption may be a problem when the GC has not been used for a day or more. Therefore, it is good practice to prime the GC by injecting a high level ICAL solution prior to running a CCV if the instrument has not been used for sample analysis for several days.***

Quantitation Calculation: Follow internal standard method of quantitation defined in the SOW SOM01.2, Exhibit D/SVOA, following equations in 11.2.1.6.2 Soil/Sediment EQ. 6. Response factors for alkylated PAH homologues are presumed equal to the response factor of the respective un-substituted (parent) compound. With the exception of 1-Methylnaphthalene and 2-Methylnaphthalene, alkylated PAH homologues should not be included in the calibration solution.

The Laboratory shall quantify and report total alkylated (Cx) Naphthalenes (as specified in Table 1) , total alkylated (Cx) Fluorenes and total (Cx) alkylated Chrysenes. The alkylated Phenanthrenes + Anthracenes must be reported as total alkylated (Cx) Phenanthrenes/Anthracenes. Total C2 Phenantherenes/Anthracenes will be quantitated using the RRF of the parent compound Phenantherene. The final concentrations must be totaled and reported as C2 Phenanthrenes/Anthracenes. Similarly, alkylated Fluoranthenes + Pyrenes will be quantitated using the RRF of the parent compound Fluoranthene and the final concentration as total alkylated (Cx) Fluoranthenes/Pyrenes.

Only the parent compounds and the isomers specified in Table 1 should be reported as individual compounds.

Analyte Identification: The extracted ion current profiles of the primary m/z and the confirmatory ion for each analyte must meet the following criteria:

- The pattern of each group and the retention time window for the group is established by the analysis of the Standard Reference Oil. Relative Retention Time (RRT) is useful to inter-compare the identity of compounds in samples and reference standard that elute within like alkylated PAH homologue groupings.

- The alkylated PAH homologue groupings (e.g. C3-Naphthalene) appear as clusters or groups of isomers and must be integrated as individual groups in their entirety. The total response of each group of alkylated homologues is used in the quantitative determination of the concentration of the entire group.

- The characteristic masses of each analyte of interest must be in the same scan or within one scan of each other. The retention time must fall within +/- 0.2 min of the retention time of the

authentic parent compounds that define each alkylated homologue grouping determined by the analysis of the daily calibration check or reference oil standard, respectively.

- The relative peak heights of the primary mass ion, compared to the confirmation or secondary mass ion, must fall within 30 percent of the relative intensities of the masses in a reference mass spectrum. The reference mass spectrum is obtained from the continuing calibration solution or the Standard Reference Oil for the parent compounds and alkylated homologues, respectively. Supportive data may be included in some instances for a compound that does not meet secondary ion confirmation criteria but is determined to be present in a sample after close inspection of the data by a qualified mass spectrometrist.

- Mass-spectra of an alkylated PAH group in a real sample quantified below reporting limits may not be definitive and subject to distortion by interfering peaks from non-target compounds especially when dominated by spectra of non-target compounds present at high concentrations. Consequently, the relative peak heights of primary mass-ion to the confirmation ion, will not always be within 30 percent of the relative intensities of the masses in a reference mass spectrum.

- Data not meeting the criteria established in this section are appropriately qualified or re-analyzed.

Special Reporting Requirements for the Electronic Data Deliverable (EDD) and Hardcopy:

In order to recalculate the final concentrations of the Cx alkylated PAH homologues of Napthalenes and Fluorenes listed in Table 1, additional initial calibration (ICAL) and opening and closing continuing calibration (CCV) information must be be provided in the electronic data deliverable (EDD). The Analysis/Analyte and AnalysisGroup/Analyte node(s), including the Peak node(s) for each, of the parent compound must be copied and reported using the 'Analyte Code' (see Table 1) as the CASRegistryNumber data element of each Cx alkylated PAH homologue being quantitated from the parent compound. Sum the 16 PAH compounds concentration highlighted bold italic in Table 1 and report as Total 16 PAH. Total PAH shall be the sum of all analyte concentrations, including alkylated homologues, in Table 1 except 1-Methylnaphthalene and Methylnaphthalene.

***Note:** The exception is reporting the PeakID element on the Peak node using the specified "Quantitaion Ion" for the Cx alkylated PAH homologues listed in Table 1.*

The reporting limit for the sample will be adjusted for actual sample size, moisture content, and dilutions. The Laboratory shall use SOW sample qualifiers for reporting final results with the following exceptions: A detected target compound result at or above the Laboratory adjusted MDL and below the sample-specific reporting limit (sample adjusted CRQL) shall be reported and qualified with a "J" flag, but any target compound detected below the adjusted MDL shall be flagged with a "U". All non-detected target compounds shall be qualified with a "U" flag.

The Laboratory shall qualify all detects below the sample adjusted CRQL and at or above the Laboratory adjusted MDL using a "J" qualifier. For reporting on the EDD the adjusted MDL for each target compound shall be reported in field "DetectionLimit" under

"ReportedResult" node for all samples, LCS (if applicable) and method blank in the EDD as specified in Exhibit H of the SOW. The Laboratory shall report "J" qualifier for any detected compound that has a concentration at or above the adjusted MDL and below the sample adjusted CRQL. This "J" qualifier should be populated in field "LabQualifier" under "ReportedResult" and "Analyte" nodes in the EDD as well as column "Q" on the following hardcopy Forms 1 specified in Exhibit B of the SOW.

In addition, the ResultType element under both the Analyte and ReportedResult nodes of compounds that have concentrations (results) below the MDLs shall be reported with "=" and the concentration in the Result element; however, the LabQualifiers elements under both nodes shall be reported with "U" qualifier. On the hardcopy Form 1, any compound that have a concentration (result) below the MDL shall be reported with an adjusted CRQL value and a "U" flag on column "Q".

Additional SOM01.2 Forms 6 and Forms 7 are not required. This requirement is not necessary for the other combined Cx alkylated PAH homologues listed in Table 1.The Standard Reference Oil, called SRO## must be reported using the same QC code as an opening CCV. The Laboratory shall modify a Form 7 (per SRO##) to report the Retention Times of the alkylated PAH homologue groups. The Laboratory shall submit all raw data associated after each copy of the modified Form 7.

Forms 1, 6 and 7 must be revised to include the list of target analytes as specified in Table 1. All other SOW hardcopy Forms and raw data must be submitted according to the SOW requirements. Please contact SMO for additional information, if necessary.

Reporting Requirements:

Hardcopy and electronic data ***(SEDD Stage 3)*** reporting are required as specified per SOW SOM01.2. All hardcopy and electronic data shall be adjusted to incorporate modified specifications. This includes attaching a copy of the requirements for modified analysis to the SDG Narrative. If specific problems occur with incorporation of the modified analysis into the hardcopy and/or electronic deliverable, the Laboratory shall contact the DASS Manager within the Sample Management Office (SMO) at (703) 818-4233 or via e-mail at CCSSUPPORT@fedcsc.com for resolution.

All samples analyzed for the same fraction within an SDG must be analyzed under the same fractional requirements. The Laboratory shall not include data for the same fraction with different requirements in the same SDG.

The Laboratory shall include the Modification Reference Number 1734.2 on each hardcopy data form under the "Mod. Ref. No." header appearing on each form as well as the data element "ServicesID" under the "SamplePlusMethod" node of the EDD. This should be done for the fractions affected by the modified analysis only. The "ServicesID" field should remain blank for all other fractions reported in the SDG. The Laboratory shall also document the Modification Reference Number and the Solicitation Number on the SDG Coversheet.

Clarifications/Revisions to the RFQ for Modified Analysis:

Laboratory Name:
Laboratory Comments:

Contractor Laboratory Acknowledgment Document

Analysis	Modification Reference Number	Hardcopy Turnaround Requirement	Preliminary Results (Y/N)	PDF Delivery (Y/N)	(A) Estimated No. of Samples by Matrix*	Cost For Modified Analysis	
						(B) New Per Sample Price	(A x B) Total Cost
Semivolatiles Extraction	1734.1	21 days	N	Y	83 soil	$_____	$_____
Semivolatiles SIM Analysis	1734.1	21 days	N	Y	83 soil	$_____	$_____
Semivolatiles Extraction	N/A	21 days	N	Y	5 water	$_____	$_____
Semivolatiles Analysis	N/A	21 days	N	Y	5 water	$_____	$_____
Aroclors Extraction	N/A	21 days	N	Y	83 soil 5 water	$_____	$_____
Aroclors Analysis	N/A	21 days	N	Y	83 soil 5 water	**$**_____	$_____
						Total Project Cost	$_____

Project Information
Estimated Shipping Period: 04/13/2010 through 04/23/2010
Additional Information: Please include both the analysis and PDF price in the new per sample price. Hard copy data packages will be sent to Denis Weslowski UA EPA Region 5 CRL 536 S. Clark St 1001 Chicago, IL 60604 and the PDF copy will be sent to Sara Goehl 77 W. Jackson Blvd G-17J Chicago, IL 60604.

Note: **The Government will make award to the contractor whose offer provides the best value to the Government, price and past performance considered.** The requirements in the RFQ are as stated, and the Government will reduce the line item price listed on the bid sheet for late deliverables at a rate of 5 percent per calendar day late, up to a maximum of 50 percent. The Government will treat noncompliant data and late data for Preliminary Results in accordance with the terms and conditions of the contract, using the price listed on the bid sheet as the basis for the calculation.
***Laboratory generated QC (including MS/MSD and LCS) are not billable for any analyses included in this solicitation. The Laboratory may wish to consider this when developing their quote.**

Name of Contractor Laboratory: _____

Contract Number: _____

___ Laboratory AGREES to perform analysis through the modified analysis protocol outlined in Modified Analysis Request. **By agreeing to this solicitation, the laboratory accepts samples that may be scheduled over the monthly contract capacity outlined in the laboratory's contract.**
___ Laboratory DECLINES to perform analysis through the modified analysis protocol outlined in Modified Analysis Request.

Signature of Laboratory Representative: _____ Date: _____

Signature of USEPA Contracting Officer: _____ Date: _____

Analysis: Description of the analyses being requested by the USEPA for this Case. This column is completed by SMO.

Modification Reference Number: The numerical value assigned to the technical requirements describing the changes to the Statement of Work. This column is completed by SMO.

Hardcopy Turnaround Requirement: The analytical data turnaround time required for this Case. This column is completed by SMO.

Preliminary Results: Indicates if Preliminary Results are required for the line item. This column is completed by SMO.

PDF Delivery: Indicates if PDF Delivery is required for the line item. This column is completed by SMO.

Estimated No. of Samples and sample Matrix (including QC): The client's estimated number of samples (by matrix), including billable QC samples, to be collected and shipped to the laboratory. This column is completed by SMO.

New Per Sample Price: Laboratory's sample price for analyzing the samples identified in the line item. This column is completed by the laboratory.

Total Cost: This value is the Estimated No. of Samples (including QC) multiplied by the New Per Sample Price. This column is completed by the laboratory.

Total Project Cost: Sum of the total costs for all line items. This is completed by the laboratory.

USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

INORGANIC ANALYSIS

Multi-Media, Multi-Concentration

ILM05.3
March 2004

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STATEMENT OF WORK

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EXHIBIT A

SUMMARY OF REQUIREMENTS

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Exhibit A - Summary of Requirements

Table of Contents

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1.0 PURPOSE

The purpose of the multi-media, multi-concentration inorganic analytical service is to provide analytical data for use by the U.S. Environmental Protection Agency (USEPA) in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). Other USEPA Program Offices that have similar analytical data needs also use this service.

2.0 DESCRIPTION OF SERVICE

The inorganic analytical service provides a contractual framework for laboratories. This framework applies USEPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 23 metals (including mercury) and cyanide in water/ aqueous and/or soil/sediment samples. The analytical service contract provides specific contractual requirements by which USEPA will evaluate the data.

3.0 DATA USES

This analytical service contract provides data which USEPA uses for a variety of purposes, such as: determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including: site inspections, Hazard Ranking System (HRS) scoring, remedial investigation/feasibility studies, remedial design, treatability studies, and removal actions.

The data may also be used in litigation against Potentially Responsible Parties in the enforcement of Superfund legislation. As a result, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, since it is used to make major decisions regarding public health and environmental welfare. The Contractor may be required to appear and testify to the accuracy and/or validity of the data generated.

4.0 SUMMARY OF REQUIREMENTS

4.1 Introduction to the Inorganic Statement of Work

The Statement of Work (SOW) is comprised of eight exhibits and two appendices. Exhibit A provides an overview of the SOW and its general requirements. Exhibit B contains a description of the reporting and deliverables requirements, in addition to the data reporting forms and instructions. Exhibit C specifies the Inorganic Target Analyte List (TAL) for this SOW with the Contract Required Quantitation Limits (CRQLs) for the sample matrices. Exhibit D details the required analytical procedures to be used with this SOW and resulting contracts. Exhibit E provides descriptions of required Quality Assurance/Quality Control (QA/QC), Standard Operating Procedures (SOPs), QA/QC performance, and the reporting of data. Exhibit F contains chain-of-custody and sample documentation requirements. To ensure proper understanding of the terms utilized in this SOW, a glossary can be found in Exhibit G. When a term is used in the text without explanation, the glossary meaning shall be applicable. Specifications for reporting data in computer-readable format appear in Exhibit H. Appendix A provides examples of the data format requirements specified in Exhibit H. Appendix B contains a description of the requirements for performing

modified analyses, as well as the analytical procedure for Graphite Furnace Atomic Absorption (GFAA).

4.2 Overview of Major Task Areas

For each sample, the Contractor shall perform the tasks described in each section. Specific requirements for each task are detailed in the exhibits referenced in the following sections.

4.2.1 Task I: Sample Receiving, Storage, and Disposal

4.2.1.1 Chain-of-Custody

The Contractor shall receive and maintain samples under proper chain-of-custody. All associated document control and inventory procedures shall be developed and followed. Documentation described herein shall be required to show that all procedures are strictly followed. This documentation shall be reported as the Complete Sample Delivery Group (SDG) File (CSF) (see Exhibit B). The Contractor shall establish and use appropriate procedures to safeguard confidential information received from USEPA.

4.2.1.2 Sample Scheduling/Shipments

Sample shipments to the Contractor's facility will be scheduled and coordinated by the Contract Laboratory Program (CLP) Sample Management Office (SMO). USEPA may request analyses that include all or a subset of the Inorganic Target Analytes listed in Exhibit C. The Contractor shall communicate with SMO personnel by telephone as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

4.2.1.2.1 Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing of the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier within the Contractor's geographical area. The Contractor shall be available to receive and process sample shipments at any time the delivery service is operating, including Saturdays, to ensure that short sample analysis time requirements can be met.

4.2.1.2.2 If there are problems with the samples (e.g., mixed media, containers broken or leaking) or sample documentation and paperwork (e.g., Traffic Reports/Chain of Custody Records not with shipment, sample and Traffic Report/Chain of Custody Record do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO and the USEPA Regional CLP Project Officer (CLP PO) regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO personnel and the USEPA Regional CLP PO in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

4.2.1.2.3 To monitor the temperature of the sample shipping cooler more effectively, each USEPA Regional Office may include a sample shipping cooler temperature blank with each cooler shipped. The temperature blank will be clearly labeled: USEPA COOLER

TEMPERATURE INDICATOR. The Contractor shall record the presence or absence of the cooler temperature indicator bottle on Form DC-1, Item 8 - Cooler Temperature Indicator Bottle (see Exhibit B).

4.2.1.2.3.1 When the USEPA Regional Office supplies a cooler temperature indicator bottle in the sample shipping cooler, the Contractor shall use the USEPA supplied cooler temperature indicator bottle to determine the cooler temperature. The temperature of the cooler shall be measured at the time of sample receipt by the Contractor.

4.2.1.2.3.2 The temperature of the sample shipping cooler shall be measured and recorded immediately upon opening the cooler.

4.2.1.2.3.3 To determine the temperature of the cooler: the Contractor shall locate the cooler temperature indicator bottle in the sample shipping cooler, remove the cap, and insert a calibrated thermometer into the cooler temperature indicator bottle. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the calibrated thermometer (±1°C) shall have a measurable range of 0-50°C. Other devices which can measure temperature may be used if they can be calibrated to ±1°C and have a range of 0-50°C. If a temperature indicator bottle is not present in the cooler, an alternative means of determining cooler temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining cooler temperature. The Contractor shall contact SMO and inform them that a temperature indicator bottle was not present in the cooler. The Contractor shall document the alternative technique used to determine cooler temperature in the SDG Narrative.

4.2.1.2.3.4 If the temperature of the sample shipping cooler's temperature indicator exceeds 10°C, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the Region from which the samples were shipped for instruction on how to proceed. The Region will either require that no sample analysis(es) be performed or that the Contractor proceed with the analysis(es). SMO will in turn notify the Contractor of the Region's decision. The Contractor shall document the Region's decision and the EPA sample numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.

4.2.1.2.3.5 The Contractor shall record the temperature of the cooler on Form DC-1, under Item 9 - Cooler Temperature, and in the SDG Narrative (see Exhibit B).

4.2.1.2.4 The Contractor is required to retain unused sample volume, used sample containers, and empty sample bottle containers for a period of 60 days after data submission. From time of receipt until analysis, the Contractor shall maintain <u>all</u> water/aqueous (preserved and unpreserved) and/or soil/sediment samples at 4°C (±2°C) (see Exhibit B).

4.2.1.2.5 The Contractor shall be required to routinely return sample shipping containers (e.g., coolers) to the appropriate sampling

office within 14 calendar days following shipment receipt (see contract, Section G titled, "Government Furnished Samples").

4.2.1.2.6 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case.

4.2.1.2.6.1 A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received, or
- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or
- Each 7 calendar day period (3 calendar day period for 7 day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG.

4.2.1.2.6.2 Samples may be assigned to SDGs by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory. However, PE samples received within a Case shall be assigned to an SDG containing field samples for that Case. Such assignment shall be made at the time the samples are received, and shall <u>not</u> be made retroactively.

4.2.1.2.6.3 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report/Chain of Custody Record bearing the sample number and descriptive information regarding the sample. EPA sample numbers are six digits in length. If the Contractor receives a sample number of any other length, the Contractor shall contact SMO immediately. The Contractor shall complete and sign the Traffic Report/Chain of Custody Record, recording the date of sample receipt and sample condition on receipt for each sample container. <u>The Contractor shall also follow the instructions given on the Traffic Report/Chain of Custody Record in choosing the Quality Control (QC) samples when such information is provided</u>. If no QC sample is designated on the Traffic Report/Chain of Custody Record, the Contractor shall select a sample and notify SMO for Regional acceptance. SMO shall contact the Region for confirmation immediately after notification.

4.2.1.2.6.4 The Contractor shall submit signed copies of Traffic Reports/Chain of Custody Records for all samples in a SDG to SMO within **three working days** following receipt of the last sample in the SDG. Faxed copies of Traffic Reports/Chain of Custody Records do not meet this requirement. Traffic Reports/Chain of Custody Records shall be submitted in SDG

sets (i.e., all Traffic Reports/Chain of Custody Records for a SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

4.2.1.2.6.5 EPA Case numbers, SDG numbers, and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally and in reports/ correspondence.

4.2.1.3 Modified Analysis

The Contractor may be requested by USEPA to perform modified analyses. These modifications will be within the scope of this SOW and may include, but are not limited to, analysis of additional analytes and/or lower quantitation limits. These requests will be made by the USEPA Regional CLP PO, USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch Inorganic Program Manager (ASB PM), and Contracting Officer (CO) in writing, prior to sample scheduling. If the Contractor voluntarily elects to perform these modified analyses, these analyses will be performed with no increase in per sample price. All contract requirements specified in the SOW/Specifications will remain in effect unless the USEPA CO provides written approval for the modification(s) and a waiver for associated defects. The USEPA CO approval must be obtained prior to sample scheduling.

4.2.2 Task II: Sample Preparation and Analysis

4.2.2.1 Overview

The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high (greater than 15%) levels of organic and inorganic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the analysis with appropriate caution. It is the Contractor's responsibility to take all necessary measures to ensure laboratory safety.

4.2.2.2 The Contractor shall prepare and analyze samples as described in Exhibit D. Sample preparation methods shall remain consistent for all samples analyzed within a Case. Prior to sample analysis, the Contractor shall review the Traffic Report/Chain of Custody Record for any special sample analysis instructions. Anomalies that occur during sample analysis shall be reported to SMO and the USEPA Regional CLP PO immediately.

The Contractor shall collectively review all analytical results associated with a sample. This includes undiluted, diluted, serial dilution, and interference results. The Contractor shall report any significant anomalies between these results in the SDG Narrative indicating possible matrix interferences.

4.2.2.3 Quality Assurance/Quality Control Procedures

4.2.2.3.1 The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D and E. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibits B and H.

4.2.2.3.2 The Contractor shall maintain a Quality Assurance Management Plan (QAP) with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

4.2.2.3.3 Additional QC shall be conducted in the form of the analysis of laboratory PE samples submitted to the laboratory by USEPA. Unacceptable results of all such QC or laboratory PE samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to USEPA or rejection of the data for specific analyte(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values as determined by USEPA, as well as meeting the contract requirements for analysis (Exhibit D); QA/QC (Exhibit E); data reporting and other deliverables (Exhibits B and H); and sample custody, sample documentation, and SOP documentation (Exhibit F).

4.2.3 Task III: Sample Reporting

4.2.3.1 USEPA has provided to the Contractor formats for the reporting of data (Exhibits B and H). The Contractor shall be responsible for completing and submitting analysis data sheets, computer-readable data on diskette (or via an alternate means of electronic transmission approved in advance by USEPA) in a format specified in this SOW and within the time specified in Exhibit B, Section 1.1.

4.2.3.2 Use of formats other than those designated by USEPA (see Exhibits B and H) will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to the Government shall be required.

4.2.3.3 Computer generated forms may be submitted in the hardcopy Sample Data Package(s) provided that the forms are in **exact USEPA format**. This means that the order of data elements is the same as on each USEPA required form, including form numbers and titles, page numbers, and header information.

4.2.3.4 The data reported by the Contractor on the hardcopy data forms and the associated computer-readable data submitted by the Contractor on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) shall contain identical information. If discrepancies are found during Government inspection, the Contractor shall be required to resubmit either the hardcopy forms or the computer-readable data, or both sets of data, at no additional cost to USEPA.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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Exhibit B - REPORTING AND DELIVERABLES REQUIREMENTS

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1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 Report Deliverable Schedule

The following table reiterates the contract reporting and deliverables requirements and specifies the distribution that is required for each deliverable. The turnaround times for Items B through E are 7, 14, or 21 days.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM) will notify the Contractor in writing of such changes when they occur.

TABLE 1

Item		No. of Copies[A]	Delivery Schedule	Distribution			
				SMO	Region	CLP PO[D]	QATS
A.	Sample Traffic Reports/Chain of Custody Records	1	3 working days after receipt of last sample in Sample Delivery Group (SDG).[1]	X			
B.[2]	Sample Data Package	1	XX[C] days after Validated Time of Sample Receipt (VTSR)[1] of last sample in SDG.	X			
C.[2]	Data in Computer-Readable Format	1	XX[C] days after VTSR of last sample in SDG.	X	X		
D.[2]	Results of Intercomparison Study/PE Sample Analysis Study	1	XX[C] days after VTSR of last sample in SDG.	X			X
E.[2,3]	Complete SDG File (CSF)[B]	1	XX[C] days after VTSR of last sample in SDG.		X		
F.[4]	Preliminary Results	1	Within 72 hours after receipt of each sample at laboratory, if requested.	X	X		
G.[5,6]	Quarterly Verification of ICP-AES/ICP-MS Linear Ranges and ICP-AES Interelement Correction Factors	1	Quarterly: 15th day of January, April, July, and October.	X		X	X
	Annual Verification of Method Detection Limits (MDLs)	1	Annually: 15th day of January.	X		X	X

TABLE 1 (Con't)

Item		No. of Copies[A]	Delivery Schedule	Distribution			
				SMO	Region	CLP PO[D]	QATS
H.[6,7]	Standard Operating Procedures (SOPs)	1	Revise within 30 days after contract award and receipt of USEPA comments. Submit within 7 days of receipt of written request to recipients as directed. (See Exhibit E, Section 6) Submit within 14 days of amended SOP(s) as directed in Exhibit E, Section 6.4.	As Directed Amended SOPs distributed to CLP PO and QATS			
I.[6,7]	Quality Assurance Management Plan (QAP)	1	Revise within 30 days after contract award and receipt of USEPA comments. Submit within 7 days of receipt of written request to recipients as directed. (See Exhibit E, Section 5) Submit within 14 days of amended QAP as directed in Exhibit E, Section 5.3.	As Directed Amended QAP distributed to CLP PO and QATS			
J.	Electronic Instrument Data	Lot	Retain for 3 years after data submission. Submit within 7 days after receipt of written request by the USEPA Regional CLP PO. (See Exhibit E, Section 13)	As Directed			

Footnotes:

[A]The number of copies specified is the number of copies required to be delivered to each recipient.

[B]Contractor-concurrent delivery to USEPA's designated recipient [e.g., Quality Assurance Technical Support (QATS)] may be required upon request by the USEPA OSRTI ASB Inorganic Program Manager (ASB PM). Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the USEPA ASB PM.

[C]The number of days associated with these elements will be provided in the associated laboratory contract document and will also be provided at the time of sample scheduling by the Sample Management Office (SMO) Contractor.

[D]The CLP PO is the USEPA Regional Contract Laboratory Program (CLP) Project Officer (CLP PO) designated on the contract.

[1]Validated Time of Sample Receipt (VTSR) is the date of sample receipt at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report/Chain of Custody Record. Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less with the same laboratory turnaround and not exceeding 20 samples [excluding Performance Evaluation (PE) samples]. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. See Exhibit A for further description.

[2]**DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE.** Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. This includes resubmission of both the hardcopy and electronic deliverable. The date of delivery of the SDG, or any sample within the SDG, is the date all samples have been delivered. **If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables received after this time will be considered late.**

[3]Complete SDG File (CSF) will contain the original Sample Data Package plus all of the original documents described in Exhibit B, Section 2.6.

[4]If required at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of all Form Is (see Exhibit B, Section 2.9). Facsimile or electronic transmittal is required as requested by the Region. Electronic transmittals shall be transmitted as WordPerfect, MS Word, PDF, or other USEPA-approved formats. The Contractor will be notified of the format, fax numbers, or email address(es) at the time of sample scheduling. Sample Traffic Reports/Chain of Custody Records and SDG Cover Sheets shall be submitted with the Preliminary Results. The Contractor shall document all communication in a telephone log.

Preliminary Results Delivery Schedule:

If a sample requiring Preliminary Results arrives before 5 p.m., the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day.

[5]Also required in each Sample Data Package.

[6]See Exhibit E for description. Time is cited in calendar days.

Footnotes (Con't):

[7]The Contractor shall deliver both hardcopy and electronic (i.e., diskette) copies of the Standard Operating Procedures (SOPs) and Quality Assurance Management Plan (QAP).

1.2 Distribution

The following addresses correspond to the "Distribution" column in Exhibit B, Section 1.1, Table 1.

SMO: USEPA Contract Laboratory Program (CLP)
Sample Management Office (SMO)[1]
15000 Conference Center Drive
Chantilly, VA 20151-3808

Region: USEPA REGIONS: SMO will provide the Contractor with the list of addressees for data delivery for the 10 USEPA Regions. SMO will provide the Contractor with updated Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

USEPA Regional CLP Project Officer (CLP PO):

SMO will provide the Contractor with the list of addresses for the USEPA Regional CLP POs. SMO will provide the Contractor with updated name/address lists as necessary throughout the period of the contract.

QATS: USEPA Contract Laboratory Program (CLP)
Quality Assurance Technical Support (QATS) Laboratory[2]
2700 Chandler Avenue, Building C
Las Vegas, NV 89120
Attn: Data Audit Staff

In addition, the mailing and delivery addresses for the USEPA ASB Inorganic Program Manager (ASB PM) are:

Mailing Address: USEPA OSRTI Analytical Services Branch
Ariel Rios Building (5204G)
1200 Pennsylvania Avenue, N.W.
Washington, DC 20460
Attn: CLP Inorganic Program Manager

Fed-Ex/Overnight Delivery: USEPA OSRTI Analytical Services Branch
1235 Jefferson Davis Highway
Crystal Gateway I, 12th Floor
Arlington, VA 22202
Attn: CLP Inorganic Program Manager

[1]The SMO is a Contractor-operated facility operating under the SMO contract awarded and administered by USEPA.

[2]The QATS laboratory is a Contractor-operated facility operating under the QATS contract awarded and administered by USEPA.

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1 Introduction

The Contractor shall provide reports and other deliverables as specified in Exhibit B, Section 1.1. The required content and form of each deliverable is described in this exhibit. All reports and documentation **shall be**:

- Legible;
- Clearly labeled and completed in accordance with instructions in this exhibit;
- Arranged in the order specified in this section;
- Paginated sequentially according to instructions in this exhibit; and
- Double-sided.

NOTE: Complete Sample Delivery Group (SDG) Files (CSFs) need not be double-sided. (The CSF is composed of original documents.) However, Sample Data Packages delivered to the USEPA Contract Laboratory Program (CLP) Sample Management Office (SMO) and the Region, [and USEPA designated recipients, e.g., Quality Assurance Technical Support (QATS), upon written request] must be double-sided.

2.1.1 The Contractor shall use EPA Case numbers, SDG numbers, and EPA sample numbers to identify samples received under this contract, both verbally and in reports and correspondence. The contract number shall be specified in all correspondence.

2.1.2 Section 4 of this exhibit contains the required Data Reporting Forms in Agency-specified format. Section 3 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide USEPA with all required data. Data elements and field descriptors for reporting data in computer-readable format are contained in Exhibit H.

2.2 Resubmission of Data

If submitted documentation does not conform to the above criteria, the Contractor is required to resubmit such documentation with deficiency(ies) corrected within 4 business days, at no additional cost to USEPA.

2.2.1 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, through the USEPA Regional CLP Project Officer (CLP PO) action, or through a Regional data reviewer’s request, the data shall be clearly marked as “Additional Data” and shall be sent to both contractual data recipients (SMO and Region) and to USEPA’s designated recipient (e.g., QATS) when a written request for the Sample Data Package has been made. A cover letter shall be included which describes what data is being delivered, to which USEPA Case(s) the data pertains, and **who requested the data**.

2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data shall be sent to the two contractual data recipients (SMO and Region) and to USEPA’s designated recipient (e.g., QATS) when a written request for the Sample Data Package has been made. In all instances, the Contractor shall include a color-coded cover sheet (Laboratory

Response to Results of Contract Compliance Screening) provided by SMO. Electronic deliverables shall be submitted or resubmitted to SMO and the Region. Revised DC-1 and DC-2 forms shall be resubmitted to SMO and the Region.

2.3 Quality Assurance (QA) Management Plan and Standard Operating Procedures (SOPs)

The Contractor shall adhere to the requirements in Exhibits E and F.

2.4 Sample Traffic Reports/Chain of Custody Records

Each sample received by the Contractor will be labeled with an EPA sample number and will be accompanied by a Sample Traffic Report/Chain of Custody Record bearing the sample number and descriptive information regarding the sample. The current CLP Traffic Report is the "Inorganic Traffic Report & Chain of Custody Record". The CLP Traffic Report/Chain of Custody Record is one form divided into two sections: the Traffic Report section which consists of everything above the Chain of Custody Record section, and the bottom section which is the Chain of Custody Record. The Contractor shall complete the CLP Traffic Report/Chain of Custody Record (marked "Lab Copy for Return to SMO"), recording the date of sample receipt, verifying the number of samples, and signing the CLP Traffic Report/Chain of Custody Record.

Upon receipt, the Contractor shall sign for receipt of samples. The laboratory signature box is located at the bottom of the CLP Traffic Report/Chain of Custody Record in the Chain of Custody Record section. The laboratory sample custodian or designated recipient opening and verifying the contents of the cooler shall then verify receipt of all samples identified within the CLP Traffic Report section and sign and date the signature box located in the upper half of the CLP Traffic Report/Chain of Custody Record. If a non-CLP Traffic Report/Chain of Custody Record is submitted with the samples, for example a Regional Traffic Report/Chain of Custody Record, then the Contractor shall (1) sign and date receipt of the samples to maintain the chain-of-custody and (2) the sample custodian or designated recipient shall sign and date the Traffic Report/Chain of Custody Record to verify sample information.

The Contractor shall also enter the Sample Delivery Group (SDG) number, Case number, and the laboratory contract number on the CLP Traffic Report/Chain of Custody Record, in the appropriate boxes. The EPA sample number of the first sample received in the SDG is the SDG number. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. Under no circumstances should any SDG number be replicated within a Case. If necessary, select an alternative sample number for the SDG number. The SDG number is also reported on all data reporting forms (see Exhibit B, Section 3 - Form Instructions). If the laboratory is requested to transfer samples to another facility, the Contractor shall date and enter the name of the facility to where the samples will be transferred on the CLP Traffic Report/Chain of Custody Record.

2.4.1 The Contractor shall submit Traffic Reports/Chain of Custody Records in SDG sets (i.e., Traffic Reports/Chain of Custody Records for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached. The SDG Cover Sheet shall contain the following items:

- Laboratory name;
- Contract number;
- Sample analysis price (full sample price from the contract);
- Case number; and
- List of EPA sample numbers of all samples in the SDG, identifying the **first** and **last** samples received, and their Laboratory Receipt Dates (LRDs).

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the sample with the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the sample with the highest sample number (considering both alpha and numeric designations).

2.4.2 EPA field sample numbers are six digits in length and continuous (without spaces or hyphens). If the Contractor receives sample numbers of any other length, the Contractor shall contact SMO immediately. The original Sample Traffic Report/Chain of Custody Record page marked "Lab Copy for Return to SMO", with laboratory receipt information and signed with original Contractor signature, shall be submitted for each sample in the SDG.

2.4.3 If samples are received at the laboratory with multi-sample Traffic Reports/Chain of Custody Records, all the samples on one multi-sample Traffic Report/Chain of Custody Record may not necessarily be in the same SDG. In this instance, the Contractor shall make the appropriate number of photocopies of the Traffic Report/Chain of Custody Record, and submit one copy with each SDG Cover Sheet.

2.5 Sample Data Package

The Sample Data Package shall include data for analysis of all samples in one SDG, including field and analytical samples, blanks, spikes, duplicates, and Laboratory Control Samples (LCSs). The Sample Data Package shall be complete before submission, and shall be consecutively paginated (starting with page number one and ending with the number of all pages in the package). The Sample Data Package shall include the following:

2.5.1 Cover Documentation

2.5.1.1 Cover Page for the inorganic analyses Data Package shall include: laboratory name; laboratory code; contract number; Case number; SDG number; Non-Routine Analytical Service (NRAS) number (if appropriate); EPA sample numbers in alphanumeric order showing EPA sample numbers cross-referenced with laboratory Sample ID numbers; and completion of the questions on use of background and interelement corrections for the samples.

2.5.1.1.1 The Cover Page shall contain the following statement, <u>verbatim</u>: **"I certify that this Sample Data Package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy Sample Data Package and in the computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) has been authorized by the Laboratory Manager or the Manager's designee, as verified by**

the following signature." This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

2.5.1.2 SDG Narrative. This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name, Case number, SDG number, contract number, and detailed documentation of any Quality Control (QC), sample, shipment, and/or analytical problems encountered in processing the samples reported in the Sample Data Package. The Contractor shall include any technical and administrative problems encountered and the resolution or corrective actions taken. This includes documenting the alternative technique used to determine cooler temperature if a temperature indicator bottle is not present in the cooler. The Contractor shall also provide, in the SDG Narrative, sufficient information, including equations or curves (at least one equation or curve per method), to allow the recalculation of sample results from raw instrument output. The Contractor shall also include a discussion of any flexibility Statement of Work (SOW) modification. This includes attaching a copy of the USEPA approved modification form to the SDG Narrative. Additionally the Contractor shall also identify and explain any differences which exist between the Form Is and supporting documentation provided in the data package and those previously provided as Preliminary Results.

2.5.1.3 Sample Log-In Sheet [Form DC-1]

2.5.1.4 Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

2.5.1.5 Sample Traffic Reports/Chain of Custody Records

2.5.2 Sample Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. Data should be arranged in increasing alphanumeric EPA sample number order, followed by the QC analyses data, quarterly and annual verification of method and instrument parameters forms, raw data, and copies of the digestion and distillation logs.

2.5.2.1 Inorganic Analysis Data Sheet [Form IA-IN and Form IB-IN]. Tabulated analytical results of the requested analytes shall be included. The validation and release of these results is authorized by a specific signed statement on the Cover Page. In the event that the laboratory cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

2.5.2.1.1 Appropriate concentration units shall be specified and entered on Forms IA-IN and IB-IN. The quantitative values shall be reported in units of micrograms per Liter (UG/L) for water samples and milligrams per kilogram (MG/KG) for solid samples. (No other units are acceptable.) Results for solid samples shall be reported on a dry weight basis. Analytical results shall be reported to two significant figures if the result value is less than 10 and to three significant figures if the value is greater than or equal to 10. Results for percent solids shall be reported to one decimal place. The preceding discussion concerning significant numbers applies to Forms IA-

IN, IB-IN, and IX-IN only. For other forms, follow the instructions specific to those forms as discussed in this exhibit.

2.5.2.2 Quality Control (QC) Data

2.5.2.2.1 The QC summary for inorganic analysis shall contain the forms listed below.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

2.5.2.2.1.1 Initial and Continuing Calibration Verification [Form IIA-IN]

2.5.2.2.1.2 CRQL Check Standard [Form IIB-IN]

2.5.2.2.1.3 Blanks [Form III-IN]

2.5.2.2.1.4 ICP-AES Interference Check Sample [Form IVA-IN]

2.5.2.2.1.5 ICP-MS Interference Check Sample [Form IVB-IN]

2.5.2.2.1.6 Matrix Spike Sample Recovery [Form VA-IN]

2.5.2.2.1.7 Post-Digestion Spike Sample Recovery [Form VB-IN]

2.5.2.2.1.8 Duplicates [Form VI-IN]

2.5.2.2.1.9 Laboratory Control Sample [Form VII-IN]

2.5.2.2.1.10 ICP-AES and ICP-MS Serial Dilutions [Form VIII-IN]

2.5.2.2.1.11 Method Detection Limits (Annually) [Form IX-IN]

2.5.2.2.1.12 ICP-AES Interelement Correction Factors (Quarterly) [Form XA-IN]

2.5.2.2.1.13 ICP-AES Interelement Correction Factors (Quarterly) [Form XB-IN]

2.5.2.2.1.14 ICP-AES and ICP-MS Linear Ranges (Quarterly) [Form XI-IN]

2.5.2.2.1.15 Preparation Log [Form XII-IN]

2.5.2.2.1.16 Analysis Run Log [Form XIII-IN]

2.5.2.2.1.17 ICP-MS Tune [Form XIV-IN]

2.5.2.2.1.18 ICP-MS Internal Standards Relative Intensity Summary [Form XV-IN]

2.5.2.3 Raw Data

For each reported value, the Contractor shall include in the Sample Data Package all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the quarterly and annual verification of method and instrument parameters submitted as a part of each Sample Data Package. When analysis of the ICP-AES or ICP-MS target analytes listed in Exhibit C of this SOW (or any subset or additional analytes) is requested, the raw data shall include, for

all samples, not only the results for the requested analyte(s), but also those for all the interferents (Exhibit D/ICP-AES, Table 1, or Exhibit D/ICP-MS, Section 7.2.4.4.1, as appropriate). The raw data shall also contain the results of any other analyte(s) which have been determined to interfere with the requested analytes(s).

2.5.2.3.1 Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Batch Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the Method Detection Limit (MDL). Raw data shall not be corrected for dilutions or volume adjustments. All Atomic Absorption (AA), Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES), and Inductively Coupled Plasma - Mass Spectrometer (ICP-MS) instruments shall provide a legible hardcopy of the direct real-time instrument readout (i.e., strip charts, printer tapes, etc.) or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included. A hardcopy of the instrument's direct readout shall be included for cyanide if the instrumentation has the capability.

2.5.2.3.2 The order of raw data in the Sample Data Package for inorganic analyses shall be: ICP-AES, Graphite Furnace Atomic Absorption (GFAA), ICP-MS, Mercury, and Cyanide. All raw data shall include concentration units for ICP, and absorbances or concentration units for Mercury and Cyanide.

2.5.2.3.3 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.5.2.3.4 Raw data shall be labeled with EPA sample numbers and appropriate codes, shown in Exhibit B, Table 2 - Codes for Labeling Data, following, to unequivocally identify:

- Calibration standards, including source and preparation date. Standard preparation logbooks can be submitted if they contain this information;
- Initial and Continuing Calibration Blanks (ICBs/CCBs) and Preparation Blanks (PBs);
- Initial and Continuing Calibration Verification (ICV/CCV) standards, Interference Check Samples (ICSs), serial dilution samples, Contract Required Quantitation Limit (CRQL) Check Standard (CRI), LCS, and post digestion spike;
- Diluted and undiluted samples (by EPA sample number) and all weights, dilutions, and volumes used to obtain the reported values (if the volumes, weights, and dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters is sufficient);
- Duplicates;
- Spikes (indicating standard solutions used, final spike concentrations, and volumes involved). If spike information (source, concentration, volume) is consistent

for a given SDG, a general statement outlining these parameters is sufficient;

- Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation; and

- Time and date of each analysis. Instrument run logs can also be submitted if they contain time and date of analysis. If the instrument does not automatically provide times of analysis, these shall be manually entered on all raw data (e.g., ICV/CCV, blanks, and the CRQL Check Standard).

Table 2
Codes for Labeling Data[1,2]

Sample	XXXXXX
Sample Not Part of the SDG	ZZZZZZ
Duplicate	XXXXXXD
Matrix Spike	XXXXXXS
Serial Dilution	XXXXXXL
Analytical Spike/Post Digestion/Distillation Spike	XXXXXXA
Instrument Calibration Standards:	
ICP	S or S0 for blank standard
Atomic Absorption and Cyanide	S0, S10,...etc.
Initial Calibration Verification	ICV
Initial Calibration Blank	ICB
Continuing Calibration Verification	CCV
Continuing Calibration Blank	CCB
Interference Check Samples:	
Solution A	ICSA
Solution AB	ICSAB
CRQL Check Standard	CRI
Laboratory Control Samples:	
Aqueous (Water)	LCSW
Solid (Soil/Sediment)	LCSS
Preparation Blank (Water)	PBW
Preparation Blank (Soil)	PBS
Linear Range Analysis Standard	LRS
Baseline Correction	BASELINE
Reslope	RESLOPE
Cyanide Mid-Range Standard	MIDRANGE
ICP-MS Tune Check	TUNE

[1]The numeric suffix that follows the "S" suffix for the standards indicates the true value of the concentration of the standard in ug/L.

[2]ICP-AES and ICP-MS calibration standards usually consist of several analytes at different concentrations. Therefore, no numeric suffix can follow the ICP calibration standards unless all the analytes in the standard are prepared at the same concentrations. For instance, the blank for ICP shall be formatted "S0".

2.5.2.4 Digestion and Distillation Logs. The following logs shall be submitted as appropriate for each preparation procedure: digestion logs for ICP-AES, ICP-MS, mercury preparations, and cyanide. These logs shall include: (1) date; (2) sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; (3) sufficient information to unequivocally identify which QC samples (i.e., LCS, PB) correspond to each batch digested; (4) comments describing any significant sample changes or reactions which occur during preparation shall be entered in the log and noted in the SDG Narrative; (5) indication of pH less than 2 or greater than 12, as applicable; and (6) identification of the sample preparer(s) [signature(s)].

2.6 Complete SDG File (CSF)

As specified in the Delivery Schedule, one CSF (including the original Sample Data Package) shall be delivered to the Region concurrently with the delivery of a copy of the Sample Data Package to SMO. Delivery to USEPA's designated recipient (e.g., QATS) is only required upon written request.

2.6.1 The CSF shall contain all original documents where possible. No photocopies of original documents shall be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to USEPA with another Case/SDG in accordance with the requirements described in Exhibit F. The CSF shall contain all original documents and be numbered according to the specifications in Exhibit B, Sections 3 and 4, and Form DC-2.

2.6.2 The CSF shall consist of the following original documents in addition to the documents in the Sample Data Package.

NOTE: All Case-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other Case-specific documents generated after the CSF is sent to USEPA, as well as copies that are altered in any fashion, are also deliverables to USEPA. Send the original to the Region and a copy to SMO. Send to USEPA's designated recipient (e.g., QATS) only upon written request.

2.6.2.1 Original Sample Data Package

2.6.2.2 A completed and signed Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

2.6.2.3 All original shipping documents, including, but not limited to, the following documents:

- USEPA Sample Traffic Reports/Chain of Custody Records
- Airbills (if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information); and
- Sample Tags (if present) sealed in plastic bags.

2.6.2.4 All original receiving documents, including, but not limited to, the following documents:

- Form DC-1;
- Other receiving forms or copies of receiving logbooks; and
- SDG Cover Sheet.

2.6.2.5 All original laboratory records of sample transfer, preparation, and analysis, including, but not limited to, the following documents:

- Original preparation and analysis forms or copies of preparation and analysis logbook pages; and
- Internal sample and sample digestate and distillate transfer Chain of Custody Records.

2.6.2.6 All other original SDG-specific documents in the possession of the laboratory, including, but not limited to, the following documents:

- Telephone contact logs;
- Copies of personal logbook pages;
- All handwritten SDG-specific notes; and
- Any other SDG-specific documents not covered by the above.

2.6.3 If the Contractor does submit SDG-specific documents to USEPA after submission of the CSF, the documents shall be numbered as an addendum to the CSF and a revised Form DC-2 shall be submitted; or the documents shall be numbered as a new CSF and a new Form DC-2 shall be submitted to the Region only.

2.6.4 The Contractor shall retain a legible electronic (PDF) or hard copy of the CSF for 365 days after submission of the reconciled data package. After this time, the Contractor may dispose of the package.

2.7 Data in Computer-Readable Format

The Contractor shall provide a computer-readable copy for all samples in the SDG, as specified in Exhibit H, and delivered as specified in Exhibit B, Section 1.1. Computer-readable data deliverables shall be submitted on DOS formatted 3.5-inch high density 1.44 MB diskette(s) (or via an alternate means of electronic transmission, if approved in advance by USEPA).

2.7.1 When submitted, diskette(s) shall be packaged and shipped in such a manner that the diskette(s) cannot be bent or folded and will not be exposed to extreme heat/cold or any type of electromagnetic radiation. The diskette(s) shall be included in the same shipment as the hardcopy data, and, at a minimum, be enclosed in a diskette mailer.

2.7.2 The data shall be recorded in the file format and adhere to the file, record, and field specifications listed in Exhibit H, "Data Dictionary and Format for Data Deliverables in Computer-Readable Format".

2.8 Results of the Intercomparison and Performance Evaluation (PE) Sample Analyses

Tabulation of analytical results for intercomparison/PE sample analyses includes all requirements specified in Exhibit B, Sections 2.5 and 2.7.

2.9 Preliminary Results

The Form Is data results (including all appropriate qualifiers and flags) shall be submitted for all samples in one SDG of a Case. Sample analysis shall follow all requirements stipulated in Exhibit D. The Contractor shall clearly identify the Preliminary Results by labeling each Form I as "Preliminary Results" under the form title (e.g., under Inorganic Analysis Data Sheet). The Contractor shall also include a disclaimer in the "Comments" field on all Form Is stating that the "Data results contained on this Form I are for scanning purposes only, and may not have been validated for CLP criteria." Sample Traffic Reports/Chain of Custody Records and SDG Cover Sheets shall be submitted with the Preliminary Results.

2.9.1 The Contractor shall submit the Cover Page following the specifications in Exhibit B, Sections 2.5.1 and 3.4.1. The Cover Page shall be clearly labeled to indicate that the data being reported are Preliminary Results. The Cover Page shall contain the following statement, verbatim: **"I certify that these Preliminary Results are in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature."** This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

2.10 Quarterly Verification of Linear Ranges and Interelement Correction Factors and Annual Verification of MDLs

The Contractor shall perform and report quarterly verification of instrument linear range and annual verification of MDLs by the methods specified in Exhibit D for each instrument used under this contract. The Contractor shall also perform and report quarterly ICP-AES interelement correction factors (including method of determination), wavelengths used, and integration times. Forms reporting results for quarterly and annual verification of method and instrument parameters for the current quarter and year shall be submitted in each Sample Data Package, using Inorganic Forms IX, XA, XB, and XI. Submission of the quarterly and annual verification of method and instrument parameters shall include the raw data used to determine the values reported.

2.11 Electronic Instrument Data

The Contractor shall adhere to the requirements in Exhibit E.

2.12 Corrective Action Procedures

If the Contractor fails to adhere to the requirements detailed in this SOW, the Contractor will be in noncompliance with the contract and may be subjected to sanctions as described in the contract.

3.0 FORM INSTRUCTIONS

3.1 Introduction

This section contains specific instructions for the completion of all required Inorganic Data Reporting Forms.

3.2 General Information

Values shall be reported on the hardcopy forms according to the respective form instructions in this section. Each form submitted shall be filled out completely for all analytes before proceeding to the next form of the same type. Do not submit multiple forms if the information on those forms can be submitted on one form.

3.2.1 The data reporting forms discussed in Exhibit B, Section 3.4, and presented in Exhibit B, Section 4.0, have been designed in conjunction with the computer-readable data formats specified in Exhibit H, "Data Dictionary and Format for Data Deliverables in Computer-Readable Format". The specific length of each variable for computer-readable data transmission purposes is given in Exhibit H. Information entered on these forms shall **not** exceed the size of the field given on the form, including such laboratory-generated items as "Lab Name" and "Lab Sample ID".

NOTE: On the hardcopy forms, the space provided for entries is greater in some instances than the length prescribed for the variable as written to the electronic deliverable (see Exhibit H). Greater space is provided on the hardcopy forms for the sake of visual clarity.

3.2.2 All characters which appear on the data reporting forms presented in the contract shall be reproduced by the Contractor when submitting data, and the format of the forms submitted shall be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM). The names of various fields and analytes (i.e., "Lab Code", "Aluminum") shall appear as they do on the forms in the contract, including the options specified in the form (i.e., "Matrix (soil/water):" shall appear, not just "Matrix").

3.2.3 Alphabetic entries made onto the forms by the Contractor shall be in ALL UPPERCASE letters (i.e., "LOW", not "Low" or "low"). If an entry does not fill the entire blank space provided on the form, null characters shall be used to remove the remaining underscores that comprise the blank line (see Exhibit H for additional instructions). However, do **not** remove the underscores or vertical bar characters that delineate "boxes" on the forms.

3.3 Header Information

Six pieces of information are common to the header sections of each data reporting form. These are: Laboratory Name, Contract, Laboratory Code, Case number, Non-Routine Analytical Services (NRAS) number, and Sample Delivery Group (SDG) number. Except as noted for NRAS number, this information shall be entered on every form and shall match on all forms.

3.3.1 Laboratory Name. The "Lab Name" shall be the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.

3.3.2 Contract. The "Contract" is the number of the USEPA contract under which the analyses were performed.

3.3.3 Laboratory Code. The "Lab Code" is an alphabetic abbreviation of up to six characters, assigned by USEPA, to identify the laboratory and aid in data processing. This laboratory code will be assigned by USEPA at the time a contract is awarded. The laboratory code shall not be modified by the Contractor, except at the direction of USEPA. If a change of name or ownership occurs at the laboratory, the laboratory code will remain the same until the Contractor is directed by USEPA to use another laboratory code.

3.3.4 Case Number. The "Case No." is the SMO-assigned Case number (to five characters) associated with the sample, and reported on the Traffic Report/Chain of Custody Record.

3.3.5 NRAS Number. The "NRAS No." is the USEPA assigned number for analyses performed under Non-Routine Analytical Services (NRAS). If samples are to be analyzed under NRAS only, and reported on these forms, then enter the NRAS number and leave the Case number blank. If samples are analyzed according to the Routine Analytical Services (RAS) protocol and have additional NRAS requirements, list both the Case number and NRAS number on all forms. If the analyses have no NRAS requirements, leave the "NRAS No." field blank.

3.3.6 SDG Number. The "SDG No." is the Sample Delivery Group (SDG) number. The SDG number is the EPA sample number of the first sample received in the SDG, except when this would cause duplication. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. If fractions of the same field samples are scheduled under different turnaround times, thus creating separate SDGs containing the same sample numbers, a different sample number shall be utilized in the assignment of the SDG number for each SDG. If a situation arises where there are an insufficient number of samples for assignment of SDG numbers, the contractor shall contact SMO for the assignment of a SDG number.

3.3.7 Sample Number. The "EPA Sample No." appears either in the header information of the form or as the left column of a table summarizing data from a number of samples. When an EPA sample number is entered in the triple-spaced box in the upper right-hand corner of a form, it shall be centered on the middle line of the three lines that form the box.

3.3.7.1 **All** samples, matrix spikes, post digestion/distillation spikes, duplicates, and serial dilutions shall be identified with an EPA sample number. For samples, an EPA sample number is the unique identifying number given in the Traffic Report/Chain of Custody Record that accompanied that sample. In order to facilitate data assessment, the sample suffixes listed in Exhibit B, Table 2 - Codes for Labeling Data, must be used.

3.3.8 Other Common Fields. Other pieces of information are common to many of the data reporting forms. These include Matrix and Level.

- For "Matrix", enter "SOIL" for soil/sediment samples and "WATER" for water samples.

  NOTE: The matrix must be spelled out. Abbreviations such as "S" or "W" shall **not** be used.

- For "Level", enter the determination of concentration level. Enter as "LOW" or "MED", **not** "L" or "M".

3.3.9 Rounding Rule. For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is greater than or equal to 5, the absolute value of the result is to be rounded up; otherwise the absolute value of the result is rounded down. For example, -0.4365 rounds to -0.437 and -2.3564 rounds to -2.356. Also see "Rounding Rules" in Exhibit G.

3.3.9.1 Before evaluating a number for being in control or out of control of a certain limit [other than the Contract Required Quantitation Limit (CRQL)], the number evaluated shall be rounded using the above rounding rules to the significance reported for that limit. For example, the control limit for an Initial Calibration Verification is plus or minus 10% of the true value. Then a calculated percent recovery of 110.46 shall be reported on Form IIA-IN as 110, which is within the control limits of 90-110. On the other hand, a calculated percent recovery of 110.50 shall be reported on Form IIA-IN as 111, which is not within the 90-110 percent control limits.

NOTE: All results shall be transcribed to Inorganic Forms IIA-IN through XV-IN from the raw data to the specified number of decimal places that are described in Exhibits B and H. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. If there are not enough figures in the raw data result to enter in the specified space for that result, then zeros shall be used for decimal places to the specified number of reporting decimals for that result for a specific form. The following examples are provided:

Raw Data Result	Specified Format	Correct Entry on Form
95.99653	5.4 (to four decimal places)	95.9965
95.99653	5.3 (to three decimal places)	95.997
95.99653	5.2 (to two decimal places)	96.00
95.996	5.4 (to four decimal places)	95.9960
95.9	5.4 (to four decimal places)	95.9000

3.4 Inorganic Forms

3.4.1 Cover Page - [COVER PAGE]

3.4.1.1 Purpose. This form is used to list all samples analyzed within an SDG and provide certain analytical information and general comments. It is also the document that is signed by the Laboratory Manager to authorize and release all data and deliverables associated with the SDG.

3.4.1.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.1.2.1 For samples analyzed using this Statement of Work (SOW), enter "ILM05.3" for the SOW Number.

3.4.1.2.2 Enter an EPA sample number including spikes and duplicates (to seven spaces) of every sample analyzed within the SDG. Spikes shall contain an "S" suffix and duplicates a "D" suffix. These sample numbers shall be listed on the form in ascending alphanumeric order. Thus, if MAB123 is the lowest (considering both alpha and numeric characters) EPA sample number within the SDG, it would be entered in the first EPA sample number field. Samples would be listed below it, in ascending sequence - MAB124, MAB125, MAC111, MA1111, MA1111D, etc.

3.4.1.2.3 A maximum of 20 field sample numbers (excluding PE samples) can be entered on this form. Submit additional Cover Pages, as appropriate, if the total number of samples, duplicates, and spikes in the SDG is greater than 22.

3.4.1.2.4 A Laboratory Sample ID (to ten spaces) may be entered for each EPA sample number. If a Laboratory Sample ID is entered, it shall be entered identically (for each EPA sample number) on all associated data.

3.4.1.2.5 Enter "YES" or "NO" in answer to each of the two questions concerning Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) corrections. Each question shall be explicitly answered with a "YES" or a "NO". The third question shall be answered with a "YES" or "NO" if the answer to the second question is "YES". It shall be left blank if the answer to the second question is "NO".

3.4.1.2.6 Under "Comments", enter any statements relevant to the analyses performed under the SDG as a whole.

3.4.1.2.7 Each Cover Page shall be signed and dated, in original, by the Laboratory Manager or the Manager's designee to authorize the release and verify the contents of all data and deliverables associated with an SDG.

3.4.2 Inorganic Analysis Data Sheet [Forms IA-IN and IB-IN]

3.4.2.1 Purpose. These forms are used to tabulate and report sample analysis results for inorganic target analytes (see Exhibit C).

3.4.2.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.2.2.1 "Date Received" is the date (formatted MM/DD/YYYY) of sample receipt at the laboratory, as recorded on the Traffic Report/Chain of Custody Record [i.e., the Validated Time of Sample Receipt (VTSR)].

3.4.2.2.2 "% Solids" is the percent of solids on a weight-by-weight basis in the sample which is determined by drying the sample as specified in Exhibit D - Introduction to Analytical Methods, Section 1.6. Report percent solids to one decimal place (i.e.,

5.3%). If the percent solids is not required because the sample is fully aqueous, or is less than 1% solid, then enter "0.0".

3.4.2.2.3 Enter the appropriate concentration units (UG/L for water or MG/KG dry weight for soil). Entering "MG/KG" means "mg/kg dry weight" on this form.

3.4.2.2.4 Under the column labeled "Concentration", enter for each analyte, the value of the result [if the concentration is greater than or equal to the Method Detection Limit (MDL)] corrected for any dilutions; or, enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. The concentration result shall be reported to two significant figures if the result is less than 10 or three significant figures if the value is greater than or equal to 10.

3.4.2.2.5 Under the columns labeled "C", "Q", and "M", enter result qualifiers as identified below. If additional qualifiers are used, their explicit definitions shall be included on the Cover Page in the "Comments" section.

Forms IA-IN and IB-IN include fields for three types of result qualifiers. These qualifiers shall be completed as follows:

3.4.2.2.5.1 C (Concentration) Qualifier. Enter "J" if the reported value was obtained from a reading that was less than the CRQL but greater than or equal to the MDL. If the reading was less than the MDL, a "U" shall be entered.

The MDL obtained for a given preparation method, analysis method, and instrument shall be used for qualification of the results for samples associated with that preparation method, analysis method, and instrument. Serial dilution and post-digestion spike results shall be qualified using the MDL and CRQL values utilized for the corresponding field sample.

All three values (i.e., the instrument reading, CRQL, and MDL) shall be converted to the same units prior to determining the appropriate C (Concentration) Qualifier.

NOTE: The water CRQL (in ug/L) and the MDL obtained from direct analysis (Preparation Method "NP1") for a given analysis method and instrument shall be used to qualify the results of instrument QC standards that are not taken through a preparation procedure (e.g., ICB, CCB, and CRI for ICP-AES].

3.4.2.2.5.2 Q Qualifier. Specified entries and their meanings are as follows:

E: The reported value is estimated due to the presence of interference. An explanatory note shall be included under "Comments" on the Cover Page (if the problem applies to all samples), or on the specific Form IA-IN or Form IB-IN (if it is an isolated problem).

N: Spiked sample recovery not within control limits.

*: Duplicate analysis not within control limits.

D: The reported value is from a dilution.

3.4.2.2.5.3 M (Analysis Method) Qualifier. Specified entries and their meanings are as follows:

P:	ICP-AES
MS:	ICP-MS
CV:	Manual Cold Vapor Atomic Absorption (AA)
AV:	Automated Cold Vapor AA
AS:	Semi-Automated Spectrophotometric
C:	Manual Spectrophotometric
" ":	Where no data have been entered
NR:	If the analyte is not required to be analyzed

3.4.2.2.6 A brief physical description of the sample, both before and after digestion, shall be reported in the fields for color (before and after), clarity (before and after), texture, and artifacts. For water samples, report color and clarity. For soil samples, report color, texture, and artifacts. The following descriptive terms are recommended:

- Color - red, blue, yellow, green, orange, violet, white, colorless, brown, grey, and black;
- Clarity - clear, cloudy, and opaque; and
- Texture - fine (powdery), medium (sand), and coarse (large crystals or rocks).

If artifacts are present, enter "YES" in the artifacts field and describe the artifacts in the "Comments" field. If artifacts are not present, leave this field blank. Note any significant changes that occur during sample preparation (i.e., emulsion formation) in the "Comments" field. Enter any sample-specific comments concerning the analyte results in the "Comments" field. Also document raw instrument results that are less than minus two times the CRQL (-2xCRQL) in the "Comments" field and in the Sample Delivery Group (SDG) Narrative.

3.4.2.2.7 If more than two additional analytes were requested, submit Form IB-IN as appropriate.

3.4.3 Initial (ICV) and Continuing Calibration Verification (CCV) [Form IIA-IN]

3.4.3.1 Purpose. This form is used to report analyte recoveries from calibration verification solutions.

3.4.3.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.3.2.1 Enter the ICV Source (12 characters maximum) and the CCV Source (12 characters maximum). Enter sufficient information in the available 12 spaces to identify the manufacturer and the solution used.

Use additional Form(s) IIA-IN if more calibration verification sources were used.

3.4.3.2.2 Under "Initial Calibration Verification True", enter the value [in micrograms per Liter (ug/L), to one decimal place] of the concentration of each analyte in the ICV Solution.

3.4.3.2.3 Under "Initial Calibration Verification Found", enter the most recent value (in ug/L, to two decimal places), of the concentration of each analyte measured in the ICV Solution.

3.4.3.2.4 Under "Initial Calibration Verification %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 1 ICV Percent Recovery

$$\%R = \frac{\text{Found(I CV)}}{\text{True(ICV)}} x100$$

WHERE, "True(ICV)" is the true concentration of the analyte in the ICV Solution and "Found(ICV)" is the found concentration of the analyte in the ICV Solution.

The values used in EQ. 1 for "True(ICV)" and "Found(ICV)" shall be exactly those reported on this form.

3.4.3.2.5 Under "Continuing Calibration Verification True", enter the value (in ug/L, to one decimal place) of the concentration of each analyte in the CCV Solution.

3.4.3.2.6 Under "Continuing Calibration Verification Found", enter the value (in ug/L, to two decimal places) of the concentration of each analyte measured in the CCV Solution.

NOTE: The form contains two "Continuing Calibration Verification Found" columns. The column to the left shall contain values for the first CCV, and the column to the right shall contain values for the second CCV.

3.4.3.2.7 If more than one Form IIA-IN is required to report multiple CCVs, then the column to the left on the second form shall contain values for the third CCV, the column to the right shall contain values for the fourth CCV, and so on.

3.4.3.2.8 Under "Continuing Calibration Verification %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 2 CCV Percent Recovery

$$\%R = \frac{\text{Found(CCV)}}{\text{True(CCV)}} x100$$

WHERE, "True(CCV)" is the true concentration of each analyte, and "Found(CCV)" is the found concentration of the analyte in the CCV Solution.

The values used in EQ. 2 for "True(CCV)" and "Found(CCV)" shall be exactly those reported on this form.

NOTE: The form contains two “Continuing Calibration Verification %R” columns. Entries to these columns shall follow the sequence detailed above for entries to the “Continuing Calibration Verification Found” columns.

3.4.3.2.9 Under “M”, enter the method used or “NR”, as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.3.2.10 If more than one wavelength/mass is used to analyze an analyte, submit additional Form(s) IIA-IN as appropriate.

3.4.3.2.11 The order of reporting ICVs and CCVs for each analyte shall follow the chronological order in which the standards were run. Start with the first Form IIA-IN and move from the left to the right, continuing to the following Form IIA-INs as appropriate. For instance, the first ICV for all analytes shall be reported on the first Form IIA-IN. In a run where three CCVs were analyzed, the first CCV shall be reported in the left CCV column on the first Form IIA-IN and the second CCV shall be reported in the right column of the same form. The third CCV shall be reported in the left CCV column of the second Form IIA-IN. On the second Form IIA-IN, the ICV column and the right CCV column shall be left empty in this example. In the previous example, if a second run for an analyte was needed, the ICV of that run shall be reported on a third Form IIA-IN and the CCVs follow in the same fashion as explained before. In the case where two wavelengths are used for an analyte, all ICV and CCV results of one wavelength from all runs shall be reported before proceeding to report the results of the second wavelength used.

3.4.4 CRQL Check Standard [Form IIB-IN]

3.4.4.1 Purpose. This form is used to report analyte recoveries from analyses of the CRQL Check Standards (CRIs).

3.4.4.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.4.2.1 Enter the CRQL Check Standard Source (12 characters maximum) as explained in Exhibit B, Section 3.4.3.2.1.

3.4.4.2.2 Under “CRQL Check Standard True”, enter the value (in ug/L, to one decimal place) of the concentration of each analyte in the CRQL Check Standard that was analyzed for analytical samples associated with the SDG.

3.4.4.2.3 Under “CRQL Check Standard Initial Found”, enter the result (in ug/L, to two decimal places) measured in the CRQL Check Standard analyzed at the beginning of the run. For each analyte, enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL of the analyte if the concentration is less than the MDL. If applicable, enter the concentration qualifier “J” or “U” after the concentration (e.g., 1.96J for Lead), as specified in Exhibit B, Section 3.4.2.2.5.1.

3.4.4.2.4 Under “CRQL Check Standard Initial %R”, enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 3 CRQL Check Standard Initial Percent Recovery

$$\%R = \frac{\text{CRQL Check Standard Initial Found}}{\text{CRQL Check Standard True}} x100$$

3.4.4.2.5 Under “CRQL Check Standard Final Found”, enter the results (in ug/L, to two decimal places) measured in the CRQL Check Standard(s) analyzed after the beginning of the run. For each analyte, enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL of the analyte if the concentration is less than the MDL. If applicable, enter the concentration qualifier “J” or “U” after the concentration (e.g., 1.96J for Lead), as specified in Exhibit B, Section 3.4.2.2.5.1.

3.4.4.2.6 Under “CRQL Check Standard Final %R”, enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 4 CRQL Check Standard Final Percent Recovery

$$\%R = \frac{\text{CRQL Check Standard Final Found}}{\text{CRQL Check Standard True}} x100$$

3.4.4.2.7 All percent recovery values reported in EQs. 3 and 4 shall be calculated using the exact true and found values reported on this form. A value of zero shall be used in calculations if the analyte value is less than the MDL.

NOTE: For every initial solution reported there must be a final one. However, the opposite is <u>not</u> true. If a CRQL Check Standard was required to be analyzed in the middle of a run, it shall be reported in the “Final Found” section of this form.

3.4.4.2.8 If more CRI analyses were required or analyses were performed using more than one wavelength per analyte, submit additional Form(s) IIB-IN as appropriate.

3.4.4.2.9 The order of reporting CRIs for each analyte shall follow the chronological order in which the standards were run starting with the first Form IIB-IN and continuing to the following Forms IIB-IN as appropriate. When multiple wavelengths are used for one analyte, all the results of one wavelength shall be reported before proceeding to the next wavelength.

3.4.5 Blanks [Form III-IN]

3.4.5.1 Purpose. This form is used to report analyte concentrations found in the Initial Calibration Blank (ICB), Continuing Calibration Blanks (CCB), and the Preparation Blank (PB).

3.4.5.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.5.2.1 Enter “SOIL” or “WATER” as appropriate as the matrix of the PB. No abbreviations or other matrix descriptors may be used.

3.4.5.2.2 According to the matrix specified for the PB, enter the PB concentration units as "UG/L" for water or "MG/KG" for soil.

3.4.5.2.3 Under "Initial Calibration Blank", enter the concentration (in ug/L, to three decimal places) of each analyte in the most recent ICB, as described in Exhibit B, Section 3.4.5.2.8, below.

3.4.5.2.4 For each calibration blank associated with a given method and instrument, enter "J" under the "C" qualifier field on Form III-IN if the absolute value of the analyte concentration is less than the CRQL for water but greater than or equal to the MDL that was obtained from direct analysis (Preparation Method "NP1") using that method and instrument.

For prepared calibration blanks (e.g., mercury), the CRQL for water and the MDL for the preparation method, analysis, and instrument shall be used.

Enter "U" if the absolute value of the analyte in the blank is less than the MDL obtained from direct analysis or the preparation method.

3.4.5.2.5 Under "Continuing Calibration Blank 1", enter the concentration (in ug/L, to three decimal places) of each analyte detected in the first required CCB analyzed after the ICB, as described in Exhibit B, Section 3.4.5.2.8, below. Enter any appropriate qualifier, as explained for the "Initial Calibration Blank", to the "C" qualifier column immediately following the "Continuing Calibration Blank 1" column.

3.4.5.2.6 If up to three CCBs were analyzed, complete the columns labeled "2" and "3" in accordance with the instructions for the "Continuing Calibration Blank 1" column. If more than three CCBs were analyzed, then complete additional Form(s) III-IN as appropriate.

3.4.5.2.7 Under "Preparation Blank", enter the concentration in ug/L (to three decimal places) for a water blank, or mg/kg (to three decimal places) for a soil blank, of each analyte in the PB, as described in Exhibit B, Section 3.4.5.2.8, below. Evaluate the absolute value of the analyte concentration to determine the appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, and enter the qualifier in the "C" column immediately following the "Preparation Blank" column.

3.4.5.2.8 For all blanks, enter the concentration (positive or negative) for each analyte, if the absolute value of the concentration is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration is less than the appropriate MDL.

For example, arsenic has a MDL of 3 ug/L for Preparation Method "NP1" [CRQL for arsenic is 10 ug/L (water)]. Therefore, a CCB instrument reading of -4.2485 ug/L will be reported as -4.249J; a CCB instrument reading of -2.4356 ug/L will be reported as 10.000U; a CCB instrument reading of 4.3586 ug/L will be reported as 4.359J; and a CCB instrument reading of 2.1584 ug/L will be reported as 10.000U.

3.4.5.2.9 Under "M", enter the method used, as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.5.2.10 If more than one wavelength/mass is used to analyze an analyte, submit additional Form(s) III-IN as appropriate.

3.4.5.2.11 The order of reporting ICBs and CCBs for each analyte shall follow the chronological order in which the blanks were run starting with the first Form III-IN and moving from left to right and continuing to additional Forms III-IN. When multiple wavelengths are used for the analysis of one analyte, all the results of one wavelength shall be reported before proceeding to the next wavelength.

3.4.6 ICP-AES and ICP-MS Interference Check Sample (ICS) [Forms IVA-IN and IVB-IN]

3.4.6.1 Purpose. These forms are used to report ICS results for each ICP-AES or ICP-MS instrument used in SDG analyses.

3.4.6.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions. The instructions for Forms IVA-IN and IVB-IN are identical except where specified.

3.4.6.2.1 For “ICP Instrument ID”, enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two ICP instruments within a laboratory may have the same ICP Instrument ID.

3.4.6.2.2 Enter “ICS Source” (12 characters maximum) as explained in Exhibit B, Section 3.4.3.2.1. For USEPA solutions, include in the source name a number identifying it (e.g., EPA-LV87).

3.4.6.2.3 Under “True Sol. A”, enter the true concentration (in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte present in Solution A. Enter “0” for each analyte with no specified true value in Solution A.

3.4.6.2.4 Under “True Sol. AB”, enter the true concentration (in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte present in Solution AB. Enter “0” for each analyte with no specified true value in Solution AB.

3.4.6.2.5 Under “Initial Found Sol. A” on Form IVA-IN (ICP-AES), and “Found Sol. A” on Form IVB-IN (ICP-MS), enter the concentration (positive, negative, or zero, in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10). Enter the concentration of each analyte and interferent for ICP-AES and of each analyte for ICP-MS in the initial analysis of Solution A as required in Exhibit D.

3.4.6.2.6 Under “Initial Found Sol. A %R” on Form IVA-IN (ICP-AES), and “Found Sol. A %R” on Form IVB-IN (ICP-MS), enter the value (to the nearest whole number) of the percent recovery computed for true Solution A greater than zero according to the following equation:

EQ. 5 Initial Found Sol. A Percent Recovery

$$\%R = \frac{\text{Initial Found Solution A}}{\text{True Found Solution A}} x100$$

Leave the field blank if "True Solution A" equals zero.

3.4.6.2.7 Under "Initial Found Sol. AB" on Form IVA-IN (ICP-AES), and "Found Sol. AB" on Form IVB-IN (ICP-MS), enter the concentration (positive, negative, or zero, in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte and interferent for ICP-AES and of each analyte for ICP-MS in the initial analysis of Solution AB as required in Exhibit D.

3.4.6.2.8 Under "Initial Found Sol. AB %R" on Form IVA-IN (ICP-AES), and "Found Sol. AB %R" on Form IVB-IN (ICP-MS), enter the value (to the nearest whole number) of the percent recovery computed for True Solution AB greater than zero according to the following equation:

EQ. 6 Initial Found Sol. AB Percent Recovery

$$\%R = \frac{\text{Initial Found Solution A}}{\text{True Solution A}} x100$$

Leave the field blank if "True Solution AB" equals zero.

3.4.6.2.9 Under "Final Found Sol. A", enter the concentration (positive, negative, or zero, in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte and interferent for ICP-AES in the final analysis of Solution A as required in Exhibit D. ICP-MS analysis (Form IVB-IN) does not require a final analysis.

3.4.6.2.10 Under "Final Found Sol. A %R" enter the value (to the nearest whole number) of the percent recovery computed for true Solution A greater than zero according to the following equation:

EQ. 7 Final Found Sol. A Percent Recovery

$$\%R = \frac{\text{Final Found Solution A}}{\text{True Solution A}} x100$$

Leave the field blank if "True Solution A" equals zero.

3.4.6.2.11 Under "Final Found Sol. AB", enter the concentration (positive, negative, or zero, in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte and interferent for ICP-AES in the final analysis of Solution AB as required in Exhibit D. ICP-MS analysis (Form IVB-IN) does not require a final analysis.

3.4.6.2.12 For all found values of Solutions A and AB, enter the concentration (positive, negative, or zero) of each analyte and interferent at each wavelength used for analysis by ICP.

3.4.6.2.13 Under “Final Found Sol. AB %R”, enter the value (to the nearest whole number) of the percent recovery computed for true Solution AB greater than zero according to the following equation:

EQ. 8 Final Found Sol. AB Percent Recovery

$$\%R = \frac{\text{Final Found Solution AB}}{\text{True Solution AB}} x100$$

Leave the field empty if “True Solution AB” equals zero.

All percent recovery values reported shall be calculated using the exact true and found values reported on this form.

NOTE: For ICP-AES (Form IVA-IN), for every initial solution reported there must be a final solution reported. However, the opposite is not true. If an ICS was required to be analyzed in the middle of a run, it shall be reported in the “Final Found” section of this form.

3.4.6.2.14 If more ICS analyses were required, submit additional Form(s) IVA-IN and/or IVB-IN as appropriate.

3.4.6.2.15 The order of reporting ICSs for each analyte shall follow the chronological order in which the standards were run, starting with the first Form IVA-IN and/or IVB-IN and continuing to the following Forms IV-IN as appropriate. When multiple wavelengths/masses are used for one analyte, all the results of one wavelength/mass shall be reported before proceeding to the next wavelength/mass.

3.4.7 Matrix Spike Sample Recovery [Form VA-IN]

3.4.7.1 Purpose. This form is used to report results for the pre-digest spike.

3.4.7.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.7.2.1 Indicate the appropriate matrix, level, and concentration units (ug/L for water and mg/kg dry weight for soil) as explained in Exhibit B, Sections 2.5.2.1.1 and 3.3.8.

3.4.7.2.2 For “% Solids for Sample”, enter the percent solids (see Exhibit B, Section 3.4.2.2.2) for the original sample of EPA sample number reported on the form. Note that this number must equal the one reported on Form IA-IN for that sample.

3.4.7.2.3 In the “EPA Sample No.” box, enter an EPA sample number (7 places maximum) of the sample from which the spike results on this form were obtained. The number shall be centered in the box.

3.4.7.2.4 Under “Control Limit %R”, enter “75-125” if the sample result is less than or equal to four times the spike added value. If

the sample result is greater than four times the Spike Added (SA) value, leave this field empty.

3.4.7.2.5 Under "Spiked Sample Result (SSR)", enter the measured value (to four decimal places), in appropriate units, for each relevant analyte in the matrix spike sample. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Spiked Sample Result (SSR)" column.

3.4.7.2.6 Under "Sample Result (SR)", enter the measured value (to four decimal places) for each required analyte in the sample (reported in "EPA Sample No." box) on which the matrix spike was performed. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Sample Result (SR)" column.

3.4.7.2.7 Under "Spike Added (SA)", enter the value (to two decimal places) for the concentration of each analyte added to the sample. The same concentration units shall be used for "SSR", "SR", and "SA". If the "Spike Added" concentration is specified in the contract, the value added and reported shall be the specific concentration in appropriate units, corrected for spiked sample weight and percent solids (soils) or spiked sample volume (waters).

3.4.7.2.8 Under "%R", enter the value (to the nearest whole number) of the percent recovery for all spiked analytes computed according to the following equation:

EQ. 9 Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} x100$$

Percent recovery shall be reported, whether it is negative, positive or zero.

The values for "SSR", "SR", and "SA" must be exactly those reported on this form. A value of zero shall be used in calculations for "SSR" or "SR" if the analyte value is less than the MDL.

3.4.7.2.9 Under "Q", enter "N" if the Spike Recovery (%R) is out of the control limits (75-125) and the Sample Result (SR) is less than or equal to four times the SA.

3.4.7.2.10 Under "M", enter the method used (as explained in Exhibit B, Section 3.4.2.2.5.3) or enter "NR" if the analyte is not required in the spike.

3.4.7.2.11 If different samples were used for spike sample analysis of different analytes, additional Form(s) VA-IN shall be submitted for each sample as appropriate.

3.4.8 Post-Digestion Spike Sample Recovery [Form VB-IN]

3.4.8.1 Purpose. This form is used to report results for the post-digest spike recovery which is based upon the addition of a known quantity of analyte to an aliquot of the digested sample.

3.4.8.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.8.2.1 In the "EPA Sample No." box, enter an EPA sample number (seven characters maximum) of the sample from which the spike results on this form were obtained. The number shall be centered in the box.

3.4.8.2.2 The "Control Limit %R" and "Q" fields shall be left blank until limits are established by USEPA. At that time, the Contractor will be informed how to complete these fields.

3.4.8.2.3 Under "Spiked Sample Result (SSR)", enter the measured value (in ug/L, to two decimal places) for each analyte in the post-digest spike sample. Enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL for the analyte if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Spiked Sample Result (SSR)" column.

3.4.8.2.4 Under "Sample Result (SR)", enter the measured value (in ug/L, to two decimal places) for the concentration of each analyte in the sample (reported in "EPA Sample No." box) on which the spike was performed. Enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL for the analyte if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Sample Result (SR)" column.

3.4.8.2.5 Under "Spike Added (SA)", enter the value (in ug/L, to one decimal place) for each analyte added to the sample. If the SA concentration is specified in the contract, the value added and reported shall be that specific concentration in appropriate units.

3.4.8.2.6 Under "%R", enter the value (to the nearest whole number) of the percent recovery for all spiked analytes computed according to EQ. 9 in Exhibit B, Section 3.4.7.2.8. Percent recovery shall be reported, whether it is negative, positive, or zero. The values for "SSR", "SR", and "SA" must be exactly those reported on this form. A value of zero shall be substituted for "SSR" or "SR" if the analyte value is less than the MDL.

3.4.8.2.7 Under "M", enter the method used as explained in Exhibit B, Section 3.4.2.2.5.3, or enter "NR" if the spike was not required.

3.4.8.2.8 If different samples were used for spike sample analysis of different analytes, additional Form(s) VB-IN shall be submitted.

3.4.9 Duplicates [Form VI-IN]

3.4.9.1 Purpose. The duplicates form is used to report results of duplicate analyses. Duplicate analyses are required for percent solids values and all analyte results.

3.4.9.2 Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.9.2.1 Indicate the appropriate matrix, level, and concentration units (ug/L for water and mg/kg dry weight for soil) as explained in Exhibit B, Sections 2.5.2.1.1 and 3.3.8.

3.4.9.2.2 For “% Solids for Sample”, enter the percent solids (as explained in Exhibit B, Section 3.4.2.2.2) for the original sample of the EPA sample number reported on the form. Note that this number must equal the one reported on Form IA-IN for that sample.

3.4.9.2.3 For “% Solids for Duplicate”, enter the percent solids (as explained in Exhibit B, Section 3.4.2.2.2) for the duplicate sample of the EPA sample number reported on the form.

3.4.9.2.4 In the “EPA Sample No.” box, enter EPA sample number (seven characters maximum) of the sample from which the duplicate sample results on this form were obtained. The number shall be centered in the box.

3.4.9.2.5 Under “Control Limit”, enter the CRQL (in appropriate units, ug/L for water or mg/kg dry weight basis corrected for the original sample weight and percent solids) for the analyte if either the sample or duplicate value was less than 5 times the CRQL. If the sample and duplicate values were greater than or equal to 5 times the CRQL, or if the sample and duplicate values were less than the CRQL, leave the field empty.

3.4.9.2.6 Under “Sample (S)”, enter the original measured value (to four decimal places) for the concentration of each analyte in the sample (reported in “EPA Sample No.” box) on which a duplicate analysis was performed. Concentration units are those specified on the form. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the “C” qualifier column immediately following the “Sample (S)” column.

3.4.9.2.7 Under “Duplicate (D)”, enter the measured value (to four decimal places) for each analyte in the duplicate sample. Concentration units are those specified on the form. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in

Exhibit B, Section 3.4.2.2.5.1, to the “C” qualifier column immediately following the “Duplicate (D)” column.

3.4.9.2.8 For solid samples, the concentration of the original sample shall be computed using the weight and percent solids of the original sample. The concentration of the duplicate sample shall be computed using the weight of the duplicate sample, but the percent solids of the original sample.

3.4.9.2.9 Under “RPD”, enter the absolute value (to the nearest whole number) of the Relative Percent Difference (RPD) for all analytes detected above the MDL in either the sample or the duplicate, computed according to the following equation:

EQ. 10 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S + D)/2} x100$$

The values for “S” and “D” shall be exactly those reported on this form. A value of zero shall be substituted for “S” or “D” if the analyte concentration is less than the MDL in either one. If the analyte concentration is less than the MDL in both “S” and “D”, leave the “RPD” field empty.

3.4.9.2.10 Under “Q”, enter “*” if the duplicate analysis for the analyte is out of control. If both sample and duplicate values are greater than or equal to 5 times the CRQL, then the RPD must be less than or equal to 20% to be in control. If either the sample or duplicate value is less than 5 times the CRQL, then the absolute difference between the sample and duplicate values shall be less than the CRQL to be in control.

3.4.9.2.11 If both values are below the CRQL, then no control limit is applicable.

3.4.9.2.12 Under “M”, enter method used as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.10 Laboratory Control Sample [Form VII-IN]

3.4.10.1 Purpose. This form is used to report results for the solid and aqueous LCSs.

3.4.10.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.10.2.1 For the Solid LCS Source (12 characters maximum), enter the appropriate EPA sample number if EPA provided standard was used. Substitute an appropriate number provided by EPA for LCS solutions prepared in the future. If other sources were used, identify the source. For the aqueous LCS Source, enter the source name (12 characters maximum) as explained in Exhibit B, Section 3.4.3.2.1.

3.4.10.2.2 Under “Aqueous True”, enter the value (in ug/L, to one decimal place) of the concentration of each analyte in the Aqueous LCS Standard Source.

3.4.10.2.3 Under “Aqueous Found”, enter the measured concentration (in ug/L, to two decimal places) of each analyte found in the Aqueous LCS solution.

3.4.10.2.4 Under “Aqueous %R”, enter the value of the percent recovery (to the nearest whole number) computed according to the following equation:

EQ. 11 Aqueous LCS Percent Recovery

$$\%R = \frac{\text{Solid LCS Found}}{\text{Solid LCS True}} x100$$

3.4.10.2.5 Under “Solid True”, enter the value (in mg/kg, to one decimal place) of the concentration of each analyte in the solid LCS Source.

3.4.10.2.6 Under “Solid Found”, enter the measured value (in mg/kg, to one decimal place) of each analyte found in the solid LCS solution. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL.

3.4.10.2.7 Under “C”, enter “J” or “U” or leave empty, to describe the found value of the solid LCS as explained in Exhibit B, Section 3.4.2.2.5.1.

3.4.10.2.8 Under “Limits”, enter the lower limit (in mg/kg, to one decimal place) in the left column, and the upper limit (in mg/kg, to one decimal place) in the right column, for each analyte in the solid LCS solution.

3.4.10.2.9 Under “Solid %R”, enter the value of the percent recovery (to the nearest whole number) computed according to the following equation:

EQ. 12 Solid LCS Percent Recovery

$$\%R = \frac{\text{Solid LCS Found}}{\text{Solid LCS True}} x100$$

3.4.10.2.10 The values for true and found aqueous and solid LCSs used in EQs. 11 and 12 shall be exactly those reported on this form. If the analyte concentration is less than the MDL, a value of zero shall be substituted for the aqueous and solid LCS found.

3.4.10.2.11 Submit additional Form(s) VII-IN as appropriate if more than one aqueous LCS or solid LCS was required.

3.4.11 ICP-AES and ICP-MS Serial Dilutions [Form VIII-IN]

3.4.11.1 Purpose. This form is used to report results for ICP-AES and ICP-MS serial dilutions.

3.4.11.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.11.2.1 In the "EPA Sample No." box, enter EPA sample number (7 places maximum) of the sample for which serial dilution analysis results on this form were obtained. The number shall be centered in the box.

3.4.11.2.2 Under "Initial Sample Result (I)", enter the instrument measured value (in ug/L to two decimal places) for each ICP analyte. This value shall not be corrected for any dilution. Enter the instrument measured value (if the concentration is greater than or equal to the MDL); or enter the CRQL if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Initial Sample Result (I)" column.

NOTE: The initial sample concentration for an analyte does not have to equal the value for that analyte reported on Form IA-IN for that sample. It is the value of the analyte's instrument measured value (uncorrected for dilution) that is within the linear range of the instrument.

3.4.11.2.3 Under "Serial Dilution Result (S)", enter the instrument measured value corrected for a five-fold dilution (in ug/L to two decimal places) for each ICP analyte in the diluted sample. Enter the corrected instrument measured value (if the concentration is greater than or equal to the MDL); or enter the CRQL if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Serial Dilution Result (S)" column.

NOTE: The "Serial Dilution Result (S)" is obtained by multiplying by five the instrument measured value (in ug/L) of the serially diluted sample. The "C" qualifier for the serial dilution shall be established based on the serial dilution result before correcting it for the five-fold dilution regardless of the value reported on Form VIII-IN.

For example, if the instrument readout value for the "Initial Sample Result (I)" for silver in a two-fold diluted sample MAX123 is 1164.36 ug/L, and the instrument readout value for the "Serial Dilution Result (S)" for silver in a ten-fold diluted sample MAX123 (MAX123L) is 241.67 ug/L, then the concentration reported for silver in the "Initial Sample Result (I)" column will be 1164.36 ug/L (not 2 times the instrument readout value which equals 2328.72 ug/L), and the concentration reported for silver in the "Serial Dilution Result (S)" column will be five times the instrument readout value which equals 1208.35 ug/L (not 10 times the instrument readout value which equals 2416.70 ug/L).

3.4.11.2.4 Under “% Difference”, enter the absolute value (to the nearest whole number) of the percent difference in concentration of required analytes, between the original sample and the diluted sample (adjusted for dilution) according to the following formula:

EQ. 13 Serial Dilution Percent Difference

$$\%\text{Difference} = \frac{|I - S|}{I} x100$$

The values for “I” and “S” used to calculate percent difference in EQ. 13 shall be exactly those reported on this form. A value of zero shall be substituted for “S” if the analyte concentration is less than the MDL. If the analyte concentration in (I) is less than the MDL concentration, leave the “% Difference” field empty.

3.4.11.2.5 Under “Q”, enter “E” if the percent difference is greater than 10% and the original sample concentration (reported on Form IA-IN) is greater than 50 times the MDL reported on Form IX-IN.

3.4.11.2.6 Under “M”, enter the method of analysis for each analyte as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.12 Method Detection Limits (Annually) [Form IX-IN]

3.4.12.1 Purpose. This form documents the Method Detection Limits (MDLs) for each preparation method and instrument that the Contractor used to obtain data for the SDG. Only the methods, instruments, and wavelengths used to generate data for the SDG shall be included. The Contractor shall also report MDLs, obtained from direct analysis, for each instrument used to obtain data for the SDG. The MDLs obtained from direct analysis shall be used in the qualification of data associated with samples and instrument QC standards that are not taken through a preparation procedure. Although the MDLs are determined annually, a copy of the annual MDLs shall be included with each Sample Data Package on Forms IX-IN.

3.4.12.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.12.2.1 Enter the Analysis Method qualifier as specified in Exhibit B, Section 3.4.2.2.5.3, in the “Instrument Type” field.

3.4.12.2.2 Enter the Instrument ID in the “Instrument ID” field (12 characters maximum). These instrument IDs are used to uniquely identify each instrument that the laboratory used to perform the analysis.

3.4.12.2.3 Enter the date (formatted MM/DD/YYYY) on which the MDL analysis was performed in the “Date” field.

3.4.12.2.4 For “Preparation Method”, enter the method of preparation (three characters maximum) for which the MDLs listed on Form IX-IN were established. Use appropriate sample preparation codes as specified below:

HW1: Hotplate/Block digestion for ICP-AES analysis of water samples.
HW2: Hotplate/Block digestion for ICP-MS analysis of water samples.
HW3: Hotplate/Block digestion for ICP-MS analysis of water samples.
MW1: Microwave digestion for ICP-AES analysis of water samples.
MW2: Microwave digestion for ICP-AES analysis of water samples.
HS1: Hotplate/Block digestion for ICP-AES analysis of soil samples.
HS2: Hotplate/Block digestion for ICP-AES analysis of soil samples.
MS1: Microwave digestion for ICP-AES analysis of soil samples.
CW1: Preparation for the Manual Cold Vapor AA analysis of water samples.
CS1: Preparation for the Manual Cold Vapor AA analysis of soil samples.
CW2: Preparation for the Automated Cold Vapor AA analysis of water samples.
DW1: Distillation for the manual and semi-automated spectrophotometric analysis of water samples.
DW2: Midi-distillation for the semi-automated spectrophotometric analysis of water samples.
DS1: Distillation for the manual and semi-automated spectrophotometric analysis of soil samples.
DS2: Midi-distillation for the semi-automated spectrophotometric analysis of soil samples.
NP1: No preparation.

3.4.12.2.5 Enter the concentration units (UG/L for water or MG/KG wet weight for soil) for the results reported on Form IX-IN in the "Concentration Units" field. Enter "UG/L" for MDL results obtained from direct analysis (Preparation Method "NP1").

3.4.12.2.6 Under "Wavelength/Mass", enter the wavelength in nanometers (nm) to two decimal places or the mass in atomic mass units (amu) to two decimal places for each analyte for which an MDL has been established and is listed in the MDL column. If more than one wavelength or mass is used for an analyte, use additional Form(s) IX-IN as appropriate to report the MDL.

3.4.12.2.7 Contract Required Quantitation Limits (in ug/L for water and mg/kg for soil) as established in Exhibit C, shall be reported in the column headed "CRQL". The CRQL shall be reported in ug/L on Form(s) IX-IN associated with Preparation Method "NP1".

3.4.12.2.8 Under "MDL", enter the MDL (in ug/L for water and direct analysis, or mg/kg for soil, to two significant figures for values less than 10, and three significant figures for values greater than or equal to 10) as determined by the Contractor for each analyte analyzed by the instrument for which the ID is listed on this form. When calculating MDL values, always round up to the appropriate significant figure (e.g., 14.81 rounds to 14.9 and 146.6 rounds to 147). This deviation from the rounding rule is necessary to prevent the reporting of detected values for results that fall in the noise region of the calibration curve.

NOTE: Zeros used to set the decimal point in a number less than one are not significant but all trailing zeros are significant.

For example, a calculated MDL value of 0.074 ug/L will be reported as 0.074 and a calculated MDL value of 0.1 or 0.08 will be reported as 0.10 and 0.080, respectively.

3.4.12.2.9 Use additional Form(s) IX-IN if more preparation methods, instruments and wavelengths or masses are used. Note that the date on this form shall not exceed the analysis dates in the Sample Data Package or precede them by more than twelve months.

3.4.12.2.10 Use the “Comments” section to indicate alternative wavelengths and the conditions under which they are used.

3.4.13 ICP-AES Interelement Correction Factors (Quarterly) [Form XA-IN]

3.4.13.1 Purpose. This form documents for each ICP-AES instrument the interelement correction factors applied by the Contractor to obtain data for the SDG. Although the correction factors are determined quarterly, a copy of the results of the quarterly interelement correction factors shall be included with each Sample Data Package on Form XA-IN and Form XB-IN as appropriate.

3.4.13.2 Instructions. Complete the header information according to instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.13.2.1 Enter the ICP-AES Instrument ID (12 characters maximum), which is a unique number designated by the Contractor to identify each ICP-AES instrument used to produce data in the Sample Data Package. If more than one ICP-AES instrument is used, submit additional Form(s) XA-IN as appropriate.

3.4.13.2.2 Report the date (formatted as MM/DD/YYYY) on which these correction factors were determined for use. This date shall not exceed the ICP-AES analysis dates in the Sample Data Package or precede them by more than three calendar months.

3.4.13.2.3 Under “Wavelength”, list the wavelength in nm (to two decimal places) used for each ICP-AES analyte. If more than one wavelength is used, submit additional Form(s) XA-IN or Form(s) XB-IN as appropriate.

3.4.13.2.4 Under “Al”, “Ca”, “Fe”, and “Mg”, enter the correction factor (negative, positive or zero, to seven decimal places, 10 characters maximum) for each ICP-AES analyte. Correction factors for one other analyte shall be reported using the empty column and listing the analyte’s chemical symbol in the blank two-space header field provided for that column.

3.4.13.2.5 If corrections are not applied for an analyte, a zero shall be entered for that analyte to indicate that the corrections were determined to be zero. Correction factors for more than one additional analyte shall be reported using Form XB-IN.

NOTE: Correction factors for Al, Ca, Fe, and Mg are all required and are to be listed first (as they appear on Form XA-IN).

3.4.14 ICP-AES Interelement Correction Factors (Quarterly) [Form XB-IN]

3.4.14.1 Purpose. This form is used if correction factors for analytes other than Al, Ca, Fe, Mg, and one more analyte of the Contractor's choice were applied to the analytes analyzed by ICP-AES.

3.4.14.2 Instructions. Complete this form following the instructions for Form XA-IN (see Exhibit B, Section 3.4.13) by listing the chemical symbol for additional analytes in the heading of the empty columns in the two-space fields provided.

3.4.14.2.1 Columns of correction factors for additional analytes shall be entered left to right starting on Form XA-IN and proceeding to Form XB-IN, according to the alphabetical order of their chemical symbols.

3.4.15 ICP-AES and ICP-MS Linear Ranges (Quarterly) [Form XI-IN]

3.4.15.1 Purpose. This form documents the quarterly linear range analysis for each ICP instrument that the Contractor used to obtain data for the SDG.

3.4.15.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.15.2.1 Enter the ICP Instrument ID (12 characters maximum), which is a unique number designated by the Contractor to identify each ICP instrument used to produce data for the SDG. If more than one ICP instrument is used, submit additional Form(s) XI-IN as appropriate.

3.4.15.2.2 Report the date (formatted as MM/DD/YYYY) on which these linear ranges were analyzed. This date shall not exceed the dates of analysis by ICP in the Sample Data Package and shall not precede the analysis dates by more than three calendar months.

3.4.15.2.3 Under "Integ. Time (Sec.)", enter the integration time (in seconds to two decimal places) used for each measurement taken from the ICP instrument.

3.4.15.2.4 Under "Concentration", enter the concentration (in ug/L) that is the upper limit of the ICP instrument linear range as determined in Exhibit D. Any measurement above it is out of the linear range, and thus, is an estimated value and shall be diluted into the linear range.

3.4.15.2.5 Under "M", enter the method of analysis for each analyte as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.15.2.6 If more instruments or analyte wavelengths/masses are used, submit additional Form(s) XI-IN as appropriate.

3.4.16 Preparation Log [Form XII-IN]

3.4.16.1 Purpose. This form is used to report the preparation run log.

3.4.16.1.1 All field samples and all Quality Control (QC) preparations (including duplicates, matrix spikes, LCSs, PBs, and re-preparations) associated with the SDG shall be reported on Form XII-IN. In addition, for mercury analyses, all prepared

calibration standards and QC standards (e.g., ICV, CCV, ICB, CCB, CRI) shall also be reported on Form XII-IN. For cyanide analyses, the distilled ICV and the mid-range standard shall also be reported on Form XII-IN.

3.4.16.1.2 Submit one Form XII-IN per batch, per method, if no more than thirty-two preparations, including QC preparations, were performed. If more than 32 preparations per batch, per method, were performed, then submit additional copies of Form XII-IN as appropriate. Submit a separate Form XII-IN for each batch.

3.4.16.1.3 The order in which the Preparation Logs are submitted is very important. Form XII-IN shall be organized by method, by batch. Later batches within a method shall follow earlier ones. Each batch shall start on a separate Form XII-IN.

3.4.16.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.16.2.1 For “Preparation Method”, enter the method of preparation (three characters maximum) for which the preparations listed on Form XII-IN were made, as specified in Exhibit B, Section 3.4.12.2.4.

3.4.16.2.2 Under “EPA Sample No.”, enter EPA sample number of each sample in the SDG, and of all other preparations such as duplicates, matrix spikes, LCSs, PBs, and re-preparations (all formatted according to Exhibit B, Table 2). All EPA sample numbers shall be listed in ascending alphanumeric order, continuing to the next Form XII-IN if applicable.

3.4.16.2.3 Under “Preparation Date”, enter the date (formatted MM/DD/YYYY) on which each sample was prepared for analysis by the method indicated in the header section of the form.

NOTE: The date never changes on a single Form XII-IN because the form shall be submitted per batch.

3.4.16.2.4 Under “Weight”, enter the wet weight (in grams, to two decimal places) of each soil sample prepared for analysis by the method indicated in the header section of the form. If the sample matrix is water, then leave the field empty.

3.4.16.2.5 Under “Volume”, enter the final volume (in mL, to the nearest whole number) of the preparation for each sample prepared for analysis by the method indicated in the header section of the form. This field shall have a value for each sample listed.

3.4.17 Analysis Run Log [Form XIII-IN]

3.4.17.1 Purpose. This form is used to report the sample analysis run log.

3.4.17.1.1 A run is defined as the totality of analyses performed by an instrument throughout the sequence initiated by, and including, the first SOW-required calibration standard or tune standard, and terminated by, and including, the CCV and CCB following the last SOW-required analytical sample.

3.4.17.1.2 All field samples and all QC analyses (including tunes, calibration standards, ICVs, CCVs, ICBs, CCBs, CRIs, ICSs, LRSs, LCSs, PBs, duplicates, serial dilutions, matrix spikes,

and post-digestion/distillation spikes) associated with the SDG shall be reported on Form XIII-IN. The run shall be continuous and inclusive of all analyses performed on the particular instrument during the run.

3.4.17.1.3 Submit one Form XIII-IN per run if no more than thirty-two (32) analyses, including instrument calibration, were analyzed in the run. If more than thirty-two analyses were performed in the run, submit additional Form(s) XIII-IN as appropriate.

3.4.17.1.4 The order in which the Analysis Run Logs are submitted is very important. Form XIII-IN shall be organized by method, and by run. Later runs within a method shall follow earlier ones. Each analytical run shall start on a separate Form XIII-IN. Therefore, instrument calibration or tune shall be the first entry on the form for each new run. In addition, the run is considered to have ended if it is interrupted for any reason, including termination for failing QC parameters.

3.4.17.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.17.2.1 For "Instrument ID", enter the Instrument ID (12 characters maximum) which shall be an identifier designated by the Contractor to uniquely identify each instrument used to produce data which are required to be reported in the SDG deliverable. If more than one instrument is used, submit additional Form(s) XIII-IN as appropriate. The Instrument ID shall exactly match that reported on Forms IVA, IVB, IX, XA, XB, XI, XIV, and XV.

3.4.17.2.2 For "Analysis Method", enter the method code (two characters maximum) according to the specifications in Exhibit B, Section 3.4.2.2.5.3.

3.4.17.2.3 For "Start Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was started.

3.4.17.2.4 For "End Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was ended.

3.4.17.2.5 Under "EPA Sample No.", enter EPA sample number of each analysis, including all QC operations applicable to the SDG (formatted according to Exhibit B, Table 2). All EPA sample numbers shall be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XIII-IN for the instrument run, if applicable. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical run, shall be reported. Those analyses shall be identified with EPA sample number of "ZZZZZZ".

3.4.17.2.6 Under "D/F", enter the dilution factor (to two significant figures) by which the final digestate or distillate needed to be diluted for each analysis to be performed. The dilution factor does not include the dilution inherent in the preparation as specified by the preparation procedures in Exhibit D.

3.4.17.2.7 The dilution factor is required for all entries on Form XIII-IN.

NOTE: For a particular sample a dilution factor of “1.0” shall be entered if the digestate or distillate was analyzed without adding any further volume of dilutant or any other solutions to the “Volume” or an aliquot of the “Volume” listed on Form XII-IN for that sample.

3.4.17.2.8 For USEPA supplied solutions such as ICVs, ICSs, and LCSs, a dilution factor shall be entered if the supplied solution had to be diluted to a dilution different from that specified by the instructions provided with the solution. The dilution factor reported in such a case shall be that which would make the reported true values on the appropriate form for the solution equal those that were supplied with the solution by USEPA. For instance, ICV-2(0887) has a true value of 104.0 ug/L at a 20-fold dilution. If the solution is prepared at a 40-fold dilution, a dilution factor of "2.0" shall be entered on Form XIII-IN and the uncorrected instrument reading is compared to a true value of 52 ug/L. In this example, Form IIA-IN will have a true value of 104.0 regardless of the dilution used. The found value for the ICV shall be corrected for the dilution listed on Form XIII-IN using the following formula:

EQ. 14 ICV/CCV Correction for Dilution

$$\text{Found value on Form II} = \text{Instrument readout (ug/L)} \times D/F$$

3.4.17.2.9 Under “Time”, enter the time (in military format - HHMM) at which each analysis was performed.

3.4.17.2.10 Under “Analytes”, enter “X” in the column of the designated analyte to indicate that the analyte value was used from the reported analysis to report data in the SDG. Leave the column empty for each analyte if the analysis was not used to report the particular analyte.

3.4.17.2.11 Entering “X” appropriately is very important. The “X” is used to link the samples with their related QC. It also links the dilution factor with the appropriate result reported on Inorganic Forms I-VIII. For each analyte result reported on any of the Forms I-VIII, there shall be one, and only one, properly identified entry on Form XIII-IN for which an “X” is entered in the column for that analyte.

3.4.17.2.12 If, on Form XIII-IN, an “X” is entered in the column for an analyte for a field sample associated with a dilution factor greater than 1.0, flag the data for that analyte with a “D” on the appropriate Form IA-IN or Form IB-IN.

3.4.18 ICP-MS Tune [Form XIV-IN]

3.4.18.1 Purpose. This form is used to report the tuning results for each ICP-MS instrument used in SDG analyses.

3.4.18.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.18.2.1 For “ICP-MS Instrument ID”, enter an identifier that uniquely identifies a specific instrument within the Contractor

laboratory. No two ICP-MS instruments within a laboratory may have the same ICP-MS Instrument ID.

3.4.18.2.2 Report the date (formatted as MM/DD/YYYY) on which the ICP-MS tune was performed. This date shall not exceed the dates of analysis by ICP-MS in the Sample Data Package.

3.4.18.2.3 For "Avg. Measured Mass (amu)", enter the average mass calculated from the five or more tune analyses (in atomic mass units, to two decimals places) measured for each isotope.

3.4.18.2.4 For "Avg. Peak Width at Peak Height (amu)" enter the average peak width calculated from the analysis (in atomic mass units, to two decimal places) at the percent of peak height recommended by the instrument manufacturer for each isotope.

3.4.18.2.5 For "%RSD", enter the percent Relative Standard Deviation of the absolute signals (intensities) for each isotope calculated from the five or more tune analyses.

3.4.19 ICP-MS Internal Standards Relative Intensity Summary [Form XV-IN]

3.4.19.1 Purpose. This form is used to report the relative internal standard intensity levels during a run for ICP-MS. The relative intensity of each of the internal standards in all analyses performed by ICP-MS must be reported on the form. If more than one ICP-MS instrument or run is used, submit additional Form(s) XV-IN as appropriate. All runs for the lowest alphanumeric instrument must be reported in ascending order before proceeding to the runs for the next highest instrument.

3.4.19.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.19.2.1 For "ICP-MS Instrument ID", enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two ICP-MS instruments within a laboratory may have the same ICP-MS Instrument ID.

3.4.19.2.2 For "Start Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was started.

3.4.19.2.3 For "End Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was ended.

3.4.19.2.4 Under "EPA Sample No.", enter EPA sample number of each analysis, including all QC operations applicable to the SDG. All EPA sample numbers must be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XV for the instrument run, if applicable. The order must agree with the order reported on Form XIII-IN for that run. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical run, must be reported. Those analyses must be identified with EPA sample number of "ZZZZZZ." Samples identified as "ZZZZZZ" need not have intensities reported for internal standards.

3.4.19.2.5 Under "Time", enter the time (in military format - HHMM) at which each analysis was performed.

3.4.19.2.6 Under “Internal Standards %RI for:”, enter the chemical symbol and elemental expression number of the internal standard in the “Element” header field provided to indicate the internal standard and elemental expression for which the Relative Intensity (RI) of the internal standards will be calculated in that column.

3.4.19.2.6.1 In the “Element” column, enter the internal standard relative intensity (to the nearest whole number) of the internal standard for each sample analysis listed on the form (excluding “ZZZZZZ”). The internal standard relative intensity (%RI) is calculated using the following formula:

EQ. 15 Internal Standard Percent Relative Intensity

$$\%RI = \frac{I_n}{I_o} x100$$

WHERE, “I_o” is the intensity of the internal standard in the blank calibration standard and “I_n” is the intensity of the internal standard in the EPA sample number in the same units.

3.4.19.2.7 Under the “Q” column to the right of each “Element” column, enter an “R” if the %RI for a field sample, PE, duplicate, or spike is less than 60 or greater than 125; otherwise leave the field blank.

3.4.19.2.8 Columns of internal standard RI must be entered left to right starting with the internal standards of the lower mass on the first Form XV-IN and proceeding to the following Form XV-IN as appropriate. All Forms XV-IN for the lowest numeric instrument must be reported in ascending order by the run number before proceeding to the next Form XV.

3.4.19.3 All field samples and all QC samples (including calibration standards, ICVs, CCVs, ICBs, CCBs, CRIs, ICSs, LCS, PB, serial dilutions, duplicates, PE samples, and spikes) associated with the SDG must be reported on Form XV-IN. The run must be continuous and inclusive of all analyses performed on the particular instrument during the run.

3.4.19.4 Submit one Form XV-IN per run if no more than 32 analyses, including instrument calibration, were analyzed in the run. If more than 32 analyses were performed in the run, submit additional Form(s) XV-IN as appropriate. Each new run must be started on the first line of Form XV-IN.

3.5 Sample Log-In Sheet [Form DC-1]

3.5.1 Purpose. This form is used to document the receipt and inspection of samples and containers. At least one original Form DC-1 is required for each sample shipping container (e.g., cooler). If the samples in a single sample shipping container must be assigned to more than one SDG, the original Form DC-1 shall be placed with the deliverables for the SDG of the lowest alpha-numeric number and a copy of Form DC-1 shall be placed with the deliverables for the other SDG(s). The copies should be identified as “copy(ies)”, and the location of the original should be noted on the copies.

3.5.2 Instructions

3.5.2.1 Sign and date the airbill. (If an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information).

3.5.2.2 Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in Item 1.

3.5.2.3 Record the custody seal numbers in Item 2.

3.5.2.4 Open the container, remove the enclosed sample documentation, and record the presence/absence of USEPA forms (i.e., Traffic Reports/Chain of Custody Records, packing lists) and airbills or airbill stickers in Items 3 and 4. Specify if there is an airbill present or an airbill sticker in Item 4. Record the airbill or sticker number in Item 5.

3.5.2.5 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample bottles (i.e., intact, broken, leaking) and presence or absence of sample tags in Items 6 and 7.

3.5.2.6 Record the presence or absence of a cooler temperature indicator bottle in Item 8.

3.5.2.7 Record the cooler temperature in Item 9.

3.5.2.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and mark the appropriate answer in Item 10.

3.5.2.9 The log-in date should be recorded at the top of Form DC-1; record the date and time of cooler receipt at the laboratory in Items 11 and 12.

3.5.2.10 If there are no problems observed during receipt, sign and date (include the time) Form DC-1 and Traffic Report/Chain of Custody Record, and write the sample numbers in the "EPA Sample No." column.

3.5.2.11 Record the pH for all aqueous samples received.

3.5.2.12 Record the appropriate sample tags and assigned laboratory numbers, if applicable.

3.5.2.13 Any comments should be made in the "Remarks" column.

3.5.2.14 Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the "Sample Transfer" block located in the bottom left corner of Form DC-1. Sign and date the sample transfer block.

3.5.2.15 For Items 1, 3, 4, 6, 7, 8 and 10, circle the appropriate response. Responses can be underlined if this form is completed by automated equipment. Unused columns and spaces shall be crossed out, initialed, and dated.

3.5.2.16 If there are problems observed during receipt (including samples that have not been preserved to the proper pH) or an answer marked

with an asterisk (e.g., “absent*”) was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

3.6 Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

3.6.1 Purpose. The CSF Inventory Sheet is used to record both the inventory of Complete SDG File (CSF) documents and the number of documents in the original Sample Data Package which is sent to the USEPA Region.

3.6.2 Instructions

3.6.2.1 Organize all EPA-CSF documents as described in Exhibit B, Sections 2 and 3. Assemble the documents in Exhibit B, Section 2 in the order specified on Form DC-2, and stamp each page with the consecutive number. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record in the “Comments” section on Form DC-2 all intentional gaps in the page numbering sequence (for example, “page numbers not used, XXXX-XXXX, XXXX-XXXX”). If there are no documents for a specific document type, enter an “NA” in the empty space.

3.6.2.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review Form DC-2 to determine if it is most appropriate to place them under Categories 33, 34, 35, or 36. Category 36 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

3.6.2.3 If it is necessary to insert new or inadvertently omitted documents, the Contractor shall follow these steps:

- Number all documents to be inserted with the next sequential numbers and file the inserts in their logical positions within the CSF (e.g., file document 1000 between documents 6 and 7).

- Identify where the inserts are filed in the CSF by recording the document numbers and their locations under the “Other Records” section of Form DC-2 (e.g., document 1000 is filed between 6 and 7).

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

EXHIBIT B

INORGANIC FORMS

COVER PAGE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

SOW No.: _____

EPA Sample No.	Lab Sample ID

		ICP-AES	ICP-MS
Were ICP-AES and ICP-MS interelement corrections applied?	(Yes/No)	_____	_____
Were ICP-AES and ICP-MS background corrections applied?	(Yes/No)	_____	_____
If yes, were raw data generated before application of background corrections?	(Yes/No)	_____	_____

Comments:

_____

_____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

1A-IN
INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Level: (low/med) _____ Date Received: _____

% Solids: _____

Concentration Units (**ug/L** or **mg/kg** dry weight): _____

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum				
7440-36-0	Antimony				
7440-38-2	Arsenic				
7440-39-3	Barium				
7440-41-7	Beryllium				
7440-43-9	Cadmium				
7440-70-2	Calcium				
7440-47-3	Chromium				
7440-48-4	Cobalt				
7440-50-8	Copper				
7439-89-6	Iron				
7439-92-1	Lead				
7439-95-4	Magnesium				
7439-96-5	Manganese				
7439-97-6	Mercury				
7440-02-0	Nickel				
7440-09-7	Potassium				
7782-49-2	Selenium				
7440-22-4	Silver				
7440-23-5	Sodium				
7440-28-0	Thallium				
7440-62-2	Vanadium				
7440-66-6	Zinc				
57-12-5	Cyanide				

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

_____
_____
_____
_____

1B-IN
INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Level: (low/med) _____ Date Received: _____

% Solids: _____

Concentration Units (**ug/L** or **mg/kg** dry weight): _____

CAS No.	Analyte	Concentration	C	Q	M

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

_____
_____
_____
_____

2A-IN
INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Initial Calibration Verification Source: _____

Continuing Calibration Verification Source: _____

Concentration Units: ug/L

	Initial Calibration Verification			Continuing Calibration Verification					M
Analyte	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

(1) Control Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

2B-IN
CRQL CHECK STANDARD

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

CRQL Check Standard Source: _____

Concentration Units: ug/L

Analyte	CRQL Check Standard				
	Initial			Final	
	True	Found*	%R(1)	Found*	%R(1)
Aluminum					
Antimony					
Arsenic					
Barium					
Beryllium					
Cadmium					
Calcium					
Chromium					
Cobalt					
Copper					
Iron					
Lead					
Magnesium					
Manganese					
Mercury					
Nickel					
Potassium					
Selenium					
Silver					
Sodium					
Thallium					
Vanadium					
Zinc					
Cyanide					

(1) Control Limits: 70-130 with the following exceptions:
ICP-AES - Antimony, Lead, and Thallium: 50-150.
ICP-MS - Cobalt, Manganese, and Zinc: 50-150.

* If applicable, enter the concentration qualifier “J” or ”U” after the concentration in these columns (e.g., 0.20U for Mercury).

3-IN
BLANKS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Preparation Blank Matrix (soil/water): _____

Preparation Blank Concentration Units (ug/L or mg/kg): _____

Analyte	Initial Calibration Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Preparation Blank	C	M
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide											

4A-IN
ICP-AES INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-AES Instrument ID: _____ ICS Source: _____

Concentration Units: ug/L

Analyte	True		Initial Found				Final Found			
	Sol. A	Sol. AB	Sol. A	%R	Sol. AB	%R	Sol. A	%R	Sol. AB	%R
Aluminum										
Antimony										
Arsenic										
Barium										
Beryllium										
Cadmium										
Calcium										
Chromium										
Cobalt										
Copper										
Iron										
Lead										
Magnesium										
Manganese										
Nickel										
Potassium										
Selenium										
Silver										
Sodium										
Thallium										
Vanadium										
Zinc										

4B-IN
ICP-MS INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ ICS Source: _____

Concentration Units: ug/L

Analyte	True		Found			
	Sol. A	Sol. AB	Sol. A	%R	Sol. AB	%R
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Carbon						
Chloride						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Molybdenum						
Nickel						
Phosphorus						
Potassium						
Selenium						
Silver						
Sodium						
Sulfur						
Thallium						
Titanium						
Vanadium						
Zinc						

5A-IN
MATRIX SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

% Solids for Sample: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

_____
_____
_____
_____

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5B-IN
POST-DIGESTION SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

Concentration Units: ug/L

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

_____
_____
_____
_____

6-IN
DUPLICATES

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

% Solids for Sample: _____ % Solids for Duplicate: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

7-IN
LABORATORY CONTROL SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Solid LCS Source: _____

Aqueous LCS Source: _____

	Aqueous (ug/L)			Solid (mg/kg)					
Analyte	True	Found	%R	True	Found	C	Limits		%R
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

8-IN
ICP-AES and ICP-MS SERIAL DILUTIONS

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

Concentration Units: ug/L

Analyte	Initial Sample Result (I)	C	Serial Dilution Result (S)	C	% Difference	Q	M
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							

9-IN
METHOD DETECTION LIMITS (ANNUALLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Instrument Type: _____ Instrument ID: _____ Date: _____

Preparation Method: _____

Concentration Units (ug/L or mg/kg): _____

Analyte	Wavelength /Mass	CRQL	MDL
Aluminum			
Antimony			
Arsenic			
Barium			
Beryllium			
Cadmium			
Calcium			
Chromium			
Cobalt			
Copper			
Iron			
Lead			
Magnesium			
Manganese			
Mercury			
Nickel			
Potassium			
Selenium			
Silver			
Sodium			
Thallium			
Vanadium			
Zinc			
Cyanide			

Comments:

10A-IN
ICP-AES INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-AES Instrument ID: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for: Al	Ca	Fe	Mg	___
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

_____
_____
_____
_____

10B-IN
ICP-AES INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-AES Instrument ID: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for: ____	____	____	____	____
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

_____
_____
_____
_____

11-IN
ICP-AES and ICP-MS LINEAR RANGES (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP Instrument ID: _____ Date: _____

Analyte	Integ. Time (Sec.)	Concentration (ug/L)	M
Aluminum			
Antimony			
Arsenic			
Barium			
Beryllium			
Cadmium			
Calcium			
Chromium			
Cobalt			
Copper			
Iron			
Lead			
Magnesium			
Manganese			
Nickel			
Potassium			
Selenium			
Silver			
Sodium			
Thallium			
Vanadium			
Zinc			

Comments:

_____
_____
_____
_____

12-IN
PREPARATION LOG

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Preparation Method: _____

EPA Sample No.	Preparation Date	Weight (gram)	Volume (mL)

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13-IN
ANALYSIS RUN LOG

Lab Name: ______________________________ Contract: ____________________

Lab Code: _______ Case No.: _______ NRAS No.: _________ SDG No.: _________

Instrument ID: ____________ Analysis Method: _______________

Start Date: _____________ End Date: ______________

EPA Sample No.	D/F	Time	Analytes																									
			AL	SB	AS	BA	BE	CD	CA	CR	CO	CU	FE	PB	MG	MN	HG	NI	K	SE	AG	NA	TL	V	ZN	CN		

14-IN
ICP-MS Tune

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ Date: _____

Element - Mass	Avg. Measured Mass (amu)	Avg. Peak Width at Peak Height (amu)	%RSD
Be - 9			
Mg - 24			
Mg - 25			
Mg - 26			
Co - 59			
In - 113			
In - 115			
Pb - 206			
Pb - 207			
Pb - 208			

Comments:

_____

_____

_____

_____

15-IN
ICP-MS Internal Standards Relative Intensity Summary

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ Start Date: _____ End Date: _____

EPA Sample No.	Time	Internal Standards %RI For:									
		Element	Q	Element	Q	Element	Q	Element	Q	Element	Q

SAMPLE LOG-IN SHEET

Lab Name					Page __ of __
Received By (Print Name)					Log-in Date
Received By (Signature)					
Case Number		Sample Delivery Group No.			NRAS Number

Remarks:		EPA Sample #	Aqueous Sample pH	Corresponding		Remarks: Condition of Sample Shipment, etc.
				Sample Tag #	Assigned Lab #	
1. Custody Seal(s)	Present/Absent* Intact/Broken					
2. Custody Seal Nos.	_____ _____					
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*					
4. Airbill	Airbill/Sticker Present/Absent*					
5. Airbill No.	_____ _____					
6. Sample Tags	Present/Absent*					
Sample Tag Numbers	Listed/Not Listed on Traffic Report/Chain of Custody Record					
7. Sample Condition	Intact/Broken*/ Leaking					
8. Cooler Temperature Indicator Bottle	Present/Absent*					
9. Cooler Temperature	_____					
10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree?	Yes/No*					
11. Date Received at Lab	_____					
12. Time Received	_____					
Sample Transfer						
Fraction	Fraction					
Area #	Area #					
By	By					
On	On					

* Contact SMO and attach record of resolution

Reviewed By		Logbook No.
Date		Logbook Page No.

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____

CITY/STATE _____

CASE NO. _____ SDG NO. _____

SDG NOs. TO FOLLOW _____ _____ _____

NRAS NO. _____

CONTRACT NO. _____

SOW NO. _____

All documents delivered in the Complete SDG File must be original documents where possible. (Reference - Exhibit B Section 2.6)

		PAGE NOs.		CHECK	
		FROM	TO	LAB	REGION
1.	Cover Page				
2.	SDG Narrative				
3.	Sample Log-In Sheet (DC-1)				
4.	Inventory Sheet (DC-2))				
5.	Traffic Report/Chain of Custody Record(s)				
	Inorganic Analysis				
6.	Data Sheet (Form I-IN)				
7.	Initial & Continuing Calibration Verification (Form IIA-IN)				
8.	CRQL Standard (Form IIB-IN)				
9.	Blanks (Form III-IN)				
10.	ICP-AES Interference Check Sample (Form IVA-IN)				
11.	ICP-MS Interference Check Sample (Form IVB-IN)				
12.	Matrix Spike Sample Recovery (Form VA-IN)				
13.	Post-Digestion Spike Sample Recovery (Form VB-IN)				
14.	Duplicates (Form VI-IN)				
15.	Laboratory Control Sample (Form VII-IN)				
16.	ICP-AES and ICP-MS Serial Dilutions (Form VIII-IN)				
17.	Method Detection Limits (Annually) (Form IX-IN)				
18.	ICP-AES Interelement Correction Factors (Quarterly) (Form XA-IN)				
19.	ICP-AES Interelement Correction Factors (Quarterly) (Form XB-IN)				
20.	ICP-AES and ICP-MS Linear Ranges (Quarterly) (Form XI-IN)				
21.	Preparation Log (Form XII-IN)				

	PAGE NOs. FROM	TO	CHECK LAB	REGION
22. Analysis Run Log (Form XIII-IN)	____	____	____	____
23. ICP-MS Tune (Form XIV-IN)	____	____	____	____
24. ICP-MS Internal Standards Relative Intensity Summary (Form XV-IN)	____	____	____	____
25. ICP-AES Raw Data	____	____	____	____
26. GFAA Raw Data (If Applicable)	____	____	____	____
27. ICP-MS Raw Data	____	____	____	____
28. Mercury Raw Data	____	____	____	____
29. Cyanide Raw Data	____	____	____	____
30. Preparation Logs Raw Data	____	____	____	____
31. Percent Solids Determination Log	____	____	____	____
32. USEPA Shipping/Receiving Documents				
Airbill (No. of Shipments ____)	____	____	____	____
Sample Tags			____	____
Sample Log-In Sheet (Lab)	____	____	____	____
33. Misc. Shipping/Receiving Records (list all individual records)				
Telephone Logs	____	____	____	____
_____	____	____	____	____
_____	____	____	____	____
34. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)				
_____	____	____	____	____
_____	____	____	____	____
35. Internal Original Sample Prep & Analysis Records (describe or list)				
Prep Records _____	____	____	____	____
Analysis Records _____	____	____	____	____
Description _____	____	____	____	____
36. Other Records (describe or list)				
Telephone Communications Log	____	____	____	____
_____	____	____	____	____
_____	____	____	____	____

37. Comments: _____

_____

_____

_____

Completed by:

(CLP Lab) _____ _____ _____

(Signature) (Print Name & Title) (Date)

Audited by:

(USEPA) _____ _____ _____

(Signature) (Print Name & Title) (Date)

EXHIBIT C

INORGANIC TARGET ANALYTE LIST
WITH CONTRACT REQUIRED
QUANTITATION LIMITS

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EXHIBIT C - INORGANIC TARGET ANALYTE LIST WITH CONTRACT REQUIRED QUANTITATION LIMITS

Table of Contents

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1.0 INORGANIC TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)

Analyte	CAS Number	ICP-AES CRQL for Water[1,2,3,4] (µg/L)	ICP-AES CRQL for Soil[1,2,3,4,5] (mg/kg)	ICP-MS CRQL for Water[1,2,4] (µg/L)
Aluminum	7429-90-5	200	20	--
Antimony	7440-36-0	60	6	2
Arsenic	7440-38-2	10	1	1
Barium	7440-39-3	200	20	10
Beryllium	7440-41-7	5	0.5	1
Cadmium	7440-43-9	5	0.5	1
Calcium	7440-70-2	5000	500	--
Chromium	7440-47-3	10	1	2
Cobalt	7440-48-4	50	5	1
Copper	7440-50-8	25	2.5	2
Iron	7439-89-6	100	10	--
Lead	7439-92-1	10	1	1
Magnesium	7439-95-4	5000	500	--
Manganese	7439-96-5	15	1.5	1
Mercury	7439-97-6	0.2	0.1	--
Nickel	7440-02-0	40	4	1
Potassium	7440-09-7	5000	500	--
Selenium	7782-49-2	35	3.5	5
Silver	7440-22-4	10	1	1
Sodium	7440-23-5	5000	500	--
Thallium	7440-28-0	25	2.5	1
Vanadium	7440-62-2	50	5	1
Zinc	7440-66-6	60	6	2
Cyanide	57-12-5	10	2.5	--

[1]The CRQLs are the minimum levels of quantitation acceptable under the contract Statement of Work (SOW).

[2]Subject to the restrictions specified in Exhibit D, any analytical method specified in ILM05.3 Exhibit D may be utilized as long as the documented Method Detection Limits (MDLs) are less than one-half the CRQLs.

[3]Mercury is analyzed by cold vapor atomic absorption. Cyanide is analyzed by colorimetry/spectrophotometry.

[4]Changes to the Inorganic Target Analyte List (TAL) (e.g., adding an additional analyte) or CRQLs may be requested under the modified analysis clause in the contract.

[5]The CRQLs for soil are based on 100% solids and on the exact weights and volumes specified in Exhibit D. Samples with less than 100% solids may have CRQLs greater than those listed in the table above.

Request for Quote (RFQ) for Modified Analysis

Date: September 16, 2009

Subject: Modification Reference Number:
Title: Acid Volatile Sulfide/Simultaneously Extractable Metals
Sample Matrix: Soil/Sediment
Fraction Affected: Metals and Cyanide
Statement of Work: ILM05.4

Purpose:

The Contractor Laboratory is requested to perform the following modified analyses under the Inorganic Statement of Work (SOW) ILM05.4, based on the additional specifications listed below. Unless specifically modified by this modification, all analyses, Quality Control (QC), and reporting requirements specified in SOW ILM05.4 remain unchanged and in full force and effect. The number of samples requested in this modification is not guaranteed.

Please note that accepting a modified analysis request is voluntary, and that the Laboratory is not required to accept the modified analysis. There will be no adverse effect to the Laboratory for not accepting the modified analysis request. However, once the Laboratory accepts the request for modified analysis, it shall perform the analysis in accordance with this modification and as specified in SOW ILM05.4.

The Laboratory is requested to review the modification described herein, determine whether or not it shall accept the requested modified analyses, and complete the attached response form. The Laboratory shall provide comments in response to the required changes in the designated area, in order to ensure that the modified analysis can be completed in accordance with the specifications described herein.

Modification to the SOW Specifications:

The contract Laboratory shall analyze soil/sediment samples for Acid Volatile Sulfide/Simultaneously Extractable Metals (AVS/SEM) by method EPA-821-R-91-100 (December 2, 1991) which is available at NSCEP/NEPIS (http://www.epa.gov/nscep/) as method 821R91100 as indicated on the Traffic Report/Chain of Custody Record and the Laboratory Scheduling Notification form.

Acid Volatile Sulfide

The Contract Required Quantitation Limit (CRQL) for AVS is 0.5 mg/kg

The Contractual holding time for AVS is 12 days from the Laboratory Receipt Date.

To minimize the loss of sulfide, the Laboratory shall use glass tubing in the distillation apparatus. Plastic tubing should only be used to form sleeves for glass butt joints.

The Laboratory shall describe the method used to remove oxygen from the purge gas in the SDG Narrative.

The Laboratory is not required to distill the calibration standards or QC standards.

The Laboratory shall:

- Analyze for AVS per the colorimetric procedure described in the method. The Laboratory may use a semi-automated equivalent procedure using the same reagents and wavelength.
- Calibrate the instrument with at least 4 standards, one of which will be at or below the CRQL.
- Analyze ICV/CCV at appropriate mid-point concentrations.
- Prepare a Preparation Blank with each preparation batch.
- Prepare a Matrix Spike at the midpoint of the calibrated range for each SDG. No Post-distillation spike is required. Report the results on Form 5A with recovery windows of 50 – 150%.
- Prepare a Duplicate sample analysis for each SDG. Report the results on Form 6 using a control limit of 35% RPD.
- Prepare a Laboratory Fortified Blank (LFB) by spiking 10 g of clean sand with sulfide for each preparation batch. Spike the LFB at the midpoint of the calibrated range prior to distillation. The recovery limits for the LFB are 60 – 130%. Report the results of the LFB analysis on Form 7. This analysis serves as the LCS for the preparation batch.
- Add AVS to Forms 1, 2A, 3, 5A, 6, 7, 9, and 13.

Simultaneously Extractable Metals

The Laboratory shall directly analyze the SEM extract for metals by ICP-AES without additional digestion. The Laboratory shall matrix match the calibration and QC standards to the sample

matrix (0.5M HCl). The Laboratory shall report the SEM metals results in mg/kg based on the mass of sediment extracted and the final volume of the SEM extract based on the method.

The Contract Required Quantitation Limits (CRQLs) for the following analytes have been modified.

Analyte	CRQL (mg/kg)
Cd	0.2
Cu	0.4
Pb	3.0
Ni	0.5
Ag	1.0
Zn	0.4

A Method Detection Limit (MDL) study by the preparation and analysis procedure used is required. The MDL for each analyte shall be less than one half of the CRQL for each analyte listed above. The raw data must be kept on file at the Laboratory and submitted upon EPA request.

The Laboratory shall:

- Analyze a CRQL Check Standard (CRI) at the modified CRQL converted to µg/L.
- Prepare a Preparation Blank with each preparation batch.
- Prepare a Matrix Spike sample for each SDG at the levels specified in the SOW. Post-digestion Spikes are not required.
- Prepare a Duplicate sample analysis for each SDG.
- Prepare a Laboratory Fortified Blank (LFB) by spiking 10 g of clean sand with the target metals for each preparation batch. Spike the LFB at the Matrix Spike level prior to distillation. The recovery limits for the LFB are 80 – 120%. Report the results of the LFB analysis on Form 7. This analysis serves as the LCS for the preparation batch.

Reporting Requirements:

Hardcopy and electronic data reporting are required as specified per SOW ILM05.4. All hardcopy and electronic data shall be adjusted to incorporate modified specifications. This includes attaching a copy of the requirements for modified analysis to the SDG Narrative. If specific problems occur with incorporation of the modified analysis into the hardcopy and/or electronic deliverable, the Laboratory shall contact the DASS Manager within the Sample Management Office (SMO) at (703) 818-4233 or via email at CCSSUPPORT@fedcsc.com for resolution.

All samples analyzed for the same fraction within an SDG must be analyzed under the same fractional requirements. The Laboratory shall not include data for the same fraction with different requirements in the same SDG.

The Laboratory shall include the Modification Reference Number xxxx.0 on each hardcopy data form under the "NRAS No:" header appearing on each form as well as the "NRAS No." field on the Record type 21 of the electronic deliverable (if diskette deliverable is required). The Laboratory shall also document the Modification Reference Number and Solicitation Number on the SDG Coversheet.

Clarifications/Revisions to the RFQ for Modified Analysis:

Laboratory Name:
Laboratory Comments:

USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

ORGANICS ANALYSIS

Multi-Media, Multi-Concentration

SOM01.1
May 2005

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STATEMENT OF WORK

TABLE OF CONTENTS

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EXHIBIT A

SUMMARY OF REQUIREMENTS

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Exhibit A - Summary of Requirements

Table of Contents

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1.0 PURPOSE

The purpose of the multi-media, multi-concentration organic analytical service is to provide analytical data for use by the U.S. Environmental Protection Agency (USEPA) in support of its investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). Other USEPA Program Offices that have similar analytical data needs also use this service.

2.0 DESCRIPTION OF SERVICE

The organic analytical service provides a contractual framework for laboratories to apply USEPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 52 volatile, 67 semivolatile, 21 pesticide, and 9 Aroclor target compounds in water and soil/sediment samples. The analytical service provides the methods to be used, and the specific contractual requirements by which USEPA will evaluate the data. This service uses Gas Chromatograph/Mass Spectrometer (GC/MS) and Gas Chromatograph/Electron Capture Detector (GC/ECD) methods to analyze the target compounds.

3.0 DATA USES

This analytical service provides data that USEPA uses for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of a hazardous waste site, including, but not limited to, site inspections; Hazard Ranking System (HRS) scoring; remedial investigation/feasibility studies; remedial design; treatability studies; and removal actions.

The data may also be used in litigation against Potentially Responsible Parties (PRPs) in the enforcement of Superfund legislation. As a result, the Contractor must be aware of the importance of maintaining the integrity of the data generated under the contract, since it is used to make major decisions regarding public health and environmental welfare. The Contractor may be required to appear and testify to the accuracy and/or validity of the data generated.

4.0 SUMMARY OF REQUIREMENTS

4.1 Introduction to the Statement of Work

This Statement of Work (SOW) is designed as part of the documentation for a contract between USEPA and a commercial laboratory performing analyses in support of USEPA Superfund programs. The SOW is comprised of eight exhibits and one appendix. Exhibit A provides an overview of the SOW and its general requirements. Exhibit B contains a description of the reporting and deliverables requirements, in addition to the data reporting forms and the form instructions. Exhibit C specifies the Target Compound List (TCL) for this SOW with the Contract Required Quantitation Limits (CRQLs) for the sample matrices. Exhibit D details the specific analytical procedures to be used with this SOW and resulting contracts. Exhibit E provides descriptions of required Quality Assurance/Quality Control (QA/QC), Standard Operating Procedures (SOPs), and procedures used for evaluating analytical methodologies, QA/QC performance, and the reporting of data. Exhibit F contains chain-of-custody and sample documentation requirements which the Contractor

shall follow. To ensure proper understanding of the terms utilized in this SOW, a glossary can be found in Exhibit G (when a term is used in the text without explanation, the glossary meaning shall be applicable). Specifications for reporting electronic data appear in Exhibit H. Appendix A contains a listing of USEPA Registry Names, Synonyms, and Chemical Abstracts Service (CAS) Registry Numbers.

4.2 Overview of Major Task Areas

For each sample, the Contractor shall perform the tasks described in this section. Specific requirements for each task are detailed in the exhibits as referenced.

4.2.1 Task I: Sample Receiving, Storage, and Disposal

4.2.1.1 Chain-of-Custody

The Contractor shall receive and maintain samples under proper chain-of-custody procedures. All associated document control and inventory procedures shall be developed and followed. Documentation, as described herein, shall be required to show that all procedures are being strictly followed. This documentation shall be reported as the Complete Sample Delivery Group (SDG) File (CSF) (Exhibit B). The Contractor shall establish and use appropriate procedures to safeguard confidential information received from USEPA. See Exhibit F for specific requirements.

4.2.1.2 Sample Scheduling/Shipments

Sample shipments to the Contractor's facility will be scheduled and coordinated by the Contract Laboratory Program (CLP) Sample Management Office (SMO). The Contractor shall communicate with SMO personnel by telephone, fax, and/or email, as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

4.2.1.2.1 Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing required for the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier service within the Contractor's geographical area. The Contractor shall be available to receive and process sample shipments at any time the delivery service is operating, including Saturdays.

4.2.1.2.2 If there are problems with the samples (e.g., mixed media, containers broken or leaking) or sample documentation/paperwork (e.g., Traffic Report/Chain of Custody Records (TR/COCs) not with shipment, sample and TR/COC numbers do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall notify SMO personnel and the USEPA Regional CLP Project Officer (CLP PO) in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

4.2.1.2.3 To monitor the temperature of the sample shipping cooler more effectively, each USEPA Regional office may include a sample shipping cooler temperature blank with each cooler shipped.

The temperature blank will be clearly labeled: EPA COOLER TEMPERATURE INDICATOR. The Contractor shall record the presence or absence of the cooler temperature indicator bottle on Form DC-1, Item 8 - Cooler Temperature Indicator Bottle (Exhibit B).

4.2.1.2.3.1 When the USEPA Regional office supplies a cooler temperature indicator bottle in the sample shipping cooler, the Contractor shall use the USEPA-supplied cooler temperature indicator bottle to determine the cooler temperature. The temperature of the cooler shall be measured at the time of sample receipt by the Contractor.

4.2.1.2.3.2 The temperature of the sample shipping cooler shall be measured and recorded immediately upon opening the cooler, and prior to unpacking the samples or removing the packing material.

4.2.1.2.3.3 To determine the temperature of the cooler, the Contractor shall locate the cooler temperature indicator bottle in the sample shipping cooler, remove the cap, and insert a calibrated thermometer into the cooler temperature indicator bottle. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the calibrated thermometer (±1°C) shall have a measurable range of 0-50°C. Other devices that can measure temperature may be used if they can be calibrated to ±1°C and have a range of 0-50°C. If a temperature indicator bottle is not present in the cooler, an alternative means of determining cooler temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining cooler temperature. The Contractor shall contact SMO and inform them that a temperature indicator bottle was not present in the cooler. The Contractor shall document the alternative technique used to determine cooler temperature in the SDG Narrative.

4.2.1.2.3.4 If the temperature of the sample shipping cooler's temperature indicator exceeds 10°C, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the Region from which the samples were shipped for instructions on how to proceed. The Region will either require that no sample analysis(es) be performed or that the Contractor proceed with the analysis(es). SMO will in turn notify the Contractor of the Region's decision. The Contractor shall document the Region's decision and the EPA Sample Numbers of all samples for which temperatures exceed 10°C in the SDG Narrative.

4.2.1.2.3.5 The Contractor shall record the temperature of the cooler on the Form DC-1, Item 9 - Cooler Temperature, and in the SDG Narrative (Exhibit B).

4.2.1.2.4 The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation expressed in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

4.2.1.2.5 The Contractor is required to retain unused sample volume, partially used sample volume in original sample container, used sample containers, and empty sample bottle containers for a period of 60 days after data submission. From time of receipt until analysis, the Contractor shall maintain all water (preserved and unpreserved) and/or preserved soil/sediment samples at 4°C (±2°C). The Contractor shall maintain all unpreserved soil/sediment samples at -7°C (±2°C).

4.2.1.2.6 The Contractor shall be required to routinely return sample shipping containers (e.g., coolers) to the appropriate sampling office within 14 calendar days following shipment receipt (Contract Clause entitled "Government Furnished Supplies and Materials").

4.2.2 Task II: Sample Preparation and Analysis

4.2.2.1 Overview

The Contractor is advised that the samples received under the contract are usually from known or suspected hazardous waste sites and may contain high levels of organic and inorganic materials of a potentially hazardous nature. For example, the Contractor should not assume that samples that are scheduled for trace volatiles analysis do not contain analytes at concentrations appropriate for other methods. If there is any doubt about the appropriateness of a selected method, the Contractor should contact SMO for further guidance. It is the Contractor's responsibility to take all necessary measures to ensure laboratory safety.

4.2.2.2 If analysis by the Selected Ion Monitoring (SIM) technique is requested, analysis by the appropriate full scan method must be performed prior to the SIM analysis. If the full scan analysis detects all the SIM target compounds at or above the CRQLs, then the SIM analysis is not to be performed.

4.2.2.3 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique USEPA Case Number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case.

4.2.2.3.1 A Case consists of one or more SDG(s). An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received; or
- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
- Each 7 calendar day period (3 calendar day period for 7 day turnaround) during which field samples in a Case are received (said period beginning with receipt of the first sample in the SDG).

In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual

turnaround time. Preliminary Results have **no impact** on defining the SDG.

4.2.2.3.2 Samples may be assigned to SDGs by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory. However, PE samples received within a Case shall be assigned to an SDG containing field samples for that Case. Such assignment shall be made at the time the samples are received, and shall not be made retroactively.

4.2.2.3.3 Each sample received by the Contractor will be labeled with an EPA Sample Number, and accompanied by a TR/COC bearing the Sample Number and descriptive information regarding the sample.

4.2.2.3.4 The Contractor shall submit signed copies of TR/COCs for all samples in an SDG to SMO within **three working days** following receipt of the last sample in the SDG. Faxed copies of TR/COCs do not meet this requirement. TR/COCs shall be submitted in SDG sets (i.e., all TR/COCs for an SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

4.2.2.3.5 USEPA Case Numbers, SDG Numbers, and EPA Sample Numbers shall be used by the Contractor in identifying samples received under the contract, both verbally and in reports/correspondence.

4.2.2.4 If insufficient sample volume (less than the required amount) is received to perform the analysis, the Contractor shall contact SMO to inform them of the problem. SMO will contact the Region for instructions. The Region will either approve that no sample analysis be performed, or require that a reduced volume be used for the sample analysis. No other changes in the analysis will be permitted. SMO will notify the Contractor of the Region's decision. The Contractor shall document the Region's decision in the SDG Narrative.

4.2.2.5 Preparation Techniques

The Contractor will prepare samples as described in Exhibit D. For semivolatile, pesticide, and Aroclor samples, an aliquot is extracted with a solvent and concentrated. The concentrated extract is subjected to fraction-specific cleanup procedures and then analyzed by Gas Chromatograph/Mass Spectrometer (GC/MS) for semivolatiles, and Gas Chromatograph/Electron Capture Detector (GC/ECD) for the pesticides and Aroclors target compounds listed in Exhibit C. For volatile samples, an aliquot is purged with an inert gas, trapped on a solid sorbent, and then desorbed onto the GC/MS for analysis of the target compounds listed in Exhibit C.

4.2.2.6 Analytical Techniques

The target compounds listed in Exhibit C shall be identified as described in the methodologies given in Exhibit D. Automated computer programs may be used to facilitate the identification of compounds.

4.2.2.7 Qualitative Verification of Compounds

The volatile and semivolatile compounds identified by GC/MS techniques shall be verified by an analyst competent in the interpretation of mass spectra by comparison of the suspect mass spectrum to the mass spectrum of a standard of the suspected

compound. This procedure requires the use of multiple internal standards.

4.2.2.7.1 If a compound initially identified by GC/MS techniques cannot be verified, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the Contractor shall report that identification and proceed with quantitation.

4.2.2.7.2 The pesticide and Aroclor compounds identified by GC/ECD techniques shall be verified by an analyst competent in the interpretation of gas chromatograms and by comparison of the Retention Times (RTs) of the suspected unknowns with the RTs of respective standards of the suspected compounds. Pesticide compounds shall also be confirmed by GC/MS techniques if the compounds are of sufficient concentration to be detected by the GC/MS. Aroclor compounds of sufficient concentration need to be confirmed by GC/MS techniques only if requested by the Region.

4.2.2.8 Quantitation of Verified Compounds

The Contractor shall quantitate components identified by GC/MS techniques by the internal standard method stipulated in Exhibit D. Where multiple internal standards are required by USEPA, the Contractor shall perform quantitation utilizing the internal standards specified in Exhibit D. The Contractor shall quantitate components analyzed by GC/ECD techniques by the external standard method stipulated in Exhibit D. The Contractor shall also perform an initial 5 point calibration, verify its linearity, determine the breakdown of labile components, and determine calibration factors for all standards analyzed by GC/ECD techniques, as described in Exhibit D.

4.2.2.9 Tentative Identification of Non-Target Sample Components

For each analysis of a sample, the Contractor shall conduct mass spectral library searches to determine tentative compound identifications as follows: for each volatile sample, the Contractor shall conduct a search to determine the possible identity of up to 30 organic compounds of greatest concentration which are not Deuterated Monitoring Compounds (DMCs), internal standard compounds, or alkanes, and are not target compounds listed in Exhibit C under volatiles or semivolatiles. For each semivolatile sample, the Contractor shall conduct a search to determine the possible identification of up to 30 organic compounds of greatest concentration which are not DMCs, internal standard compounds, or alkanes, and are not target compounds listed in Exhibit C under volatiles or semivolatiles. In performing searches, the NIST/EPA/NIH (2002 release or later) and/or Wiley (1991 release or later), or equivalent, mass spectral library shall be used.

NOTE: Substances with responses less than 10% of the nearest internal standard are not required to be searched in this fashion.

4.2.2.10 Quality Assurance/Quality Control (QA/QC) Procedures

The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D and E. Records documenting the use of the protocol shall be maintained in accordance with the

document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibit B and Exhibit H.

4.2.2.10.1 The Contractor shall maintain a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection, as well as the quality assessment measures performed by management to ensure acceptable data production.

4.2.2.10.2 Additional QC shall be conducted in the form of the analysis of PE samples submitted to the laboratory by USEPA. Unacceptable results of all such QC or PE samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to USEPA or rejection of data for specific compound(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values, as determined by USEPA, as well as meeting the contract requirements for analysis (Exhibit D), QA/QC (Exhibit E), data reporting and other deliverables (Exhibits B and H), and sample custody, sample documentation, and SOP documentation (Exhibit F). As an alternative to data rejection, USEPA may require reanalysis of non-compliant samples. Reanalysis will be performed by the Contractor at no additional cost to USEPA, unless it is determined that the PE sample(s) was defective.

4.2.2.11 Modified Analysis

The Contractor may be requested by USEPA to perform modified analyses. These modifications may include, but are not limited to: additional compounds, sample matrices other than soil/sediment or water, and lower quantitation limits. These requests will be made by the USEPA Regional CLP PO, USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Organic Program Manager (PM), and USEPA Contracting Officer (CO) in writing, prior to sample scheduling. All contract requirements specified in the SOW/specifications will remain in effect unless the USEPA CO provides written approval for the modification(s) and a waiver for associated defects. The USEPA CO approval must be obtained prior to sample scheduling.

4.2.3 Task III: Sample Reporting Requirements and Resubmission of Data

4.2.3.1 USEPA has provided the Contractor with formats for the reporting of data (Exhibits B and H). The Contractor shall be responsible for completing and submitting analysis data sheets and electronic data in the format specified in this SOW and within the time specified in Exhibit B, Section 1.1.

4.2.3.2 Use of formats other than those designated by USEPA will be deemed as non-compliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to USEPA shall be required.

4.2.3.3 Computer-generated forms may be submitted in the hardcopy Sample Data Package(s) provided that the forms are in **exact USEPA format**. This means that the order of data elements is the same as on each USEPA-required form, including form numbers and titles, page numbers, and header information.

4.2.3.4 If the submitted data package does not conform to the specified contractual or technical criteria, the Contractor will be required to resubmit the data package and electronic data deliverable with all deficiencies corrected at its own expense. The Contractor will respond within 7 days to requests for additional information or explanations that result from the Government's inspection activities. If the Contractor is required to submit or resubmit data as a result of a Regional request, the data shall be clearly marked as ADDITIONAL DATA. The Contractor shall include a cover letter that describes which data are being delivered, to which EPA Case Number the data pertain, and who requested the data. Any and all resubmissions must be in accordance with the documentation requirements of this SOW.

4.2.3.5 The data reported by the Contractor on the hardcopy data forms and the associated electronic data submitted by the Contractor shall contain identical information. If discrepancies are found during Government inspection, the Contractor shall be required to resubmit either the corrected hardcopy forms or the corrected electronic data, or both sets of corrected data, at no additional cost to USEPA.

4.2.3.6 In addition, the Contractor must be aware of the importance of maintaining the integrity of the data generated under the contract, since it is used to make major decisions regarding public health and environmental welfare. The data may also be used in litigation against Potentially Responsible Parties (PRPs) in the enforcement of Superfund legislation.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 Report Deliverable Schedule

The following table reiterates the contract reporting and deliverables requirements specified in the Contract Schedule (Performance/Delivery Schedule) and specifies the distribution that is required for each deliverable. The turnaround times for Items B through D listed below are 7, 14, and 21 days.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The US Environmental Protection Agency (USEPA) Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Organic Program Manager (PM) will notify the Contractor, in writing, of such changes when they occur.

TABLE 1

Report Deliverable Schedule

Item		No. of Copies[A]	Delivery Schedule	Distribution	
				SMO	Region
A.[2]	Sample Traffic Reports/ Chain of Custody Records	1	3 working days after receipt of last sample in an Sample Delivery Group (SDG).[1]	X	
B.[2]	Sample Data Package[B]	1	XX[C] days after receipt of last sample in an SDG.	X	
C.[2]	Electronic Data Deliverable	1	XX[C] days after receipt of last sample in an SDG.	X	
D.[2, 3]	Complete SDG File	1	XX[C] days after receipt of last sample in an SDG.		X
E.[2]	Hardcopy Data in PDF Format	1	XX[C] days after receipt of last sample in an SDG		X

TABLE 1

Report Deliverable Schedule (Con't)

Item		No. of Copies[A]	Delivery Schedule	Distribution	
				SMO	Region
F.[4]	Preliminary Results (VOA Analyses)	1	Within 48 hours after receipt of each sample in an SDG at laboratory, if requested.	X	X
	Preliminary Results (SV, PEST, and ARO Analyses)	1	Within 72 hours after receipt of each sample in an SDG at laboratory, if requested.	X	X
G.[5]	Standard Operating Procedures-- Technical and Evidentiary	1	Revise within 60 days after contract award. Submit within 7 days of receipt of written request to recipients as directed.	As directed	
H.[5]	Quality Assurance Plan	1	Revise within 60 days after contract award. Submit within 7 days of receipt of written request to recipients as directed.	As directed	
I.	GC/MS GC/ECD Electronic Data	Lot	Retain for 3 years after data submission. Submit within 7 days after receipt of written request by CLP PO.	As directed	

TABLE 1 (Con't)

Report Deliverable Schedule

Item		No. of Copies[A]	Delivery Schedule	Distribution	
				SMO	Region
J.[6]	Extracts	Lot	Retain for 365 days after data submission. Submit within 7 days after receipt of written request by CLP PO or SMO, at USEPA's direction.	As directed	
K.	Method Detection Limit Study		Submit to USEPA within 7 days after receipt of written request by CLP PO or SMO, at USEPA's direction.	As directed	

Laboratories:

[A]The number of copies specified are the number of copies required to be delivered to each recipient.

[B]Contractor-concurrent delivery to USEPA-designated recipient [e.g., Quality Assurance Technical Support(QATS)] may be required upon request by the USEPA Regional Contract Laboratory Program Project Officer (CLP PO). Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the CLP PO. Supplemental data (i.e., logbooks) may be requested in writing from the Regional staff or QATS. All written communication sent by USEPA must include the laboratory's CLP PO in the distribution list. If the CLP PO has not been included in the distribution list, contact the OSRTI ASB Organic Program Manager.

[C]The number of days associated with these elements will be provided in the associated laboratory contract document, and will also be provided at the time of the sample scheduling by the Sample Management Office (SMO) Contractor.

[1]A Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less (3 calendar day period for 7-day turnaround) and not exceeding 20 samples [excluding Performance Evaluation (PE) samples] and scheduled under the same contractual turnaround time. Note that Preliminary Results have no impact on defining the SDG. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. See Exhibit A for further description.

[2]**DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Delivery shall be made such that all designated recipients receive the item on the same calendar day. The Data Receipt Data (DRD) of the SDG and any samples within the SDG is the date that the Electronic Data Deliverable (EDD) and the Hardcopy of the Deliverable have both been received. If one of these items is delivered at a later date, the date that the last item is delivered is the SDG DRD. If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables delivered after this time will be considered late.**

[3]Complete Sample Delivery Group File (CSF) will contain the original Sample Data Package plus all of the original documents described under Section 2.6.

[4]If requested at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of Form I and Form I TIC analytical results, by fraction, for field and Quality Control (QC) sample analyses via facsimile or email, Form X for Pesticides, and Form X for Aroclors. The Contractor may submit Preliminary Results in electronic format after obtaining permission from USEPA. The Contractor will be notified of the fax number or email address at the time of sample scheduling. Sample Traffic Report/Chain of Custody Records (TR/COCs) and SDG Cover Sheets shall be submitted with the Preliminary Results. The Contractor shall contact SMO after confirming transmission. The Contractor shall document all communication in a telephone contact log.

[5] See Exhibit E and Exhibit F for a more detailed description.

[6]Method Detection Limit (MDL) Study is to be performed annually, or for each new instrument, whichever is more frequent. The information should be available on file and provided to USEPA within 7 days after the receipt of a written request.

Preliminary Results Delivery Schedule:

If the sample arrives before 5 p.m., the Preliminary Results for that sample are due within the required turnaround time. If the sample is received after 5 p.m., the Preliminary Results for that sample are due within the required turnaround time beginning at 8 a.m. the following day. **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables delivered after this time will be considered late.**

NOTE: As specified in the Contract Schedule (Government Furnished Supplies and Materials), unless otherwise instructed by the CLP SMO based on a Regional decision, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than 60 days following submission of the reconciled CSF. Sample disposal and disposal of unused sample bottles/containers are the responsibility of the Contractor, and should be done in accordance with all applicable laws and regulations governing disposal of such materials.

1.2 Distribution

The following addresses correspond to the "Distribution" column in Table 1 of Section 1.1:

SMO: USEPA Contract Laboratory Program
Sample Management Office (SMO)[1]
15000 Conference Center Drive
Chantilly, VA 20151-3808

USEPA REGIONS:

SMO will provide the Contractor with the list of addresses for the 10 USEPA Regions. SMO will provide the Contractor with updated Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

USEPA ASB Organic Program Manager (PM):
Mailing Address:

USEPA OSRTI Analytical Services Branch
Ariel Rios Building (5204G)
1200 Pennsylvania Avenue, N.W.
Washington, D.C. 20460
Attn: CLP Organic Program Manager

Fed-Ex/Overnight Delivery:

USEPA OSRTI Analytical Services Branch
1235 Jefferson Davis Highway
Crystal Gateway I, 12th Floor
Arlington, VA 22202
Attn: CLP Organic Program Manager

USEPA Regional CLP Project Officer (CLP PO):

SMO will provide the Contractor with the list of addresses for the USEPA Regional CLP POs. SMO will provide the Contractor with updated address/name lists as necessary throughout the period of the contract.

QATS: USEPA Contract Laboratory Program
Quality Assurance Technical Support Laboratory[2]
2700 Chandler Avenue, Building C
Las Vegas, NV 89120
Attn: Data Audit Staff

[1]SMO is a Contractor-operated facility operating under the SMO contract, awarded and administered by USEPA.

[2]The QATS Laboratory is a Contractor-operated facility operating under the QATS contract, awarded and administered by USEPA.

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1 Introduction

The Contractor shall provide reports and other deliverables as specified in the Contract Schedule (Performance/Delivery Schedule). The required content and form of each deliverable is described in this Exhibit. All reports and documentation **must be**:

- Legible;
- Clearly labeled and completed in accordance with instructions in this exhibit;
- Arranged in the order specified in this section;
- Paginated consecutively in ascending order starting from the Sample Delivery Group (SDG) Narrative;
- Copies must be legible and double-sided; and
- Information reported on the forms listed in this Exhibit [excluding the Sample Log-In Sheet (DC-1) and the Complete SDG File (CSF) Inventory Sheet (DC-2)] must be either typewritten or computer-generated. Handwritten corrections of the information must be legible, signed, and dated.

NOTE: CSFs need not be double-sided. (The CSF is composed of original documents.) However, Sample Data Packages delivered to the Sample Management Office (SMO), and USEPA-designated recipients [e.g., Quality Assurance Technical Support (QATS)] upon written request, must be double-sided.

2.1.1 Requirements for each deliverable item cited in the Contract Schedule (Performance/Delivery Schedule) are specified in Sections 2.3 through 2.11. Prior to submission, the Contractor shall arrange items and the components of each item in the order listed in these sections.

2.1.2 The Contractor shall use EPA Case Numbers (including SDG numbers) and EPA Sample Numbers to identify samples received under the contract, both verbally and in reports/correspondence. The Contract Number shall be specified in all correspondence.

2.1.3 If Selected Ion Monitoring (SIM) analysis is performed, then all SIM data (Forms and raw data) must be arranged at the end of the subsection (i.e., Trace VOA-SIM must be at the end of the Trace-VOA section and SV-SIM must be at the end of the Semivolatiles section).

2.2 Resubmission of Data

If submitted documentation does not conform to the above criteria, the Contractor is required to resubmit such documentation with deficiency(ies) corrected within 6 business days, at no additional cost to USEPA. Only the nonconforming documentation is required to be resubmitted (i.e., if only the hardcopy in Portable Document Format (PDF) is nonconforming, then a resubmittal of only the corrected hardcopy is required).

2.2.1 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, or through a USEPA Regional Contract Laboratory Program Project Officer (CLP PO) action, or through a Regional data reviewer's request, the data shall be

clearly marked as ADDITIONAL DATA and shall be sent to both contractual data recipients (SMO and the Region), and to the USEPA-designated recipient (e.g., QATS) within 7 days of a written request for the Sample Data Package. The Contractor shall include a cover letter that describes which data are being delivered, to which USEPA Case(s) the data pertain, and **who requested the data**.

2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data shall be sent to both contractual data recipients (SMO and the Region), and to the USEPA-designated recipient (e.g., QATS when a written request for the Sample Data Package has been made within 6 business days of receipt of CCS results of first submission data). In all instances, the Contractor shall include a color-coded COVER SHEET (Laboratory Response To Results of Contract Compliance Screening) provided by SMO.

2.3 Quality Assurance Plan (QAP) and Standard Operating Procedures (SOPs)

The Contractor shall adhere to the requirements in Exhibits E and F.

2.4 Traffic Report/Chain of Custody Records (TR/COCs)

Each sample received by the Contractor will be labeled with an EPA Sample Number. EPA Sample Numbers are five digits in length and continuous (without spaces or hyphens). Each sample will be accompanied by a Sample TR/COC bearing the Sample Number and descriptive information regarding the sample. The Contractor shall complete the TR/COC (marked "Lab Copy for Return to SMO"), recording the date of sample receipt and shall sign the TR/COC. Information shall be recorded for each sample in the SDG.

2.4.1 The Contractor shall submit TR/COCs in SDG sets (i.e., TR/COCs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached. The SDG Cover Sheet shall contain the following items:

- Laboratory name;
- Contract number;
- Modification number;
- Sample analysis price (full sample price from the contract);
- Case Number;
- List of fractions analyzed; and
- List of EPA Sample Numbers of all samples in the SDG, identifying the **first** and **last** samples received, and the Laboratory Receipt Dates (LRDs).

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest Sample Number (considering both alpha and numeric designations); the "last" sample received would be the highest Sample Number (considering both alpha and numeric designations).

2.4.2 EPA Sample Numbers are five digits in length and continuous (without spaces or hyphens). If the Contractor receives Sample Numbers of any other length, the Contractor shall contact SMO immediately.

2.4.3 Each TR/COC shall be clearly marked with the SDG Number, entered below the LRD on the TR/COC. The TR/COC for the **last** sample received in the SDG shall be clearly marked "SDG–FINAL SAMPLE". The SDG Number is the EPA Sample Number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG Number shall be the lowest Sample Number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

2.4.4 If samples are received at the laboratory with multi-sample TR/COCs, all the samples on one multi-sample TR/COC may not necessarily be in the same SDG. In this instance, the Contractor shall make the appropriate number of photocopies of the TR/COC, and submit one copy with each SDG Cover Sheet.

2.5 Sample Data Package

The Sample Data Package is divided into the six major units described in this section. The last four units are each specific to an analytical fraction (Trace Volatiles/SIM, Low/Medium Volatiles, Semivolatiles/SIM, Pesticides, and Aroclors). If analysis by SIM is required, report all data for SIM analysis as a subsection at the end of the applicable fraction. If the analysis of a fraction is not required, then that fraction-specific unit is not required as a deliverable. The Sample Data Package shall include data for the analyses of all samples in one SDG, including: field samples; dilutions; reanalyses; blanks; Laboratory Control Samples (LCSs); and any requested Matrix Spikes and Matrix Spike Duplicates (MS/MSDs). The Contractor shall retain a copy of the Sample Data Package for 365 days after final acceptance of data. After this time, the Contractor may dispose of the package.

2.5.1 SDG Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: Laboratory Name; Case Number; EPA Sample Numbers in the SDG, differentiating between initial analyses and reanalyses; SDG Number; Contract Number; and detailed documentation of any Quality Control (QC), sample, shipment, and/or analytical problems encountered in processing the samples reported in the data package. For soil samples collected and pre-weighed in the field for volatiles analysis, the laboratory shall document all discrepancies between sample weights determined in the field and in the laboratory in the SDG Narrative. For aqueous samples, the laboratory shall report all samples where headspace or air bubbles are present. The laboratory shall also document how soil samples for volatiles analysis were handled upon receipt (e.g., storage in refrigerator, transferred to closed-system vials and frozen, etc.).

The Contractor shall document, in the SDG Narrative, the alternative technique used to determine cooler temperature if a temperature indicator bottle is not present in the cooler. Any temperature deviations (>10°C) should be noted for the affected EPA samples. The Contractor shall also provide, in the SDG Narrative, sufficient information, including equations or curves (at least one equation or curve per method), to allow the recalculation of sample results from raw instrument output. The Contractor shall also include a discussion of any flexibility Statement of Work (SOW) modifications. This includes attaching a copy of the USEPA-approved modification

form to the SDG Narrative. Additionally, the Contractor shall also identify and explain any differences that exist between the Form Is and supporting documentation provided in the data package and those previously provided as Preliminary Results.

All Gas Chromatography (GC) columns used for analysis shall be documented here, by fraction. List the GC column identification--brand name, the internal diameter, in millimeters (mm), and the length, in meters (m), packing/coating material, and film thickness. The trap used for volatile analysis shall be described here. List trap name, when denoted by the manufacturer, its composition (packing material/brand name, amount of packing material, in length). The Contractor shall include any technical and administrative problems encountered, the corrective actions taken, the resolution, and an explanation for all flagged edits (e.g., manual edits) on quantitation lists. The Contractor shall document in the SDG Narrative all instances of manual integration.

The SDG Narrative shall contain the following statement, verbatim: "**I certify that this Sample Data Package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy Sample Data Package and in the electronic data deliverable has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.**" This statement shall be directly followed by an original signature of the Laboratory Manager or designee with typed lines below it containing the signer's name and title, and the date of signature.

2.5.1.1 Whenever data from sample reanalyses are submitted, the Contractor shall state in the SDG Narrative for **each** reanalysis whether the reanalysis is billable, and if so, why. This includes required billable reanalysis for Aroclor samples meeting the criteria in Exhibit D Aroclors, Section 11.3.8.

2.5.1.2 The Contractor shall list the pH determined for each water sample submitted for volatiles analysis. This information may appear as a simple list or table in the SDG Narrative. The purpose of this pH determination is to ensure that all water volatiles samples were acidified in the field. No pH adjustment is to be performed by the Contractor on water samples for volatiles analysis.

2.5.1.3 The Contractor shall submit in writing all email correspondences or telephone conversations with SMO or the Region.

2.5.2 Traffic Report/Chain Of Custody Records (TR/COC)

The Contractor shall include a copy of the TR/COCs submitted in Section 2.4 for all of the samples in the SDG. The TR/COCs shall be arranged in increasing EPA Sample Number order, considering both letters and numbers. Copies of the SDG Cover Sheet are to be included with the copies of the TR/COCs. (See Section 2.4 for more detail on reporting requirements for TR/COCs.) In the case of multi-sample TR/COCs, the Contractor shall make the appropriate number of photocopies of the TR/COC so that a copy is submitted with each applicable data package. In addition, in any instance where samples from more than one multi-sample TR/COC are in the same data package, the Contractor shall submit a copy of the SDG Cover Sheet with copies of the TR/COCs.

2.5.3 Volatiles Data

2.5.3.1 Volatiles Quality Control (QC) Summary

2.5.3.1.1 Deuterated Monitoring Compound (DMC) Recovery (Form II VOA-1, VOA-2, VOA-3, VOA-4, VOA-SIM1, VOA-SIM2)

2.5.3.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (Form III VOA-1, VOA-2): This data shall be provided upon USEPA Region's request for analysis of MS/MSDs.

2.5.3.1.3 Method Blank Summary (Form IV VOA, VOA-SIM): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

2.5.3.1.4 Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance Check (Form V VOA): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

NOTE: This form is not required for the optional analysis when submitting data using the SIM technique.

2.5.3.1.5 Internal Standard Area and RT Summary (Form VIII VOA, VOA-SIM): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

2.5.3.2 Volatiles Sample Data

Sample data shall be arranged with the Volatile Organics Analysis Data Sheet (Form I VOA-1, VOA-2, including Form I VOA-TIC), followed by the raw data for volatile samples. The sample data shall be placed in order of increasing EPA Sample Number, considering both letters and numbers. Volatile sample data for SIM analysis must be arranged together with the rest of the SIM Volatiles data at the end of the subsection.

2.5.3.2.1 Target Compound Results, Volatile Organics Analysis Data Sheet (Form I VOA-1, VOA-2). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C - Volatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 2.5.1). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.5.3.2.2 Tentatively Identified Compounds (TICs) (Form I VOA-TIC). Form I VOA-TIC is the tabulated list of the highest probable match for up to 30 organic compounds that are not target compounds, DMCs, internal standard compounds, or alkanes, and are not listed in Exhibit C - Volatiles and Semivolatiles. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. The tabulated list includes the Chemical Abstracts Service (CAS) Number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found.

NOTE: This form is not required when submitting data for the optional analysis using the SIM technique.

2.5.3.2.3 Reconstructed Total Ion Chromatograms (for each sample including dilutions and reanalyses). Reconstructed ion chromatograms shall be normalized to the largest nonsolvent component and shall contain the following header information:

- EPA Sample Number;
- Date and time of analysis;
- GC/MS instrument identifier;
- Laboratory File Identifier; and
- Analyst ID.

NOTE: Each Selected Ion Current Profile (SICP) for samples taken through the optional analysis using the SIM technique shall be labeled as in this section.

2.5.3.2.3.1 Internal standards and DMCs shall be labeled with the names of compounds, either directly out from the peak or on a printout of Retention Times (RTs) if RTs are printed over the peak. Labeling of other compounds is not required and should not detract from the legibility of the required labels.

2.5.3.2.3.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system report shall be included in all Sample Data Packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below.

- EPA Sample Number;
- Date and time of analysis;
- RT or scan number of identified target compounds;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- On column concentration/amount, including units;
- GC/MS instrument identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.5.3.2.3.3 In all instances where the data system report has been edited, or where manual integration or manual quantitation has been performed, the GC/MS Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation

report. In addition, a hardcopy printout of the Extracted Ion Current Profile (EICP) of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C - Volatiles, internal standards, and DMCs.

2.5.3.2.4 Other Required Information. For each sample, by each compound identified, the following items shall be included in the data package:

- Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C - Volatiles that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. This includes target compounds that are identified during the optional analysis using the SIM technique. Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra; and

- Copies of mass spectra of organic compounds not listed in Exhibit C with associated best-match spectra (maximum of three best matches). Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.

2.5.3.3 Volatiles Standards Data

2.5.3.3.1 Initial Calibration Data (Form VI VOA-1, VOA-2, VOA-3, VOA-SIM) shall be included in order by instrument, if more than one instrument is used.

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, labeled as in Section 2.5.3.2.3. Spectra are not required.

- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.

- Labels for standards shall reflect the concentrations of the non-ketone analytes in µg/L. (If the non-ketone analytes have a concentration of 5.0 µg/L then the reported label shall be RRF5.0.

  NOTE: For low-level soil samples, the concentration of the low standard is 2.5 µg/L. Since 10 mL purge volumes are required for low-level soil standards, the reported label shall be RRF2.5.

- EICPs displaying each manual integration.

2.5.3.3.2 Continuing Calibration Verification Data (Form VII VOA-1, VOA-2, VOA-3, VOA-SIM) shall be included in order by instrument, if more than one instrument is used.

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour) calibration verifications, labeled as in Section 2.5.3.2.3. Spectra are not required.

- When more than one Continuing Calibration Verification (CCV) is performed, forms shall be in chronological order, by instrument.

- EICPs displaying each manual integration.

2.5.3.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C - Volatiles, internal standards, and DMCs.

2.5.3.4 Volatiles Raw QC Data

2.5.3.4.1 4-Bromofluorobenzene data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.5.3.2.3.

- Mass listing, labeled as in Section 2.5.3.2.3.

- Reconstructed total ion chromatogram, labeled as in Section 2.5.3.2.3.

2.5.3.4.2 Blank data shall be arranged by type of blank (method, storage, instrument) and shall be in chronological order, by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I VOA-1, VOA-2, VOA-SIM).

- Tentatively Identified Compounds (Form I VOA-TIC) even if none are found.

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.3.2.3.

- Target compound spectra with laboratory-generated standard, labeled as in Section 2.5.3.2.4. Data systems that are incapable of dual display shall provide spectra in the following order:

  -- Raw target compound spectra.

  -- Enhanced or background-subtracted spectra.

  -- Laboratory-generated standard spectra.

- GC/MS library search spectra for TICs, labeled as in Section 2.5.3.2.4.

- Quantitation/calculation of TIC concentrations.

2.5.3.4.3 Volatiles Matrix Spike Data

- Tabulated results (Form I VOA-1, VOA-2) of target compounds. Form I VOA-TIC is not required.

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.3.2.3. Spectra are not required.

2.5.3.4.4 Volatiles Matrix Spike Duplicate Data

- Tabulated results (Form I VOA-1, VOA-2) of target compounds. Form I VOA-TIC is not required.

- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.3.2.3. Spectra are not required.

2.5.4 Semivolatiles Data

2.5.4.1 Semivolatiles QC Summary

2.5.4.1.1 Deuterated Monitoring Compound Recovery (Form II SV-1, SV-2, SV-3, SV-4, SV-SIM)

2.5.4.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (Form III SV-1, SV-2, SV-SIM): This data shall be provided upon the USEPA Region's request for analysis of MS/MSDs.

2.5.4.1.3 Method Blank Summary (Form IV SV, SV-SIM): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

2.5.4.1.4 GC/MS Instrument Performance Check (Form V SV): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

NOTE: This form is not required when submitting data for the analysis of Polynuclear Aromatic Hydrocarbons (PAHs)/pentachlorophenol using the SIM technique.

2.5.4.1.5 Internal Standard Area and RT Summary (Form VIII SV-1, SV-2, SV-SIM1, SV-SIM2): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

2.5.4.2 Semivolatiles Sample Data

Sample data shall be arranged in packets with the Semivolatiles Organics Analysis Data Sheet (Form I SV-1, SV-2, including Form I SV-TIC), or Form I SV-SIM, if optional analysis of PAHs and pentachlorophenol is requested, followed by the raw data for semivolatiles samples. These sample packets shall be placed in order of increasing EPA Sample Number, considering both letters and numbers.

2.5.4.2.1 Target Compound Results, Semivolatiles Organics Analysis Data Sheet (Form I SV-1, SV-2). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C - Semivolatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (Section 2.5.1). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.5.4.2.2 Semivolatile Tentatively Identified Compounds (Form I SV-TIC). Form I SV-TIC is the tabulated list of the highest probable match for up to 30 organic compounds that are not DMCs, internal standard compounds, or alkanes, and are not target compounds listed in Exhibit C - Volatiles and Semivolatiles. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} that contains only C-H and C-C single bonds. The tabulated list includes the CAS Number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found.

NOTE: This form is not required when submitting data for the optional analysis of PAHs/pentachlorophenol using the SIM technique.

2.5.4.2.3 PAHs/Pentachlorophenol Analysis Data Sheet (Form I SV-SIM). This data form shall be submitted upon the USEPA Region's request for optional analysis of PAHs/pentachlorophenol using the SIM technique. The specific target PAHs/pentachlorophenol listed in Exhibit C - Semivolatiles shall be included. The validation and release of these results are authorized by a specific signed statement in the SDG Narrative (Section 2.5.1). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.5.4.2.4 Reconstructed Total Ion Chromatograms (for each sample, including dilutions and reanalyses). Reconstructed ion chromatograms shall be normalized to the largest nonsolvent component and shall contain the following header information:

- EPA Sample Number;
- Volume Injected (µL);
- Date and time of analysis;
- GC/MS instrument identifier;
- Laboratory File Identifier; and
- Analyst ID.

NOTE: Each SICP for samples taken through the optional analysis of PAHs/pentachlorophenol using the SIM technique shall be labeled as in Section 2.5.4.2.4.

2.5.4.2.4.1 Internal standard compounds and DMCs shall be labeled on reconstructed ion chromatography or SICPs with the names of

compounds, either directly out from the peak or on a printout of RTs if RTs are printed over the peak.

2.5.4.2.4.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system report shall be included in all Sample Data Packages, in addition to the reconstructed ion chromatogram or SICP for optional PAHs/pentachlorophenol analysis. The complete data system report shall include all of the information listed below. For laboratories that do not use automated data system procedures, a laboratory "raw data sheet" containing the following information shall be included in the Sample Data Package, in addition to the chromatogram:

- EPA Sample Number;
- Date and time of analysis;
- RT or scan number of identified target compounds;
- Ion used for quantitation with measured area;
- Copy of area table from data system;
- On column concentration/amount, including units;
- GC/MS instrument identifier;
- Laboratory File Identifier; and
- Analyst ID.

2.5.4.2.4.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C - Semivolatiles, internal standards, and DMCs.

2.5.4.2.5 Other Required Information. For each sample, by each compound identified, the following items shall be included in the data package.

- Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C - Semivolatiles that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. This includes PAH/pentachlorophenol target compounds that are identified during the optional analysis using the SIM technique. Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.

- Copies of mass spectra of non-DMCs/non-internal standard organic compounds not listed in Exhibit C - Semivolatiles with associated best-match spectra (maximum of three best matches). This includes the mass spectra for tentatively identified alkanes. Spectra shall be labeled with EPA Sample Number, Laboratory File Identifier, date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.

2.5.4.3 Semivolatiles Standards Data

2.5.4.3.1 Initial Calibration Data (Form VI SV-1, SV-2, SV-3) or Form VI SV-SIM (when optional analysis of PAHs/pentachlorophenol is performed) shall be included in order by instrument, if more than one instrument is used.

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, labeled as in Section 2.5.4.2.4. Spectra are not required.

- When optional analysis of PAHs/pentachlorophenol is requested, then SICPs and quantitation reports for the initial calibration standards (five-point), labeled as in Section 2.5.4.2.4, shall be submitted. Spectra are not required.

- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.

- Labels for standards shall reflect the concentrations of the majority of the analytes in ng/µL. (If the majority of the analytes have a concentration of 5.0 ng/µL then the reported label shall be RRF5.0.

- EICPs displaying each manual integration.

2.5.4.3.2 Continuing Calibration Verification Data (Form VII SV-1, SV-2, SV-3) or Form VII SV-SIM (when optional analysis of PAHs/pentachlorophenol is performed) shall be included in order by instrument, if more than one instrument is used.

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for all opening and closing CCVs, labeled as in Section 2.5.4.2.4. Spectra are not required.

- When optional analysis of PAHs/pentachlorophenol is requested, then SICPs and quantitation reports for all opening and closing CCVs, labeled as in Section 2.5.4.2.4. Spectra are not required.

- When more than one CCV is performed, forms shall be in chronological order, by instrument.

- EICPs displaying each manual integration.

2.5.4.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed,

the GC/MS Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C - Semivolatiles, internal standards, and DMCs.

2.5.4.4 Semivolatiles Raw Quality Control (QC) Data

2.5.4.4.1 Decafluorotriphenylphosphine (DFTPP) data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.5.4.2.4.
- Mass listing, labeled as in Section 2.5.4.2.4.
- Reconstructed total ion chromatogram, labeled as in Section 2.5.4.2.4.

2.5.4.4.2 Blank data shall be included in chronological order by extraction date.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I SV-1, SV-2, SV-SIM).
- Tentatively Identified Compounds (Form I SV-TIC), even if none are found.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.4.2.4.
- Target compound spectra with laboratory-generated standard, labeled as in Section 2.5.4.2.5. Data systems which are incapable of dual display shall provide spectra in the following order:
  - -- Raw target compound spectra.
  - -- Enhanced or background-subtracted spectra.
  - -- Laboratory-generated standard spectra.
- GC/MS library search spectra for TICs, labeled as in Section 2.5.4.2.4.
- Quantitation/calculation of TIC concentrations.

2.5.4.4.3 Semivolatiles Matrix Spike Data

- Tabulated results (Form I SV-1, SV-2) of target compounds. Form I SV-TIC is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.4.2.4. Spectra are not required.

2.5.4.4.4 Semivolatiles Matrix Spike Duplicate Data

- Tabulated results (Form I SV-1, SV-2) of target compounds. Form I SV-TIC is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.5.4.2.4. Spectra are not required.

2.5.4.4.5 Semivolatile Gel Permeation Chromatograph (GPC) Data

The two most recent Ultra Violet (UV) traces of the GPC calibration solution, and the reconstructed ion chromatogram and data system reports for the GPC blank shall be arranged in chronological order by GPC for the GPC calibration.

- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of RTs when the RTs are printed directly over the peak.
- Reconstructed ion chromatogram and data system report(s) labeled as specified in Section 2.5.4.2.4 for GPC blank analysis.
- Reconstructed ion chromatogram and data system report(s) for the mid-point initial calibration standard associated with the GPC blank labeled, as specified in Section 2.5.4.2.4.

2.5.5 Pesticides Data

2.5.5.1 Pesticides QC Summary

2.5.5.1.1 Surrogate Recovery (Form II PEST-1, PEST-2)

2.5.5.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (Form III PEST-1, PEST-2): MS/MSD is required for the Pesticides fraction, unless otherwise specified by the USEPA Region. See Exhibit D - Analytical Methods for Pesticides for frequency.

2.5.5.1.3 Laboratory Control Sample Recovery (Form III PEST-3, PEST-4).

2.5.5.1.4 Method Blank Summary (Form IV PEST): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.

2.5.5.2 Pesticides Sample Data

Sample data shall be arranged in packets with the Pesticides Organics Analysis Data Sheet (Form I PEST), followed by the raw data for pesticide samples. These sample packets should then be placed in order of increasing EPA Sample Number, considering both letters and numbers.

2.5.5.2.1 Target Compound Results, Pesticides Organics Analysis Data Sheet (Form I PEST). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C - Pesticides) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (Section 2.5.1). In the event that the

Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.5.5.2.2 Copies of Pesticide Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of RTs on the data system printout if RTs are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D - Analytical Methods for Pesticides, and shall be labeled with the following information:

- EPA Sample Number;
- Volume injected (µL);
- Date and time of injection;
- On column concentration/amount including units;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

2.5.5.2.3 Copies of pesticide chromatograms from the second GC column shall be included and labeled as in Section 2.5.5.2.2.

2.5.5.2.4 Data System Printout. A printout of RT, corresponding peak height or peak area, and the on column amount shall accompany each chromatogram. The printout shall be labeled with the EPA Sample Number. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the Gas Chromatograph/Electron Capture Detector (GC/ECD) Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range. The GC/ECD Operator shall also mark each integrated area with the letter "m" on the quantitation report.

2.5.5.2.5 All manual worksheets shall be included in the Sample Data Package.

2.5.5.2.6 Other Required Information. If pesticides are confirmed by GC/MS, the Contractor shall submit copies of reconstructed ion chromatograms, raw spectra, and background-subtracted mass spectra of target compounds listed in Exhibit C - Pesticides that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. Compound names shall be clearly marked on all spectra. For Toxaphene confirmed by GC/MS, the Contractor shall submit mass spectra of three major peaks from samples and standards.

2.5.5.3 Pesticides Standards Data

2.5.5.3.1 Initial Calibration of Single Component Analytes (Form VI PEST-1, PEST-2): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.2 Toxaphene Initial Calibration (Form VI PEST-3, PEST-4): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.3 Analyte Resolution Check Summary (Form VI PEST-5): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.4 Performance Evaluation Mixture (PEM)(Form VI PEST-6): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.5 Individual Standard Mixture A (Form VI PEST-7): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.6 Individual Standard Mixture B (Form VI PEST-8): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.7 Individual Standard Mixture C (Form VI PEST-9, PEST-10): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.8 Calibration Verification Summary (Form VII PEST-1): For all performance evaluation mixtures and instrument blanks, on all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.9 Calibration Verification Summary (Form VII PEST-2, PEST-3): For all mid-point concentrations of Individual Standard Mixtures A and B or C and instrument blanks used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.10 Analytical Sequence (Form VIII PEST): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.5.3.11 Florisil Cartridge Check (Form IX PEST-1): For all lots of cartridges used to process samples in the SDG, using Individual Standard Mixture A or C.

2.5.5.3.12 GPC Calibration Verification (Form IX PEST-2): For all GPC columns, in chronological order by calibration verification date.

2.5.5.3.13 Identification Summary for Single Component Analytes (Form X PEST): For all samples with positively identified single component analytes, in order by increasing EPA Sample Number.

2.5.5.3.14 Chromatograms and data system printouts shall be included for all standards, including the following:

- Resolution check mixture.

- Performance Evaluation (PE) mixtures, all.
- Individual Standard Mixture A and B, both at five concentrations, for each initial calibration.

Or

- Individual Standard Mixture C, at five concentrations, each initial calibration
- Toxaphene, at five concentrations, each initial calibration.
- All mid-point concentrations of Individual Standard Mixtures A and B or C used for calibration verification.
- All Toxaphene standards analyzed for confirmation.

2.5.5.3.15 A printout of RT and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA Sample Number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D - Analytical Methods for Pesticides, and shall be labeled with the following:

- EPA Sample Number for the standard (e.g., INDA1OK, INDA2OK, etc.). See Section 3 for details;
- Label all standard peaks for all individual compounds either directly out from the peak on the chromatogram or on the printout of RTs on the data system printout, if RTs are printed over the peak on the chromatogram;
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram;
- Date and time of injection;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

NOTE: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range. The GC/ECD Operator shall also mark each integrated area with the letter "m" on the quantitation report.

2.5.5.4 Pesticides Raw Quality Control (QC) Data

2.5.5.4.1 Blank data shall be arranged by type of blank (method, instrument, sulfur cleanup) and shall be in chronological order by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I PEST).
- Chromatogram(s) and data system printout(s) for each GC column and instrument used for analysis, labeled as in Sections 2.5.5.2.2 and 2.5.5.2.4.

2.5.5.4.2 Pesticides LCS Data

- Tabulated results (Form I PEST) of target compounds for both GC columns.
- Chromatograms and data systems printouts for both GC columns, labeled as in Sections 2.5.5.2.2 and 2.5.5.2.4.

2.5.5.4.3 Pesticides Matrix Spike Data

- Tabulated results (Form I PEST) of target compounds for both GC columns.
- Chromatograms and data system printouts for both GC columns, labeled as in Sections 2.5.5.2.2 and 2.5.5.2.4.

2.5.5.4.4 Pesticides Matrix Spike Duplicate Data

- Tabulated results (Form I PEST) of target compounds for both GC columns.
- Chromatograms and data system printouts for both GC columns, labeled as in Sections 2.5.5.2.2 and 2.5.5.2.4.

2.5.5.5 Raw Gel Permeation Chromatograph (GPC) Data

2.5.5.5.1 GPC Calibration. The UV traces for the GPC calibration solution, chromatograms, and the data system reports for the GPC blank shall be arranged in chronological order for the GPC calibration.

- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of RTs when the RTs are printed directly over the peak.
- Chromatogram and data system report(s) labeled as specified in Sections 2.5.5.2.2 and 2.5.5.2.4 for GPC blank analyses.
- Chromatogram and data system report(s) for the mid-point initial calibration standard associated with the GPC blank labeled as specified in Section 2.5.5.3.15 (i.e., Individual Standard Mixture A, Individual Standard Mixture B, Individual Standard Mixture C, and the Toxaphene standards).

2.5.5.5.2 GPC Calibration Verification. The chromatogram and the data system report(s) shall be arranged in chronological order for the GPC calibration check.

- Chromatograms and data system printouts labeled as specified in Sections 2.5.5.2.2 and 2.5.5.2.4 for the GPC calibration verification solution analyses.

- Chromatogram and data system report(s) for the mid-point initial calibration standard associated with the GPC calibration verification solution labeled as specified in Section 2.5.5.3.15 (i.e., Individual Standard Mixtures A and B or C from the initial calibration sequence).

2.5.5.6 Raw Florisil Data

The chromatogram and data system report(s) shall be arranged in chronological order by Florisil cartridge performance check analyses.

- Chromatograms and data system reports, labeled as specified in Sections 2.5.5.2.2 and 2.5.5.2.4 for the Florisil cartridge performance check analyses.

- Chromatograms and data system reports for the mid-point initial calibration standard associated with the Florisil cartridge performance check analysis, labeled as specified in Section 2.5.5.3.15 (i.e., Individual Standard Mixture A, Individual Standard Mixture B, Individual Standard Mixture C, and the 2,4,5-Trichlorophenol solution).

2.5.6 Aroclors Data

2.5.6.1 Aroclors QC Summary

2.5.6.1.1 Surrogate Recovery (Form II ARO-1, ARO-2).

2.5.6.1.2 Matrix Spike/Matrix Spike Duplicate Recovery (Form III ARO-1, ARO-2): MS/MSD is required for the Aroclors fraction, unless otherwise specified by the USEPA Region. See Exhibit D - Analytical Methods for Aroclors, for frequency.

2.5.6.1.3 LCS Recovery (Form III ARO-3, ARO-4).

2.5.6.1.4 Method Blank Summary (Form IV ARO): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.

2.5.6.2 Aroclors Sample Data

Sample data shall be arranged in packets with the Aroclors Organics Analysis Data Sheet (Form I ARO), followed by the raw data for Aroclor samples. These sample packets should then be placed in order of increasing EPA Sample Number, considering both letters and numbers.

NOTE: For a Sample analysis in which "S" flags are reported a Form I ARO is required for the original analysis (EPA Sample Number = xxxxx) in which "S" flags are reported, and a Form I ARO is required for the billable reanalysis (EPA Sample Number = XXXXXRE) of the sample performed after a valid 5-point calibration of the detected Aroclor. An additional Form I ARO is required for any necessary dilutions (EPA Sample Number = XXXXXDL).

2.5.6.2.1 Target Compound Results, Aroclors Organics Analysis Data Sheet (Form I ARO). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C - Aroclors) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (Section 2.5.1). In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.5.6.2.2 Copies of Aroclor Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of RTs on the data system printout if RTs are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D - Analytical Methods for Aroclors, and shall be labeled with the following information:

- EPA Sample Number;
- Volume injected (µL);
- Date and time of injection;
- On column concentration/amount including units;
- GC column identifier (by stationary phase and internal diameter);
- GC instrument identifier; and
- Scaling factor (label the x and y axes using a numerical scale).

2.5.6.2.3 Copies of Aroclor chromatograms from the second GC column shall be included and labeled as in Section 2.5.6.2.2.

2.5.6.2.4 Data System Printout

A printout of RT, corresponding peak height or peak area, and the on column amount shall accompany each chromatogram. The printout shall be labeled with the EPA Sample Number and standard concentration level. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range. The GC/MS Operator shall also mark each integrated area with the letter "m" in the quantitation report.

2.5.6.2.5 All manual worksheets shall be included in the Sample Data Package.

2.5.6.2.6 Other Required Information. If Aroclors are confirmed by GC/MS, the Contractor shall submit copies of reconstructed ion chromatograms. Raw spectra and background-subtracted mass spectra must be submitted for at least three major peaks of Aroclor target compounds (see Exhibit C - Aroclors) that are

identified in the sample and corresponding standard mass spectra. Compound names shall be clearly marked on all spectra.

2.5.6.3 Aroclors Standards Data

2.5.6.3.1 Initial Calibration of Aroclors (Form VI ARO-1, ARO-2, and ARO-3): For all GC columns, all instruments, in chronological order by GC column and instrument.

2.5.6.3.2 Calibration Verification Summary (Form VII ARO): For all calibration verification standards on all GC columns and instruments, in chronological order by GC column and instrument.

2.5.6.3.3 Analytical Sequence (Form VIII ARO): For all GC columns and instruments, in chronological order by GC column and instrument.

2.5.6.3.4 Identification Summary for Multicomponent Analytes (Form X ARO): For all samples with positively identified Aroclors, in order by increasing EPA Sample Number.

2.5.6.3.5 Chromatograms and data system printouts shall be included for all standards, including the following:

- All Aroclor standards used for initial calibration on each GC column and instrument.
- All Aroclor standards used for calibration verification on each GC column and instrument.
- All Aroclor standards analyzed for confirmation.

2.5.6.3.6 A printout of RT and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA Sample Number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D - Analytical Methods for Aroclors, and shall be labeled with the following:

- EPA Sample Number for the standard (e.g., AR10161OK, AR12601OK). See Section 3 for details.
- Label all standard peaks with the compound name, either directly out from the peak on the chromatogram, or on the printout of RTs on the data system printout, if RTs are printed over the peak on the chromatogram.
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram.
- Date and time of injection.
- GC column identifier (by stationary phase and internal diameter).

- GC instrument identifier.

- Scaling factor (label the x and y axes using a numerical scale).

NOTE: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/ECD Operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range. The GC/MS Operator shall also mark each integrated area with the letter "m" on the quantitation report.

2.5.6.4 Aroclors Raw Quality Control (QC) Data

2.5.6.4.1 Blank data shall be arranged in chronological order by extraction date.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I ARO).

- Chromatogram(s) and data system printout(s) for each GC column and instrument used for analysis, labeled as in Sections 2.5.6.2.2 and 2.5.6.2.4.

2.5.6.4.2 Aroclors Laboratory Control Sample (LCS) Data

- Tabulated results (Form I ARO) of target compounds for both GC columns.

- Chromatograms and data system printouts for both GC columns, labeled as in Sections 2.5.6.2.2 and 2.5.6.2.4.

2.5.6.4.3 Aroclors Matrix Spike Data

- Tabulated results (Form I ARO) of target compounds for both GC columns.

- Chromatogram(s) and data system printout(s) for both GC columns, labeled as in Sections 2.5.6.2.2 and 2.5.6.2.4.

2.5.6.4.4 Aroclors Matrix Spike Duplicate Data

- Tabulated results (Form I ARO) of target compounds for both GC columns.

- Chromatogram(s) and data system printout(s) for both GC columns, labeled as in Sections 2.5.6.2.2 and 2.5.6.2.4.

2.5.6.5 Raw Gel Permeation Chromatograph (GPC) Data

2.5.6.5.1 GPC Calibration. The UV traces for the GPC calibration solution, chromatograms, and the data system reports for the GPC blank shall be arranged in chronological order for the GPC calibration.

- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of RTs when the RTs are printed directly over the peak.

- Chromatogram and data system report(s) labeled as specified in Sections 2.5.6.2.2 and 2.5.6.2.4 for GPC blank analyses.

- Chromatogram and data system report(s) for the mid-point initial calibration standard associated with the GPC blank labeled as specified in Section 2.5.6.3.6 (i.e., AR1016OK, AR1260OK from the initial calibration).

2.6 Complete SDG File (CSF)

As specified in Section 1, the Contractor shall deliver one CSF (including the original Sample Data Package) to the USEPA Region concurrently with delivery of the Sample Data Package to SMO. Delivery to USEPA's designated recipients (e.g., QATS) is only required upon written request.

2.6.1 The CSF will contain all original documents specified in Sections 3 and 4 and on Form DC-2 (Section 3.20). No photocopies of original documents will be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to USEPA with another Case/SDG in accordance with the requirements described in Exhibit F. The contents of the CSF shall be numbered according to the specifications described in Section 3.20.

2.6.2 The CSF will consist of the following original documents in addition to the documents in the Sample Data Package.

NOTE: All SDG-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other SDG-specific documents generated after the CSF is sent to USEPA, as well as copies that are altered in any fashion, are also deliverables to USEPA. Deliver the original to the USEPA Region and a copy to SMO. Delivery to USEPA's designated recipients (e.g., QATS) is only upon written request.

2.6.2.1 Original Sample Data Package

2.6.2.2 A completed and signed Organics CSF Inventory Sheet (Form DC-2).

2.6.2.3 All original shipping documents including, but not limited to, the following documents:

- Airbills (if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information);

- USEPA Sample TR/COCs; and

- Sample tags (if present) sealed in plastic bags.

2.6.2.4 All original receiving documents including, but not limited to, the following documents:

- Form DC-1;
- Other receiving forms or copies of receiving logbooks; and
- SDG Cover Sheet.

2.6.2.5 All original laboratory records, not already submitted in the Sample Data Package, of sample transfer, preparation, and analysis including, but not limited to, the following documents:

- Log book preparation entries documenting the steps and calculations of diluted and working standards and/or receipt of stock standards showing the lot number and date of receipt or date of preparation for all standards and spiking solutions;
- Original preparation and analysis forms or copies of preparation and analysis logbook pages;
- Internal sample and sample extract transfer chain-of-custody records;
- Screening records; and
- All instrument output, including strip charts from screening activities.

2.6.2.6 All other original SDG-specific documents in the possession of the Contractor including, but not limited to, the following documents:

- Telephone contact logs;
- Copies of personal logbook pages;
- All handwritten SDG-specific notes; and
- Any other SDG-specific documents not covered by the above.

2.6.3 If the Contractor does submit SDG-specific documents to USEPA after submission of the CSF, the documents should be identified with unique accountable numbers, a revised Form DC-2 should be submitted, and the unique accountable numbers and locations of the documents in the CSF should be recorded in the "Other Records" section on the revised Form DC-2. Alternatively, the Contractor may number the newly submitted SDG-specific documents to USEPA as a new CSF and submit a new Form DC-2. The revised Form DC-2 or new Form DC-2 should be submitted to the USEPA Region only.

2.7 Electronic Data Deliverable

The Contractor shall provide an electronic data deliverable on analytical data for all samples in the SDG, as specified in Exhibit H, and delivered as specified in the Contract Schedule (Performance/Delivery Schedule).

2.8 Delivery of Hardcopy Data in PDF Format

In addition to all required deliverables identified in the laboratory's contract and the SOM01.1 SOW, the laboratory shall provide a complete copy of the hardcopy deliverable in PDF on a Compact Disc (CD) if requested by the Region.

2.8.1 The PDF file should be organized in accordance to directions provided in Exhibit B, "Reporting Requirements and Order of Data Deliverables" of the SOM01.1 SOW. The PDF file shall be bookmarked as described below for ease of data retrieval and navigation.

2.8.2 Organic data shall be bookmarked using a hierarchal bookmark structure (i.e., an overview or "parent" bookmark, and a subordinate or "child" bookmark nested underneath the "parent" bookmark). The required hierarchal bookmark structure is shown in Table 2.

TABLE 2

Hierarchal Bookmark Structure

Group Bookmark	Parent Bookmark	Child Bookmarks
Sample TR/COCs, TR/COC Cover Sheet, and SDG Narrative		
VOA (SIM and Trace)	QC Summary	Deuterated Monitoring Compound Summary
		Matrix Spike/Matrix Spike Duplicate Summary
		Method Blank
		GC/MS Instrument Performance Check
		Internal Standard Area and RT Summary
	Sample Data	Samples in increasing alphanumeric EPA Sample Number order (with supporting raw data)
	Standards Data	Initial Calibration Data
		CCV Data, including closing CCV
	Raw QC Data	BFB Data
		Blank Data
		Matrix Spike Data
		Matrix Spike Duplicate Data
VOA (Low/Med)	QC Summary	Deuterated Monitoring Compound Summary
		Matrix Spike/Matrix Spike Duplicate Summary
		Method Blank
		GC/MS Instrument Performance Check
		Internal Standard Area and RT Summary
	Sample Data	Samples in increasing alphanumeric EPA Sample Number order (with supporting raw data)
	Standards Data	Initial Calibration Data
		CCV Data, including closing CCV
	Raw QC Data	BFB Data
		Blank Data
		Matrix Spike Data
		Matrix Spike Duplicate Data

TABLE 2

Hierarchal Bookmark Structure (Con't)

SVOA (SIM and Low/Med)	QC Summary	Deuterated Monitoring Compound Summary
		Matrix Spike/Matrix Spike Duplicate Summary
		Method Blank
		GC/MS Instrument Performance Check
		Internal Standard Area and RT Summary
	Sample Data	Samples in increasing alphanumeric EPA Sample Number order (with supporting raw data)
	Standards Data	Initial Calibration Data
		CCV Data, including closing CCV
	Raw QC Data	DFTPP Data
		Blank Data
		Matrix Spike Data
		Matrix Spike Duplicate Data
		Raw GPC Data
PEST	QC Summary	Surrogate Recovery Summary
		Matrix Spike/Matrix Spike Duplicate Summary
		Laboratory Control Sample Summary
		Method Blank Summary
	Sample Data	Samples in increasing alphanumeric EPA Sample Number order (with supporting raw data)
	Standards Data	Initial Calibration/Single Component
		Initial Calibration/Multi Component
		Analyte Resolution Summary
		Performance Evaluation Mixture
		Individual Standard Mixtures A and B, or Mixture C
		Calibration Verification Summary
		Analytical Sequence
		Florisil Cartridge Check
		GPC Calibration
		Identification Summary for Single Component
		Identification Summary of Multi Component
		Chromatograms and Data System Printouts
	Raw QC Data	Blank Data
		Matrix Spike Data
		Matrix Spike Duplicate Data
		Laboratory Control Sample Data
		Raw GPC Data
		Raw Florisil Data

TABLE 2

Hierarchal Bookmark Structure (Con't)

Group Bookmark	Parent Bookmark	Child Bookmarks
ARO	QC Summary	Surrogate Recovery Summary
		Matrix Spike/Matrix Spike Duplicate Summary
		Laboratory Control Sample Summary
		Method Blank Summary
	Sample Data	Samples in increasing alphanumeric EPA Sample Number order (with supporting raw data)
	Standards Data	Initial Calibration Aroclors
		Calibration Verification Summary
		Analytical Sequence
		Identification Summary for Aroclors
		Chromatograms and Data System Printouts
	Raw QC Data	Blank Data
		Matrix Spike Data
		Matrix Spike Duplicate Data
		Laboratory Control Sample Data
		Raw GPC Data
Miscellaneous		DC-1 and DC-2 Forms, logbook information, sample tags, etc.

2.9 Preliminary Results

The Form Is and Form Xs data results shall be submitted for all samples in one SDG of a Case. This includes tabulated target compound results (Form I) for the volatile, semivolatile, pesticide, and Aroclor fractions; TICs (Form I TIC) for the volatile and semivolatile fractions; and Identification Summaries (Form X) for the pesticide and Aroclor fractions. The Contractor shall clearly identify the Preliminary Results by labeling each Form I and Form I TIC as "Preliminary Results" under each form title (e.g., under Volatile Organics Analysis Data Sheet, Volatile Organics Analysis Data Sheet Tentatively Identified Compounds).

2.10 GC/MS and GC/ECD Electronic Deliverables

The Contractor shall adhere to the requirements in Exhibit E.

2.11 Extracts

The Contractor shall preserve sample extracts at 4°C (±2°C) in bottles/vials with polytetrafluoroethylene (PTFE)-lined septa. Extract bottles/vials shall be labeled with EPA Sample Number, Case Number, and SDG Number. The Contractor shall maintain a logbook of stored extracts, listing EPA Sample Numbers and associated Case and SDG numbers. The Contractor shall retain extracts for 365 days following submission of the reconciled, complete Sample Data Package. During that time, the Contractor shall submit extracts and associated logbook pages within 7 days following receipt of a written request from the CLP PO.

3.0 FORMS INSTRUCTIONS

3.1 Introduction

This section includes specific instructions for completing the data reporting forms required under the contract. Each of the forms are specific to a given fraction (volatile, semivolatile, pesticide, or Aroclor) and, in some instances, specific to a given matrix (water or soil/sediment) within each fraction. The Contractor shall submit only those forms pertaining to the fractions analyzed for a given sample(s). For instance, if a sample is scheduled for volatiles analysis only, the Contractor shall provide only forms for the volatile fraction.

3.2 General Information

The Contractor shall report values on the hardcopy forms according to the individual form instructions in this section. For example, results for concentrations of volatile target compounds shall be reported to two significant figures if the value is greater than or equal to 5.0. Values that exceed the maximum length allowed shall be reported to the maximum possible, maintaining the specified decimal place. Unless otherwise specified, all values must be reported to at least two significant figures.

3.2.1 The data reporting forms presented in Section 4 have been designed in conjunction with the computer-readable data format specified in Exhibit H. Information entered on these forms shall **not** exceed the size of the field given on the form, including such laboratory-generated items as "Lab Name" and "Lab Sample ID".

NOTE: The space provided for entries on the hardcopy forms (Section 4) is greater in some instances than the length prescribed for the variable as written to the electronic deliverable (Exhibit H). Greater space is provided on the hardcopy forms for visual clarity.

3.2.2 When submitting data, the Contractor shall reproduce **all** characters that appear on the data reporting forms in Section 4. The format of the forms submitted shall be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval from the USEPA Regional Contract Laboratory Program Project Officer (CLP PO). The names of the various fields and compounds (i.e., "Lab Code", "Chloromethane") shall appear as they do on the forms in the contract, including the options specified in the form [i.e., "Matrix: (soil/sed/water)" shall appear, not just "Matrix"].

3.2.3 If an entry does not fill the entire blank space provided on the form, null characters shall be used to remove the remaining underscores that comprise the blank line. However, the Contractor shall **not** remove the underscores or vertical bars that delineate "boxes" on the forms. The only exception would be those underscores at the bottom of a "box" that are intended as a data entry line. (For instance, on Form 2A, line 30, if data is entered on line 30, it will replace the underscores.)

3.3 Header Information

Six pieces of information are common to the header section of each data reporting form: Laboratory Name (Lab Name); Contract; Laboratory Code (Lab Code); Case Number; Modification Reference Number (Mod. Ref. No.);

and Sample Delivery Group (SDG) Number (SDG No.). Except as noted for Mod. Ref. No., this information shall be entered on every form and shall match on every form.

3.3.1 Laboratory Name. The "Lab Name" shall be the name chosen by the Contractor to identify the laboratory. It shall not exceed 25 characters.

3.3.2 Contract. The "Contract" refers to the number of the USEPA contract under which the analyses were performed.

3.3.3 Laboratory Code. The "Lab Code" is an alphabetical abbreviation of up to six letters, as assigned by USEPA, to identify the laboratory and aid in data processing. This Laboratory Code will be assigned by USEPA at the time a contract is awarded, and shall not be modified by the Contractor, except at the direction of USEPA. If a change of name or ownership occurs at the laboratory, the Laboratory Code will remain the same until the Contractor is directed by USEPA to use another Laboratory Code.

3.3.4 Case Number. The "Case No." is the Sample Management Office (SMO)-assigned Case Number (to five characters) associated with the sample. This number is reported on the Traffic Report/Chain of Custody Record (TR/COC).

3.3.5 Modification Reference Number. The "Mod. Ref. No." is the USEPA-assigned number for analyses performed under the modified analysis clause in Exhibit A, Section 4.2.2.11. If sample analyses are performed under the modified analysis clause, the Contractor shall list both the Case Number and the Modification Reference Number on all forms. If there are no modified analysis requirements, leave the "Mod. Ref. No." field blank.

3.3.6 SDG Number. The "SDG No." field is for the SDG Number. It is the EPA Sample Number of a field sample assigned to the SDG and shall be unique for each SDG within a Case. When several samples are received together in the first SDG shipment, the SDG Number shall be the lowest Sample Number (considering both alpha and numeric designations) in the first group of samples received under the SDG. If fractions of the same field samples are scheduled under different turnaround times, thus creating separate SDGs containing the same Sample Numbers, a different Sample Number shall be utilized in the assignment of the SDG Number for each SDG. If a situation arises where there are an insufficient number of samples for assignment of SDG numbers (i.e., 1 sample with a 7-day turnaround for volatile analyses and a 14-day turnaround for semivolatile, pesticide, and Aroclor analyses), the Contractor shall contact SMO for the assignment of an SDG Number.

3.3.7 Sample Number. The "EPA Sample No." appears either in the header information of the form, or as the left column of a table summarizing data from a number of samples. When the EPA Sample Number is entered in the triple-spaced box in the upper right-hand corner of Form I, Form III, Form IV, Form V, or Form X, it should be entered on the middle line of the three lines that comprise the box.

3.3.7.1 The Contractor shall identify **all** samples, including: dilutions; reanalyses; Laboratory Control Samples (LCSs); requested Matrix Spike and Matrix Spike Duplicates (MS/MSDs); blanks; instrument performance check; and standards with an EPA Sample Number. For field samples, Matrix Spikes, and Matrix Spike Duplicates, the EPA

Sample Number is the unique identifying number given on the TR/COC that accompanied that sample. In order to facilitate data assessment, the Contractor shall use the following sample suffixes:

XXXXX	=	EPA Sample Number
XXXXXMS	=	Matrix Spike (MS) sample
XXXXXMSD	=	Matrix Spike Duplicate (MSD) sample
XXXXXRX	=	Reextracted and reanalyzed sample.
XXXXXRE	=	Reanalyzed (reinjected) sample.
XXXXXDL	=	The suffix DL is appended to the EPA Sample Number to indicate that the analytical results are a result of a dilution of the original analysis (reported as EPA Sample XXXXX). See Exhibit D for dilution requirements.
XXXXXDL2	=	Samples analyzed at a secondary dilution.
XXXXXDL3	=	Samples analyzed at a third dilution.
XXXXXME	=	Soil samples analyzed using the medium-level method when a billable low-level analysis of the same sample is also present.

3.3.7.2 There may be instances when all samples analyzed must be listed on the form, regardless of whether or not they are part of the SDG being reported (e.g., Form VIII PEST). In these instances, use ZZZZZ as the EPA Sample Number for any sample analysis **not** associated with the SDG being reported.

3.3.7.3 For blanks, the Contractor shall use the following identification scheme for the EPA Sample Number:

- Volatile method blanks shall be identified as VBLK##.
- Volatile instrument blanks shall be identified as VIBLK##.
- Volatile storage blanks shall be identified as VHBLK##.
- Semivolatile method blanks shall be identified as SBLK##.
- Pesticide method blanks shall be identified as PBLK##.
- Pesticide sulfur cleanup blanks shall be identified as PSBLK##.
- Pesticide instrument blanks shall be identified as PIBLK##.
- Aroclor method blanks shall be identified as ABLK##.
- Aroclor sulfur cleanup blanks shall be identified as ASBLK##.
- Aroclor instrument blanks shall be identified as AIBLK##.

3.3.7.3.1 The EPA Sample Number shall be unique for each blank within an SDG. Within a fraction, the Contractor shall achieve this by replacing the two-character suffix (##) of the identifier with one or two characters or numbers, or a combination of both.

For example, possible identifiers for volatile blanks would be VBLK1, VBLK2, VBLKA1, VBLKB2, VBLK10, VBLKAB, etc.

3.3.7.3.2 If the method blank is analyzed on multiple instruments, then an additional two-character suffix shall be added to make the blank EPA Sample Number unique.

3.3.7.4 The EPA Sample Number shall be unique for each LCS within the SDG. The LCSs shall be identified as follows:

- Pesticides LCS - PLCS##
- Aroclor LCS - ALCS##

Where:

P or A = Fraction (P for pesticides and A for Aroclors)

LCS = Laboratory Control Sample

= Suffix consisting of characters or numbers or both that makes the EPA Sample Number for the LCS unique in the SDG.

(1) = When reporting results on Form I, a "(1)" is appended onto the EPA Sample Number to indicate that the results are from Gas Chromatograph (GC) column (1), [e.g., PLCS01(1)].

(2) = When reporting results on Form I, a "(2)" is appended onto the EPA Sample Number to indicate that the results are from GC column (2), [e.g., ALCS01(2)].

3.3.7.5 Volatile and semivolatile instrument performance checks shall be identified as BFB## (Volatiles) and DFTPP## (Semivolatiles) where:

BFB = Bromofluorobenzene (instrument performance check compound for Volatiles analysis).

DFTPP = Decafluorotriphenylphosphine (instrument performance check compound for Semivolatiles analysis).

= One or two characters, numbers, or combinations of both to create a unique EPA Sample Number within an SDG.

3.3.7.6 Volatile and semivolatile standards shall be identified as FSTD***##, where:

F = Fraction code (V for volatiles; S for semivolatiles).

STD = Standard.

*** = Concentration of volatile standards in µg/L [e.g., 005, 010, 050, 100, and 200, or 0.5, 001, 005, 010, and 020, when trace level volatiles analyses are performed, or 0.05, 0.1, 0.5, 1.0, and 2.0 when trace level analyses by the Selected Ion Monitoring (SIM) technique are performed] or the concentration injected in ng/µL for semivolatile standards (e.g., 005, 010, 020, 040, and 080, or 0.1, 0.2, 0.4, 0.8, and 001, when optional analyses of Polynuclear Aromatic Hydrocarbons (PAHs)/pentachlorophenol are performed).

= One or two characters, numbers, or combinations of both to create a unique EPA Sample Number within an SDG.

3.3.7.7 The Contractor shall use the following scheme to identify pesticide and Aroclor standards:

Name	EPA Sample Number
Individual Mix A (CS1)	INDA1##
Individual Mix A (CS2)	INDA2##
Individual Mix A (CS3)	INDA3##
Individual Mix A (CS4)	INDA4##
Individual Mix A (CS5)	INDA5##
Individual Mix B (CS1)	INDB1##
Individual Mix B (CS2)	INDB2##
Individual Mix B (CS3)	INDB3##
Individual Mix B (CS4)	INDB4##
Individual Mix B (CS5)	INDB5##
Resolution Check	RESC##
Performance Evaluation Mixture	PEM##
Toxaphene (CS1)	TOXAPH1##
Toxaphene (CS2)	TOXAPH2##
Toxaphene (CS3)	TOXAPH3##
Toxaphene (CS4)	TOXAPH4##
Toxaphene (CS5)	TOXAPH5##
Aroclor 1016 (CS1)	AR10161##
Aroclor 1016 (CS2)	AR10162##
Aroclor 1016 (CS3)	AR10163##
Aroclor 1016 (CS4)	AR10164##
Aroclor 1016 (CS5)	AR10165##
Aroclor 1221 (CS1)	AR12211##
Aroclor 1221 (CS2)	AR12212##
Aroclor 1221 (CS3)	AR12213##
Aroclor 1221 (CS4)	AR12214##
Aroclor 1221 (CS5)	AR12215##
Aroclor 1232 (CS1)	AR12321##
Aroclor 1232 (CS2)	AR12322##

Name	EPA Sample Number
Aroclor 1232 (CS3)	AR12323##
Aroclor 1232 (CS4)	AR12324##
Aroclor 1232 (CS5)	AR12325##
Aroclor 1242 (CS1)	AR12421##
Aroclor 1242 (CS2)	AR12422##
Aroclor 1242 (CS3)	AR12423##
Aroclor 1242 (CS4)	AR12424##
Aroclor 1242 (CS5)	AR12425##
Aroclor 1248 (CS1)	AR12481##
Aroclor 1248 (CS2)	AR12482##
Aroclor 1248 (CS3)	AR12483##
Aroclor 1248 (CS4)	AR12484##
Aroclor 1248 (CS5)	AR12485##
Aroclor 1254 (CS1)	AR12541##
Aroclor 1254 (CS2)	AR12542##
Aroclor 1254 (CS3)	AR12543##
Aroclor 1254 (CS4)	AR12544##
Aroclor 1254 (CS5)	AR12545##
Aroclor 1260 (CS1)	AR12601##
Aroclor 1260 (CS2)	AR12602##
Aroclor 1260 (CS3)	AR12603##
Aroclor 1260 (CS4)	AR12604##
Aroclor 1260 (CS5)	AR12605##
Aroclor 1262 (CS1)	AR12621##
Aroclor 1262 (CS2)	AR12622##
Aroclor 1262 (CS3)	AR12623##
Aroclor 1262 (CS4)	AR12624##
Aroclor 1262 (CS5)	AR12625##
Aroclor 1268 (CS1)	AR12681##
Aroclor 1268 (CS2)	AR12682##
Aroclor 1268 (CS3)	AR12683##
Aroclor 1268 (CS4)	AR12684##
Aroclor 1268 (CS5)	AR12685##
Aroclor 1016/1260 Mix (CS1)	AR16601##
Aroclor 1016/1260 Mix (CS2)	AR16602##
Aroclor 1016/1260 Mix (CS3)	AR16603##
Aroclor 1016/1260 Mix (CS4)	AR16604##
Aroclor 1016/1260 Mix (CS5)	AR16605##

The Contractor shall replace the two-character suffix (##) of the identifier with one or two characters or numbers, or a combination of both, to create a unique EPA Sample Number within an SDG.

If one individual mix is used (Individual Mix C) then the EPA Sample Number will be INDC1## for CS1, INDC2## for CS2, etc.)

3.3.7.8 For pesticide and Aroclor standards, if the standards are injected onto both GC columns on the same instrument simultaneously, the same EPA Sample Number may be used for reporting data for the standards for both columns. If simultaneous injections are **not** made, then the same number shall **not** be used.

3.3.7.9 The EPA Sample Number for Gel Permeation Chromatograph (GPC) shall be GPC#########, where ######### is the GPC column ID. If the GPC column ID is more than nine characters, truncate at the ninth character.

3.3.7.10 The EPA Sample Number for Florisil shall be FLO#########, where ######### is the Florisil cartridge lot number. If the Florisil cartridge lot number is more than nine characters, truncate at the ninth character.

3.3.8 Other Common Fields. Several other pieces of information are common to many of the data reporting forms. These include matrix, sample weight/volume, level, Laboratory Sample Identifier, and Laboratory File Identifier.

- In the "Matrix" field, enter "Soil" for soil samples, "Sed" for sediment samples, and "Water" for water samples.

- In the "Sample wt/vol" field, enter the number of grams (for soil or sediment) or mL (for water) of sample used in the first blank. Report weights and volumes to 3 significant figures (e.g., 30.0 g, 5.00 g). Enter the units, either g or mL, in the second blank.

- The "Level" field is used for the volatile and semivolatile fractions. Enter the determination of concentration level made from the screening of soils. Enter as "TRACE" (trace volatile water only), "LOW" (volatile water and volatile and semivolatile soil), or "MED" (soil only), **not** "L" or "M".

  NOTE: There is no differentiation between low and medium soil samples for the pesticide and Aroclor fractions, and no level is entered on any of these forms.

- The "Purge Volume" field is used for volatile samples and associated calibration standards to describe the total volume of sample or calibration standard that is analyzed. For water and medium-level soil samples and their associated calibration standards, the value to be entered is "5.0 mL". For low-level soil samples and their associated calibration standards, the value to be entered is "10.0 mL".

- The Laboratory Sample Identifier is a unique laboratory-generated internal identifier pertaining to a particular analysis. The Contractor must enter the Laboratory Sample Identifier using alpha-numeric characters in the "Lab Sample ID" field. The Contractor may use the EPA Sample Number as the Laboratory Sample Identifier.

- The Laboratory File Identifier is the unique laboratory-generated name of the GC/MS data system file containing information pertaining to a particular analysis. The Contractor must enter the Laboratory File Identifier using alpha-numeric characters in the "Lab File ID" field.

3.3.8.1 The "Instrument ID" field is common to the forms containing calibration data. The identifier used by the Contractor shall include some indication of the manufacturer and/or model of the instrument, and shall contain additional characters that differentiate between all instruments of the same type in the laboratory.

3.3.8.2 Forms II, IV, V, VIII, IX, and X contain a field labeled "Page _ of _" in the bottom left-hand corner. If the number of entries required on any of these forms exceeds the available space, continue entries on another copy of the same fraction-specific form, duplicating all header information. If a second page is required, number the pages consecutively (i.e., "Page 1 of 2" and "Page 2 of 2"). If a second page is **not** required, number the page "Page 1 of 1".

NOTE: These forms are fraction-specific, and often matrix-specific within a fraction. For example, Form II VOA-1 and Form II VOA-3 are for different data. Therefore, **do not** number the pages of all 12 versions of Form II as "1 of 12", "2 of 12", etc. Number only pages corresponding to the fraction-specific and matrix-specific form.

3.3.9 Rounding Rule. For rounding off numbers to the appropriate level of precision, the Contractor shall follow these rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than or equal to 5, drop it and increase the last digit to be retained by 1 (round up).

3.4 Organics Analysis Data Sheet (Form I, All Fractions)

3.4.1 Purpose

This form is used for tabulating and reporting sample analysis, including dilutions, reanalysis, blank, LCS, and requested MS/MSD results for target compounds. If all fractions are not requested for analysis, only the pages for the fractions required shall be submitted. For example, if only volatiles analysis is requested, Form I VOA-1, VOA-2, and Form I VOA-TIC shall be submitted. An additional Form I, VOA-SIM will be required if the optional Selected Ion Monitoring (SIM) analysis is performed. If only semivolatiles analysis is requested [without the (optional) PAHs/pentachlorophenol by SIM analysis], Form I SV-1, SV-2, and Form I SV-TIC shall be submitted. Form I SV-SIM shall be submitted only if the (optional) PAHs/pentachlorophenol by SIM analysis is requested. If only the pesticide and Aroclor fractions are requested for analysis, Form I PEST and Form I ARO shall be submitted. **Furthermore, pesticide instrument blanks (PIBLKs) shall be reported on a per column/per analysis basis on Form I PEST. Each PIBLK shall be named with a unique EPA Sample Number. Distinguish between GC Column (1) and GC Column (2) results by appending a suffix "(1)" for GC Column (1) and "(2)" for GC Column (2).**

3.4.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.4.2.1 For soil and sediment samples analyzed for volatiles, enter the non-decanted Percent Moisture in the "% Moisture: not dec." field

on Form I VOA-1, VOA-2, VOA-TIC. This is the only Percent Moisture determination made for volatiles since the entire contents of the VOA vial are considered as the sample. For water samples, leave this field blank.

3.4.2.2 For soil and sediment samples analyzed for semivolatiles, pesticides, and Aroclors, enter the values for the Percent Moisture determined during the analysis in the "% Moisture" field on Form I SV-1, SV-2, SV-SIM, SV-TIC, Form I PEST, and Form I ARO. In the "Decanted: (Y/N)" field, enter "Y" if the sample had standing water above the soil or sediment that was decanted, or "N" if no water was decanted off the surface of the sample. Report Percent Moisture (decanted or not decanted) to two significant figures (e.g. 5.3 is 5.3, but 10.3 is 10). For water samples, method blanks, sulfur cleanup blanks, and instrument blanks, leave these fields blank on Form I.

3.4.2.3 For volatiles, enter the GC Column Identifier in the "GC Column" field on Form I VOA-1, VOA-2, VOA-SIM and the internal diameter in mm, to two decimal places, in the "ID" field.

3.4.2.4 For semivolatiles, pesticides, and Aroclors, enter the method of extraction in the "Extraction: (Type)" field on Form I SV-1, SV-2, SV-SIM, SV-TIC, PEST, and ARO, as "SEPF" for separatory funnel, "CONT" for continuous liquid-liquid extraction without hydrophobic membrane, "CONH" for continuous liquid-liquid extraction with hydrophobic membrane, "SONC" for sonication (soils only), "SOXH" for Soxhlet Extraction (soils only), or "PFEX" for Pressurized Fluid Extraction (soils only).

3.4.2.5 If GPC was performed, enter "Y" in the "GPC Cleanup" field on Form I SV-1, SV-2, SV-SIM, SV-TIC, PEST, or ARO. Enter "N" in this field if GPC was not performed. If GPC was performed and only half of the extract was collected enter "2.0" in the "GPC Factor" field on Form I SV-1, SV-2, SV-SIM, SV-TIC, PEST, or ARO. If GPC was performed and all of the extract was collected or if GPC was not performed enter "1.0" in the "GPC Factor" field.

NOTE: GPC is **required** for all **soil** samples analyzed for semivolatiles and pesticides; therefore, all Forms I for semivolatiles and pesticides soil samples will contain a "Y" in this field. GPC cleanup is optional for soil samples analyzed for Aroclors and for water samples analyzed for semivolatiles, pesticides, and Aroclors.

3.4.2.6 For Aroclor samples, enter "Y" in the "Acid Cleanup" field on Form I ARO.

NOTE: Acid cleanup is required for all samples analyzed for Aroclors; therefore, all Forms I ARO will contain a "Y" in this field.

3.4.2.7 For soil samples only, enter the pH for semivolatiles, pesticides, and Aroclors, reported to 0.1 pH units, on Form I SV-1, SV-2, SV-SIM, SV-TIC, PEST, and ARO.

3.4.2.8 Enter the date of sample receipt at the laboratory, as noted on the TR/Chain of Custody Record [i.e., the Validated Time of Sample Receipt (VTSR)], in the "Date Received" field. The date shall be entered as MM/DD/YYYY.

3.4.2.9 Complete the "Date Extracted" and "Date Analyzed" fields in the same format (MM/DD/YYYY). When continuous liquid-liquid extraction procedures are used for water samples, enter the date that the procedure was **started** in the "Date Extracted" field. If separatory funnel (pesticides and Aroclors only), sonication, soxhlet, Soxhlet Dean-Stark (SDS) extraction, or pressurized fluid procedures are used, enter the date that the procedure was **completed** in the "Date Extracted" field. For pesticide and Aroclor samples, enter the date of the first GC analysis performed in the "Date Analyzed" field. The date of sample receipt will be compared with the extraction and analysis dates of each fraction to ensure that contract holding times were not exceeded.

3.4.2.10 If a medium soil sample is analyzed for volatiles, enter the total volume of the methanol extract in microliters (µL) in the "Soil Extract Volume" field on Form I VOA-1, VOA-2, and VOA-TIC. This volume includes any methanol not collected from the filtration of the extract through glass wool; the volume is typically 5,000 µL (i.e., the 5 mL of methanol used for the extraction). If a medium soil sample is analyzed, enter the volume of the methanol extract added to the reagent water in the purge tube and analyzed in the "Soil Aliquot Volume" field. Enter this volume in µL.

3.4.2.11 For semivolatiles, pesticides, and Aroclors, enter the actual volume of the **most** concentrated sample extract, in µL, in the "Concentrated Extract Volume" field on Form I SV-1, SV-2, SV-TIC, SV-SIM, PEST, and ARO. For semivolatiles, this volume will typically be 1,000 µL (for water) or 500 µL (for water and soil) when GPC is performed and only 500 µL of extract is collected after GPC Cleanup. If the entire extract is collected after GPC Cleanup then this volume will typically be 1,000 µL. For pesticides and Aroclors, the volume of the most concentrated extract will typically be 10,000 µL (for water) or 5,000 µL (for water and soil) when GPC is performed. If the entire extract is collected after GPC Cleanup then this volume will typically be 10,000 µL for pesticide and Aroclor analyses and 1,000 µL for semivolatile analysis. For pesticides and Aroclors, the volume of the most concentrated extract is **not** the volume taken through the Florisil and sulfur cleanup steps. If a dilution of the sample extract is made in a subsequent analysis, this volume will remain the same, but the Dilution Factor (DF) will change.

3.4.2.12 For semivolatiles, pesticides, and Aroclors, enter the volume of the sample extract injected into the GC in the "Injection Volume" field on Form I SV-1, SV-2, SV-SIM, SV-TIC, PEST, and ARO. Report this volume in µL to one decimal place (e.g., 1.0 µL).

3.4.2.12.1 If pesticides or Aroclors are analyzed using two GC columns connected to a single injection port, enter the amount of half the volume in the syringe in the "Injection Volume" field (i.e., assume that the extract injected is evenly divided between the two columns).

3.4.2.13 If a sample or sample extract has been diluted for analysis, enter the DF value to one decimal place in the "Dilution Factor" field (i.e., a DF of 1 will be reported as 1.0; DF of 10 will be reported as 10.0).

3.4.2.14 If sulfur cleanup is employed, enter "Y" in the "Sulfur Cleanup" field; if not, enter "N" on Form I PEST and ARO.

3.4.2.15 For positively identified target compounds, the Contractor shall report the concentrations as **uncorrected** for blank contaminants.

3.4.2.16 Report all analytical results to two significant figures (i.e., if the value is 9.7, report 9.7; if the value is 10.3, report 10). For pesticide and Aroclor results, report the sample concentration (µg/L, µg/kg) of the lower of the two analyses.

3.4.2.17 Enter the appropriate concentration units, µg/L, µg/kg.

3.4.2.18 Under the column labeled "Q" for qualifier, flag each result with the specific data reporting qualifiers listed below. When reporting results to USEPA, the Contractor shall use these contract-specific qualifiers. The Contractor shall not modify the qualifiers. Up to five qualifiers may be reported on Form I for each compound. The Contractor is encouraged to use additional flags or footnotes (see the X qualifier).

The USEPA-defined qualifiers to be used are:

U: This flag indicates the compound was analyzed for but not detected. The Contract Required Quantitation Limit (CRQL) shall be adjusted according to the equation listed in Exhibit D. CRQLs are listed in Exhibit C.

J: This flag indicates an estimated value. This flag is used when: (1) estimating a concentration for Tentatively Identified Compounds (TICs) where a 1:1 response is assumed; (2) the mass spectral and Retention Time (RT) data indicate the presence of a compound that meets the volatile and semivolatile GC/MS identification criteria, and the result is less than the adjusted CRQL but greater than zero; and (3) the RT data indicate the presence of a compound that meets the pesticide and/or Aroclor identification criteria, and the result is less than the adjusted CRQL but greater than zero. For example, if the sample's adjusted CRQL is 5.0 µg/L, but a concentration of 3.0 µg/L is calculated, report it as 3.0J.

NOTE: The "J" flag is not used, and the compound is not reported as being identified for pesticide or Aroclor results less than the adjusted CRQL, if the pesticide residue analysis expert determines that the peaks used for compound identification resulted from instrument noise or other interferences (e.g., column bleed, solvent contamination).

N: This flag indicates presumptive evidence of a compound. This flag is only used for TICs, where the identification is based on a mass spectral library search and must be used in combination with the J flag. It is applied to all TIC results. For generic characterization of a TIC, such as chlorinated hydrocarbon, or for an "unknown" (no matches $\geq$ 85%), the "N" flag is not used.

P: This flag is used for pesticide and Aroclor target compounds when there is greater than 25% difference for detected concentrations between the two GC columns (see Form X). **The lower of the two values is reported on Form I and flagged with a "P".** The "P" flag is not used unless a compound is identified on both columns.

C: This flag applies to pesticide and Aroclor results when the **identification** has been confirmed by GC/MS. If GC/MS confirmation was attempted but was unsuccessful, do **not** apply this flag; use a laboratory-defined flag instead (see the X qualifier).

B: This flag is used when the analyte is found in the associated method blank as well as in the sample. It indicates probable blank contamination and warns the data user to take appropriate action. This flag shall be used for a TIC as well as for a positively identified target compound.

The combination of flags "BU" or "UB" is expressly prohibited. Blank contaminants are flagged "B" only when they are detected in the sample.

E: This flag identifies compounds whose response exceed the response of the highest standard in the initial calibration range of the instrument for that specific analysis. If one or more compounds have a response greater than the response of the highest standard in the initial calibration, the sample or extract shall be diluted and reanalyzed according to the specifications in Exhibit D. Exceptions are also noted in Exhibit D. All such compounds with responses greater than the response of the highest standard in the initial calibration shall have the result flagged with an "E" on Form I for the original analysis. The results of both analyses shall be reported on separate copies of Form I. The Form I for the diluted sample shall have "DL" suffix appended to the Sample Number.

D: If a sample or extract is reanalyzed at a DF greater than 1 (e.g., when the response of an analyte exceeds the response of the highest standard in the initial calibration), the DL suffix is appended to the Sample Number on Form I for the more diluted sample, and **all** reported concentrations on that Form I are flagged with the "D" flag. This flag alerts data users that any discrepancies between the reported concentrations may be due to dilution of the sample or extract.

NOTE 1: The "D" flag is not applied to compounds which are not detected in the sample analysis (i.e., compounds reported with the adjusted CRQL and the "U" flag).

NOTE 2: Separate Form Is are required for reporting the original analysis (EPA Sample No. XXXXX) and the more diluted sample analysis (EPA Sample No. XXXXXDL). The results from both analyses <u>cannot be combined</u> on a single Form I.

A: This flag indicates that a TIC is a suspected Aldol-condensation product.

S: This flag is used to indicate an estimated value for Aroclor target compounds where a valid 5-point initial calibration was not performed prior to the analytes detection in a sample. If an "S" flag is used for a specific Aroclor, then a reanalysis of the sample is required after a valid 5-point calibration is performed for the detected Aroclor.

X: Other specific flags may be required to properly define the results. If used, the flags shall be fully described in the SDG Narrative. Begin by using "X". If more than one flag is required, use "Y" and "Z" as needed. If more than five qualifiers are required for a sample result, use the "X" flag to represent a combination of several flags. For instance, the "X" flag might combine the "A", "B", and "D" flags for some samples. The laboratory-defined flags **are limited to** "X", "Y", and "Z".

3.5 Organics Analysis Data Sheet: Tentatively Identified Compounds (Form I VOA-TIC and Form I SV-TIC)

3.5.1 Purpose

This form is used to report analysis results for non-target compounds (e.g., compounds not listed in Exhibit C), excluding Deuterated Monitoring Compounds (DMCs) and internal standards. See Exhibit D for instructions on identification and quantitation. The Contractor shall submit Form I VOA-TIC or SV-TIC for **every analysis**, including required dilutions, reanalyses, and blanks, even if no TICs are found. Form I VOA-TIC and/or SV-TIC are not required for requested MS/MSD analysis.

3.5.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions in addition to the instructions in Section 3.4.

3.5.2.1 Report all TICs including Chemical Abstracts Service (CAS) Number (if applicable), compound name, RT, and the estimated concentration as uncorrected for blank contaminants. TICs shall be reported in chronological order for blank contaminants. TICs shall be reported in chronological order with respect to RTs. Report to two significant figures (criteria for reporting TICs are given in Exhibit D, Section 11). RT shall be reported in minutes and decimal minutes, **not** seconds or minutes:seconds.

3.5.2.2 Peaks that are suspected to be straight-chained, branched, or cyclic alkanes, and are alone or part of an alkane series, shall be library searched. Documentation for the tentative identification must be supplied. Alkane concentrations will be summed and reported as "total alkanes" on Form I VOA-TIC or SV-TIC.

3.5.2.3 If the name of a compound exceeds the 28 spaces in the TIC column, truncate the name to 28 characters. If the compound is an unknown, restrict the description to no more than 28 characters (e.g., unknown hydrocarbon).

3.5.2.4 Peaks that are suspected to be Aldol-condensation reaction products (e.g., 4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) shall be summarized on this form and flagged with an "A". The peaks shall be counted as part of the 30 most intense non-target semivolatile compounds to be searched.

3.6 DMC Recovery (Form II VOA-1, VOA-2, VOA-3, VOA-4, VOA-SIM1, VOA-SIM2, and Form II SV-1, SV-2, SV-3, SV-4, SV-SIM1, SV-SIM2)

3.6.1 Purpose

For volatiles and semivolatiles, Form II VOA-1, VOA-2, VOA-3, VOA-4, VOA-SIM1, VOA-SIM2, and Form II SV-1, SV-2, SV-3, SV-4, SV-SIM1, and SV-SIM2 are used to report the recoveries of the DMCs added to each volatile and semivolatile sample, including dilutions, reanalyses, blanks, and requested MS/MSDs. The DMCs are used to monitor the performance of the purge-and-trap GC/MS system as a whole, as well as the efficiency of the extraction procedure for semivolatiles. Form II VOA and Form II SV are matrix-specific, so that DMC recoveries for water samples are reported on a different version of Form II than the recoveries for soil samples. Soil sample recoveries are further differentiated by concentration level. Form II SV-SIM1 and Form II SV-SIM2 are used to report recoveries of the SIM DMCs only. For SIM analysis by the volatiles method, recoveries for the SIM DMC compounds need to be reported on Form II VOA-SIM1, VOA-SIM2.

3.6.2 Instructions

Complete the header information according to the instructions in Section 3.3.

NOTE: For volatiles and semivolatiles soil samples only, complete one form for each level. **Do not** mix low-level and medium-level samples on one form, and specify the level as LOW or MED. Complete the remainder of the forms using the following instructions.

3.6.2.1 For each of the volatile DMCs listed in Table 3, each of the semivolatile DMCs listed in Table 4, and each of the semivolatile SIM DMCs listed in Table 5, report the Percent Recovery to the nearest whole percentage point, and to the number of significant figures given by the Quality Control (QC) limits at the bottom of the form.

3.6.2.2 Flag each DMC recovery outside the QC limits with an asterisk ("*"). The asterisk shall be placed in the last space in each appropriate column, under the "#" symbol.

3.6.2.3 In the "TOT OUT" column, total the number of DMC recoveries that were outside the QC limits for each sample. If no DMCs were outside the limits, enter "0" (zero).

3.6.2.4 For semivolatiles, if the sample is diluted and the DMC recoveries are outside the acceptance window, enter the calculated recovery and flag the recovery with a "D" in the column underneath the "#" symbol.

3.6.2.5 Number all pages as described in Section 3.3.

TABLE 3

Volatile Deuterated Monitoring Compounds

Volatile Deuterated Monitoring Compounds		CAS Number
VDMC1	Vinyl chloride-d_3	6745-35-3
VDMC2	Chloroethane-d_5	19199-91-8
VDMC3	1,1-Dichloroethene-d_2	22280-73-5
VDMC4	2-Butanone-d_5	24313-50-6
VDMC5	Chloroform-d	865-49-6
VDMC6	1,2-Dichloroethane-d_4	17060-07-0
VDMC8	1,2-Dichloropropane-d_6	93952-08-0
VDMC9	Toluene-d_8	2037-26-5
VDMC10	trans-1,3-Dichloropropene-d_4	93951-86-1
VDMC11	2-Hexanone-d_5	4840-82-8
VDMC12	1,4-Dioxane-d_8	17647-74-4
VDMC13	1,1,2,2-Tetrachloroethane-d_2	33685-54-0
VDMC14	1,2-Dichlorobenzene-d_4	2199-69-1

TABLE 4

Semivolatile Deuterated Monitoring Compounds

Semivolatile Deuterated Monitoring Compounds		CAS Number
SDMC1	Phenol-d_5	4165-62-2
SDMC2	Bis(2-chloroethyl)ether-d_8	93952-02-4
SDMC3	2-Chlorophenol-d_4	93951-73-6
SDMC4	4-Methylphenol-d_8	190780-66-6
SDMC5	Nitrobenzene-d_5	4165-60-0
SDMC6	2-Nitrophenol-d_4	93951-78-1
SDMC7	2,4-Dichlorophenol-d_3	93951-74-7
SDMC8	4-Chloroaniline-d_4	191656-33-4
SDMC9	Dimethylphthalate-d_6	85448-30-2
SDMC10	Acenaphthylene-d_8	93951-97-4
SDMC11	4-Nitrophenol-d_4	93951-79-2
SDMC12	Fluorene-d_{10}	81103-79-9
SDMC13	4,6-Dinitro-methylphenol-d_2	93951-76-9
SDMC14	Anthracene-d_{10}	1719-06-8
SDMC15	Pyrene-d_{10}	1718-52-1
SDMC16	Benzo(a)pyrene-d_{12}	63466-71-7

TABLE 5

Semivolatile SIM Deuterated Monitoring Compounds

Semivolatile Selected Ion Monitoring (SIM) Deuterated Monitoring Compounds		CAS Number
SDMC17	Fluoranthene-d_{10}	93951-69-0
SDMC18	2-Methylnapthalene-d_{10}	7297-45-2

3.7 Surrogate Recovery (Form II PEST-1, PEST-2 and Form II ARO-1, ARO-2)

3.7.1 Purpose

Form II PEST-1, PEST-2 and Form II ARO-1, ARO-2 are used to report the recoveries of the surrogate compounds added to each pesticide and Aroclor sample, blank, LCS, and requested MS/MSD. Form II PEST and Form II ARO are matrix-specific, so surrogate recoveries for water samples are reported on a different version of Form II than surrogate recoveries for soil samples.

3.7.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.7.2.1 For each surrogate listed in Table 6, report the Percent Recovery to the nearest whole percentage point, and to the number of significant figures given by the QC limits at the bottom of the form.

3.7.2.2 Flag each surrogate recovery outside the QC limits with an asterisk ("*"). The asterisk shall be placed in the last space in each appropriate column, underneath the "#" symbol.

3.7.2.3 In the "TOT OUT" column, total the number of surrogate recoveries that were outside the QC limits for each sample. If no surrogates were outside the limits, enter "0" (zero).

3.7.2.4 If the sample is diluted and the surrogates are outside the acceptance window in any analysis, enter the calculated recovery, and flag the surrogate recoveries with a "D" in the column underneath the "#" symbol.

3.7.2.5 The pesticide and Aroclor surrogate recoveries shall be reported from **both** GC columns used for the analyses. Therefore, identify each GC column at the top of Form II PEST-1, PEST-2, and Form II ARO-1, ARO-2, entering the stationary phase in the "GC Column" field, and the internal diameter of the column in mm in the "ID" field.

3.7.2.6 The assignment of columns as "1" and "2" is left to the discretion of the Contractor when the analyses are performed by simultaneous injection into a GC containing two columns. If so analyzed, the assignment of "GC Column 1" and "GC Column 2" shall be consistent across all the reporting forms. If the analysis is **not** performed by simultaneous injection, then the assignment of GC column number shall be based on the chronological order of the two analyses.

3.7.2.7 Number all pages as described in Section 3.3.

TABLE 6

Pesticide and Aroclor Surrogates

Surrogate Compound	CAS Number
Decachlorobiphenyl (DCB)	2051-24-3
Tetrachloro-m-xylene (TCX)	877-09-8

3.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Laboratory Control Sample (LCS) Recovery

3.8.1 Matrix Spike/Matrix Spike Duplicate Recovery (All Fractions, Form III VOA-1, VOA-2; Form III SV-1, SV-2, Form III SV-SIM1, Form III SV-SIM2, Form III PEST-1, PEST-2; Form III ARO-1, ARO-2)

3.8.1.1 Purpose

This form is used to report the results of the analyses of MS/MSDs. The form is matrix-specific for volatiles, semivolatiles, pesticides, and Aroclors. For pesticides and Aroclors, complete Form III PEST-1, PEST-2 and Form III ARO-1, ARO-2 for each GC column used for analysis.

NOTE: Form III shall only be submitted for volatiles and semivolatiles if the analyses of MS/MSD samples have been requested by the Region. However, Form III is required for pesticides and Aroclors, unless otherwise specified by the Region.

3.8.1.2 Instructions

Complete the header information according to the instructions in Section 3.3. Include the EPA Sample Number for the Matrix Spike, **without** the suffixes MS or MSD.

3.8.1.2.1 For pesticides and Aroclors, enter the instrument ID, the stationary phase in the "GC Column" field, and the internal diameter of the column in millimeters (mm) in the "ID" field. The order of reporting is not important, but must be consistent with Form X.

3.8.1.2.2 For volatile water samples, specify level as TRACE or LOW on Form III VOA-1. For volatile and semivolatile soil samples, specify level as LOW or MED on Form III VOA-2, and SV-2. SDGs containing soil samples at both levels require an MS/MSD at each level; therefore, for soils, prepare one form for each level. Complete the remainder of the form using the following instructions.

3.8.1.2.3 In the first table under the "SPIKE ADDED" column, enter the calculated concentration in µg/L or µg/kg (according to the matrix) that results from dividing each spike compound amount added to the aliquot weight/volume chosen for the Matrix Spike. For instance, for base/neutral compounds in medium-level soils,

if 50 µg of spike are added to l g of soil, the results concentration is 50,000 µg/kg.

3.8.1.2.4 Enter the sample concentration in the next column, in similar units, of each spike compound detected in the original sample. If a spike compound was not detected during the analysis of the original sample, enter the sample result as "0" (zero).

3.8.1.2.5 In the "MS CONCENTRATION" column, enter the actual concentration of each spike compound detected in the Matrix Spike aliquot.

3.8.1.2.6 Calculate the Percent Recovery (%R) of each spike compound in the Matrix Spike aliquot to the nearest whole percent, according to Exhibit D. Enter the Percent Recovery in the "MS % REC" column.

3.8.1.2.7 Flag all Percent Recoveries outside the QC limits with an asterisk ("*"). The asterisk shall be placed in the last space of the "MS % REC" column, underneath the "#" symbol.

3.8.1.2.8 Follow Sections 3.8.1.2.3 through 3.8.1.2.7 to complete the lower table, using the results of the analysis of the Matrix Spike Duplicate aliquot.

3.8.1.2.9 Calculate the Relative Percent Difference (RPD) between the Matrix Spike recovery and the Matrix Spike Duplicate recovery, and enter this value in the "% RPD" column. Report the RPD to the nearest whole percent.

3.8.1.2.10 Compare the RPDs to the QC limits given on the form, and flag each RPD outside the QC limits with an asterisk ("*") in the last space of the "% RPD" column, underneath the "#" symbol.

3.8.1.2.11 Summarize the values outside the QC limits at the bottom of the page. No further action is required by the Contractor.

3.8.2 LCS Recovery (Form III PEST-3, PEST-4, and Form III ARO-3, ARO-4)

3.8.2.1 Purpose

This form is used to report the results of the analyses of LCSs for pesticides and Aroclors. The form is matrix-specific for pesticides and Aroclors.

3.8.2.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.8.2.2.1 If the LCS solution is purchased by the Contractor from a third party, report the identification number used by the third party to identify the LCS lot, if available, in the "LCS Lot No." field. If the LCS solution was prepared in-house, leave this entry blank.

3.8.2.2.2 The LCS is reported for each GC column. Enter the date analyzed, Instrument ID, GC column, and internal diameter for both GC columns. The order of reporting is not important, but

must be consistent with the information reported on Form X. All dates should be entered in MM/DD/YYYY format.

3.8.2.2.3 In the first table under the "AMOUNT ADDED" column, enter the calculated concentration in µg/L or µg/kg (according to the matrix) that results from dividing each spike compound amount added to the aliquot (weight/volume) of clean reference matrix. Under "AMOUNT RECOVERED", enter the actual concentration of each compound in the LCS calculated from analysis. Calculate the Percent Recovery of each compound in the LCS to the nearest whole percent, according to Exhibit D, and enter under "% REC". Flag all Percent Recoveries outside the QC limits with an asterisk ("*"). The asterisk must be placed in the last space of the Percent Recovery column, under the "#" symbol.

3.8.2.2.4 Complete the lower box according to the instructions in Section 3.8.2.2.3.

3.8.2.2.5 Summarize the recoveries outside the QC limits on both columns at the bottom of the page.

3.9 Method Blank Summary (Form IV, All Fractions)

3.9.1 Purpose

This form summarizes the samples associated with each method blank analysis. The Contractor shall submit the appropriate Form IV for each blank.

3.9.2 Instructions

Complete the header information according to the instructions in Section 3.3. The EPA Sample Number entered in the upper right-hand corner shall be the same number entered on Form I for the blank. Complete the remainder of the form using the following instructions.

3.9.2.1 Complete the following fields: "Instrument ID", "Date Analyzed", and "Time Analyzed". Dates shall be entered as MM/DD/YYYY. The time shall be reported using military time.

3.9.2.2 For pesticide and Aroclor method blanks, contaminants shall meet the identification criteria requiring analysis of the blank on two different GC columns (see Exhibits D - Analytical Methods for Pesticides and Analytical Methods for Aroclors). Enter the date, time, and instrument ID of both analyses of the blank on the method blank summary Form IV. The information for the two analyses is differentiated as Date Analyzed (1), Date Analyzed (2), etc. If the analyses were run simultaneously, the order of reporting is not important, but shall be consistent with the information reported on all other pesticide forms. Otherwise, Date Analyzed (1) shall indicate the analysis on Column 1, and Date Analyzed (2) shall indicate the analysis on Column 2.

3.9.2.3 For volatiles, pesticides, and Aroclors, identify the GC column and internal diameter in the appropriate fields.

3.9.2.4 For volatiles, indicate the purging method by entering "Y" for heated purge or "N" for ambient temperature purge in the "Heated Purge: Y/N" field on Form IV VOA.

3.9.2.5 For semivolatile, pesticide, and Aroclor blanks, enter the type of extraction as "CONH" for continuous liquid-liquid extraction with hydrophobic membrane, "CONT" for continuous liquid-liquid extraction without hydrophobic membrane, "SONC" for sonication, "SOXH" for Soxhlet extraction, or "PFEX" for pressurized fluid extraction on Form IV. For pesticide, and Aroclor blanks, separatory funnel extraction shall be entered as "SEPF".

3.9.2.6 For semivolatile, semivolatile-SIM, pesticide, and Aroclor method blanks, enter the date of extraction of the blank on Form IV SV, SV-SIM, PEST, or ARO (refer to Section 3.4.2.9 for more details).

3.9.2.7 Enter the reference matrix used to prepare the method blank in the "Matrix" field for all five fractions. For volatile and semivolatile soil method blanks, indicate the level as "LOW" or "MED" in the "Level" field.

3.9.2.8 If the samples associated with the pesticide and Aroclor blanks are subjected to sulfur cleanup, then the blanks shall also be subjected to sulfur cleanup. If sulfur cleanup is employed, enter "Y" in the "Sulfur Cleanup" field; if not, enter "N" on Form IV PEST, and ARO. If only some of the samples associated with the method blanks are subjected to sulfur cleanup, sulfur cleanup blanks are required in addition to the method blanks (see Exhibits D - Analytical Methods for Pesticides and Analytical Methods for Aroclors). If a sulfur cleanup blank is prepared in addition to the method blank, complete one version of Form IV associating all the samples with the method blank, and a second version of Form IV listing only those samples associated with the separate sulfur cleanup blank.

NOTE: Subjecting all samples associated with a method blank to sulfur cleanup avoids the need for two forms.

3.9.2.9 If semivolatile, semivolatile-SIM, pesticide, or Aroclor samples are subjected to GPC cleanup, then the associated blanks shall also be subjected to GPC cleanup. If the GPC Cleanup is employed, enter "Y" in the "GPC Cleanup" field; if not, enter "N" on Form IV SV, SV-SIM, PEST, and ARO.

3.9.2.10 For Aroclor blanks, enter "Y" in the "Acid Cleanup" field on Form IV ARO.

NOTE: Acid cleanup is required for all method blanks analyzed for Aroclors; therefore, all Form IV ARO will contain a "Y" in this field.

3.9.2.11 For all five fractions, as appropriate, summarize the samples including LCSs, requested MS/MSDs, storage blanks, and volatile instrument blanks, associated with a given method blank in the table, entering the EPA Sample Number and Laboratory Sample Identifier. For volatiles, enter the Laboratory File Identifier and the time of analysis of each sample. For semivolatiles, enter the Laboratory File Identifier and the date of analysis. For pesticides and Aroclors, enter the dates of both analyses as Date Analyzed (1) and Date Analyzed (2), as discussed previously.

3.9.2.12 Number all pages as described in Section 3.3.

3.10 GC/MS Instrument Performance Check and Mass Calibration (Form V VOA and Form V SV)

3.10.1 Purpose

This form is used to report the results of the GC/MS instrument performance check for the volatile and semivolatile fractions and to summarize the date and time of analyses of samples, including dilutions, reanalyses, standards, blanks, and requested MS/MSDs associated with each analysis of the Instrument Performance Check solution.

3.10.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.10.2.1 Enter the date and time of injection of the instrument performance check solution [4-Bromofluorobenzene (BFB) for volatiles--CAS Number 460-00-4, DFTPP for semivolatiles--CAS Number 5074-71-5). The date shall be entered as MM/DD/YYYY. The time shall be reported using military time.

3.10.2.2 For volatiles, identify the GC column and internal diameter on Form V VOA.

3.10.2.3 For each ion listed on the form, enter the percent relative abundance in the right-hand column of the first table. Report relative abundances to the number of significant figures given for each ion in the ion abundance criteria column.

NOTE: For both BFB and DFTPP, one or more of the high mass ions may exceed the abundance of the ion listed on the form as the nominal base peak [mass-to-charge ratio (m/z) 95 for BFB and m/z 198 for DFTPP]. Despite this possibility, all ion abundances shall be normalized to the nominal base peaks listed on Form V.

3.10.2.4 All relative abundances shall be reported as a number. If the relative abundance is zero, enter "0", not a dash or other non-numeric character. Where parentheses appear, compute the percentage of the ion abundance of the mass given in the appropriate footnote, and enter that value in the parentheses.

3.10.2.5 In the lower table, list all samples, including dilutions and reanalyses, standards, blanks, and MS/MSDs analyzed under that instrument performance check in chronological order, by time of analysis (using military time). Refer to Section 3.3.7 for specific instructions for identifying standards and blanks.

3.10.2.6 Complete the following fields for all standards, samples, including dilutions and reanalyses, blanks, and MS/MSDs: "EPA SAMPLE NO.", "LAB SAMPLE ID", "LAB FILE ID", "DATE ANALYZED", and "TIME ANALYZED".

3.10.2.7 All Form Vs listing samples, including dilutions and reanalyses, standards, blanks, and MS/MSDs must contain an opening and closing Continuing Calibration Verification (CCV). If samples are run after an initial calibration sequence, the initial calibration may be substituted for an opening CCV.

3.10.2.8 Number all pages as described in Section 3.3.

3.11 GC/MS Initial Calibration Data (Form VI VOA-1, VOA-2, VOA-3, VOA-SIM, and Form VI SV-1, SV-2, SV-3, SV-SIM)

3.11.1 Purpose

After a GC/MS system has undergone an initial five-point[3] calibration at the specific concentration levels described in Exhibit D, and after all initial calibration criteria have been met, the Contractor shall complete and submit these forms for each volatile or semivolatile target compound initial calibration performed that is relevant to the samples, including dilutions and reanalyses, blanks, and MS/MSDs in the SDG, regardless of when that calibration was performed. A calibration containing more than five points may be performed but only five points are to be reported on the Forms. The points that can be excluded are at the extreme concentration levels (below CRQL or above the required high concentration level). If analysis of trace volatiles using the SIM technique is requested, then all initial calibrations pertaining to these analytes shall be submitted on a separate Form VI-VOA. If the optional analysis of PAHs and pentachlorophenol using the SIM technique is requested, then all initial calibrations pertaining to these analytes shall be submitted on Form VI SV-SIM.

3.11.2 Instructions. Complete the header information according to the instructions in Section 3.3. Enter the Case Number and SDG Number for the current data package, regardless of the original Case for which the initial calibration was performed. Complete the remainder of the form using the following instructions.

3.11.2.1 Enter the date(s) of the calibration. If the calendar date changes during the calibration procedure, the inclusive dates shall be recorded. Dates shall be entered as MM/DD/YYYY.

3.11.2.2 Enter the injection times of the first and last of the standards analyzed in the "Calibration Times" field. Times shall be reported using military time.

3.11.2.3 For volatiles, complete the "GC Column" and "ID" fields. Indicate the purging method by entering "Y" for heated purge or "N" for ambient temperature purge in the "Heated Purge: (Y/N)" field.

3.11.2.4 For volatiles and semivolatiles, enter the concentration of each of the five standards after "RRF" in the space provided. Then enter the Laboratory File Identifier for the standards after the "=" in the space provided. For example, for the low standard, 5.0 µg/L, the Contractor shall enter 5.0 after the "RRF" in the section labeled LAB FILE ID, prior to entering the Laboratory File Identifier in the topmost row. Subsequently, 5.0 will be entered in the RRF entry in the second row, second column. If trace volatiles analysis of water samples at lower CRQLs are requested, then the Contractor shall enter 0.5 after "RRF" for the low

[3]For semivolatiles, seven compounds (2,4-Dinitrophenol, Pentacholorophenol, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, and 4,6-Dinitro-2-Methylphenol) will only require a four-point initial calibration at 10, 20, 40, and 80 total ng/µL concentrations because detection at less than 10 ng/µL per injection is difficult. If a four-point calibration is performed for these compounds, leave the "RRF5.0" column blank.

standard and 1.0 for the second level standard, etc., prior to entering the Laboratory File Identifier.

3.11.2.5 Complete the RRF data for the five calibration points, and then calculate and report the Mean Relative Response Factor ($\overline{RRF}$) for all target compounds and DMCs in the calibration standards.

3.11.2.6 The Contractor shall report the Percent Relative Standard Deviation (%RSD) for **all** compounds. See Exhibit D for equations.

3.12 GC/ECD Initial Calibration Data (Form VI PEST-1, PEST-2, PEST-3, PEST-4, ARO-1, ARO-2, and ARO-3)

3.12.1 Purpose

The initial calibration of pesticides and Aroclors involves the determination of RTs, RT time windows, and Calibration Factors (CFs). For single component pesticide target compounds, these data are calculated from the analyses of the Individual Standard Mixtures A and B or C at five different concentration levels. For Toxaphene, these data are calculated from the analyses of Toxaphene standards at five different concentration levels.

3.12.2 Instructions

Complete one Form VI for **each** GC column used for the five analyses of Individual Standard Mixture C or from the five analyses of Individual Standard Mixture A and Individual Standard Mixture B or Individual Standard Mixture C during an initial calibration. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.12.2.1 In the "Level (x CS1)" field, enter the concentration of the five calibration standards as a multiplier of CS1 (Calibration Standard 1). Therefore, for CS1, enter "1.0". The CS5 standard shall be at least 16 times CS1, but may be higher if that value lies within the linear range of the instrument, as specified in Exhibit D. Therefore, enter the appropriate multiplier for the high-point standard concentration to one decimal place.

3.12.2.2 Identify the GC column and internal diameter (in mm) in the appropriate fields.

3.12.2.3 Enter the dates of analysis of the first and last of the standards on each form in the "Date(s) Analyzed" field. Dates shall be entered as MM/DD/YYYY.

3.12.2.4 For each standard analyzed, enter the RT of each applicable analyte in minutes and decimal minutes, under the appropriate concentration level in the "RT OF STANDARDS" column on Form VI PEST-1.

3.12.2.5 Calculate the Mean RT ($\overline{RT}$) of each analyte from the five Individual Standard Mixtures: A and B, or C, and report it in the "$\overline{RT}$" column on Form VI PEST-1.

3.12.2.6 Calculate the RT window for each analyte using the specifications in Exhibit D, and enter the lower limit of the window in the "RT WINDOW" column under "FROM" and the upper limit of the window under "TO" on Form VI PEST-1. If Individual Standard Mixture C is

used, the second set of entries for the surrogates should be left blank.

3.12.2.7 For the analyses of the Individual Standard Mixtures: A, B, or C, the Contractor shall also complete the CF data on Form VI PEST-2. Prepare one form for each instrument and GC column used. Enter the CF for each compound in each of the standards. Calculate and enter a %RSD. If Individual Standard Mixture C is used, the second set of entries for the surrogates should be left blank.

3.12.2.8 For Toxaphene, the RTs, RT windows, and $\overline{RT}$ for each peak shall be reported on Form VI PEST-3 for the five-point calibration standards. The Contractor shall select at least three peaks for Toxaphene, according to the specifications in Exhibit D. The RT and CF data apply to **each** peak. Complete the upper table for GC Column (1) and the lower table for GC Column (2). The Contractor shall complete Form VI PEST-3 for each initial calibration that applies to samples in the data package.

3.12.2.9 For Toxaphene, the Contractor shall complete the CF data on Form VI PEST-4. Calculate and enter a %RSD.

3.12.2.10 Form VI ARO-1, ARO-2, and ARO-3 are used to report the initial calibration data for Aroclors. Form VI ARO-1 and ARO-2 are used to report RTs, RT windows, CFs, and %RSD from a five-point initial calibration of Aroclors 1016 and 1260. Form VI ARO-3 is used to report RTs, RT windows and CFs from the single-point initial calibration of the remaining target Aroclor compounds. If an Aroclor other than 1016 or 1260 is detected in a sample then a separate Form VI ARO-1 and ARO-2 must be submitted for the required initial calibration.

3.12.2.11 Complete one version of Form VI ARO-1, ARO-2, and ARO-3 for each GC column used to analyze Aroclor samples and each initial calibration that applies to samples in the data package. Complete the header information according to the instructions in Section 3.3. If more than 5 peaks for a particular Aroclor are to be reported, complete as many Form VI ARO-1, ARO-2, or ARO-3, as necessary, duplicating the header information and page numbering as described in Section 3.3. Complete the remainder of the form using the following instructions.

3.12.2.12 Identify the GC column and internal diameter (in mm) in the appropriate fields.

3.12.2.13 Enter the dates of the analysis of the Aroclor standards in the "Date(s) Analyzed" field. Dates shall be entered as MM/DD/YYYY.

3.12.2.14 For each of the five standards analyzed for Aroclor 1016 and 1260, and any other Aroclor if detected enter the RT for each Aroclor peak and surrogates in minutes and decimal minutes, under the appropriate concentration level (CS1, CS2, CS3, CS4, or CS5) in the "RT OF STANDARDS" column on Form VI ARO-1.

3.12.2.15 Calculate the $\overline{RT}$ for each peak (including surrogates) from the five calibration standards and report in the "MEAN RT" column on Form VI ARO-1.

3.12.2.16 Calculate the RT window for each peak (including surrogates) using the specifications in Exhibit D - Analytical Methods for Aroclors, and enter the lower limit of the window in the "RT WINDOW" column

under "FROM" and the upper limit of the window under "TO" on Form VI ARO-1. If Aroclors 1016 and 1260 are run as a combined mixture, the second set of surrogate entries should be left blank.

3.12.2.17 For the five analyses of Aroclor 1016 and 1260, and any other Aroclor if detected, the Contractor shall also complete the CF data for Form VI ARO-2. Prepare one form for each instrument and GC column used. Enter the CF for each peak (including surrogates) in each Aroclor standard. Calculate and enter a %RSD. If Aroclors 1016 and 1260 are run as a combined mixture, the second set of surrogate entries should be left blank.

3.12.2.18 For the remaining Aroclors, the RTs, RT windows, and CFs shall be reported in a similar fashion on Form VI ARO-3, for the single point calibration standards. The Contractor shall select at least three peaks for each Aroclor, according to the specifications in Exhibit D - Analytical Methods for Aroclors. The RT and CF data apply to each peak.

3.12.3 Form VI is also used to report the results of analysis of the Resolution Check Standard that shall begin each pesticide initial calibration sequence (Form VI PEST-5). The Contractor shall submit one Form VI PEST-5 for **both** GC columns.

3.12.4 Complete the header information as described in Section 3.3. Using the same assignment of first and second GC columns made for Form IV, enter the GC column identifier, internal diameter, date, and time of analysis(es). Enter the EPA Sample Number for the Resolution Check Standard. If simultaneous injections on a single GC column are used, the EPA Sample Number may be the same for both Resolution Check Standards. If simultaneous injections are **not** used, use different suffixes to identify the standards. Complete the remainder of the form using the following instructions.

3.12.4.1 List each analyte, in **RT order**, including both surrogate compounds. Thus, the order of analytes in the two boxes on this form will be different due to the dissimilarity of the stationary phases of the two GC columns used. Enter the name of each target analyte in the Resolution Check Mixture as it appears on Form I PEST.

3.12.4.2 Enter the RT of each analyte from the analysis in the "RT" column.

3.12.4.3 Calculate the resolution between each pair of analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered.

NOTE: The last resolution field will not be filled.

3.12.4.4 Form VI [PEST-6, PEST-7, PEST-8, PEST-9, and PEST-10 for each pair of Performance Evaluation Mixtures (PEMs), either CS3 Individual Standard Mixture C, or CS3 Individual Standard Mixtures A and B, respectively] shall be used to report the Percent Resolution between each pair of analytes according to the definition in Exhibit D (Analytical Methods for Pesticides), Section 9.2.4.10.

3.12.4.5 Complete the header information as described in Section 3.3. Using the same assignment of first and second GC columns made for

Form IV, enter the GC column identifier, internal diameter, date, and time of analysis. Enter the EPA Sample Number for the respective standards. If simultaneous injections are **not** used, use different suffixes to identify the standards. Complete the remainder of the form using the following instructions.

3.12.4.5.1 List each analyte, in **RT order**, including both surrogate compounds. Thus, the order of analytes in the two boxes on this form will be different due to the dissimilarity of the stationary phases of the two GC columns used. Enter the name of each target analyte in the standard as it appears on Form I PEST. Spell out the names of the surrogates as they appear on Form VII PEST-2.

3.12.4.5.2 Enter the RT of each analyte from the analysis in the "RT" column.

3.12.4.5.3 Calculate the resolution between each pair of analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered.

NOTE: The last resolution field will be left blank in each table.

3.13 GC/MS Opening and Closing Continuing Calibration Verification Data (Form VII VOA-1, VOA-2, VOA-3, VOA-SIM, and Form VII SV-1, SV-2, SV-3, SV-SIM)

3.13.1 Purpose

For volatiles and semivolatiles, this form is used to report the calibration verification of the GC/MS system by the analysis of specific calibration verification standards. Form VII is required for opening and closing CCVs for each 12-hour time period for both volatile and semivolatile target compound analyses. If analysis of trace volatiles using the SIM technique is requested, then an additional Form VII VOA shall be submitted for opening and closing CCVs for each 12-hour time period that samples are analyzed. If the optional analysis of PAHs and pentachlorophenol using the SIM technique is requested, then Form VII SV-SIM shall be submitted for opening and closing CCVs for each 12-hour time period that samples are analyzed. The Contractor shall analyze calibration verification standards and meet all criteria outlined in Exhibit D for the minimum RRF and maximum Percent Difference (%D) between initial calibration CCVs.

3.13.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.13.2.1 Enter the date (Calibration Date:) and time (Time:) of the CCV and the date(s) (Init. Calib. Dates:) and time(s) (Init. Calib. Times:) of the initial calibration (give inclusive dates if the initial calibration is performed over more than one date). Dates shall be entered as MM/DD/YYYY. Times shall be reported using military time.

3.13.2.2 For volatiles, enter "Y" if heated purge is performed or "N" if heated purge is not performed. Enter GC column identifier, internal diameter, and column length. For semivolatiles, enter the GC column identifier and internal diameter. Also enter the EPA Sample Number for the CCV standard on Form VII for volatiles and semivolatiles.

3.13.2.3 Using the appropriate initial calibration (volatile or semivolatile), enter the Mean Relative Response Factor ($\overline{RRF}$) for each target compound and DMC.

3.13.2.4 For volatiles and semivolatiles, report the concentration of the CCV standard in the space provided, after "RRF". The Contractor shall enter "50" in the space provided after "RRF" when the standard low/medium volatiles analysis for water and soil are performed and enter "20" when the standard semivolatiles analysis for water and soil are performed. If the trace volatiles analysis of water samples at lower CRQLs is requested, then the Contractor shall enter "5.0" in the space provided after "RRF". If the semivolatile analysis by the SIM method is requested enter "0.4" in the space provided after "RRF".

3.13.2.5 Report the RRF for each target and DMC from the CCV standard analysis for volatiles and semivolatiles.

3.13.2.6 Under "MIN RRF" enter the appropriate value. For an opening CCV or a closing CCV that is also used as an opening CCV for the next "12-hour period", the appropriate values can be found in Exhibit D (Table 2 in Trace Volatiles, Table 4 in Low/Medium Volatiles, and Table 4 in Semivolatiles). For a closing CCV enter "0.010" for all compounds. For a CCV that is both an opening and closing CCV, enter the values for an opening CCV.

3.13.2.7 Calculate the Percent Difference (%D) for all compounds. See Exhibits D - Analytical Methods for Volatiles and Analytical Methods for Semivolatiles for equations.

3.13.2.8 Under MAX %D enter the appropriate value. For an opening CCV and a closing CCV that is also an opening CCV for the next 12-hour period, the appropriate values can be found in Exhibit D Trace Volatiles (Tables 1 and 2), Low/Medium Volatiles (Table 4) and Semivolatiles (Table 4). For a closing CCV enter "50" for all target compounds.

3.14 GC/ECD Calibration Verification Summary (Form VII PEST-1, PEST-2, PEST-3, PEST-4, and Form VII ARO)

3.14.1 Purpose

Form VII is used to report the results of the PEMs and the CS3 concentrations of Individual Standard Mixtures C or A and B that, along with the PEM, bracket each 12-hour period of pesticides sample analyses. Form VII is also used to report the results of the mid-level Aroclor 1016/1260 standards that are used as calibration verification for Aroclors sample analyses. The Contractor shall submit Form VII PEST-1 and Form VII ARO for each 12-hour sequence analyzed. Form VII PEST-2 or Form VII PEST-3 shall be completed each time the Individual Standard Mixtures are analyzed, for each GC column used. FORM VII-PEST-4 shall be completed each time the CS3 Toxaphene standard is analyzed as part of the 72-hour confirmation requirement.

3.14.2 Instructions

Complete Form VII PEST-1, PEST-2, PEST-3, PEST-4, and Form VII ARO for each standard reported on Form VIII PEST and FORM VIII ARO. Complete the header information according to the instructions in Section 3.3. If more than 5 peaks for a particular Aroclor are to be reported, complete as many Form VII ARO as necessary, duplicating the header information and page numbering as described in Section 3.3. Complete the remainder of the forms using the following instructions.

3.14.2.1 Enter the date(s) of the initial calibration(s). Give inclusive dates if the initial calibration is performed over more than one day. Dates shall be entered as MM/DD/YYYY.

3.14.2.2 Identify the GC column and internal diameter in the appropriate fields.

3.14.2.3 On Form VII PEST-1, enter the EPA Sample Number, Laboratory Sample Identifier, and date and time of analysis for the instrument blank that preceded the 12-hour sequence (PIBLK). For the PEM that initiated or terminated the 12-hour sequence (PEM), enter the EPA Sample Number, Laboratory Sample Identifier, and date and time of analysis. Dates shall be entered as MM/DD/YYYY. Time shall be entered using military time.

3.14.2.4 When reporting data for the PEM at the **beginning** of the initial calibration sequence, leave the "EPA Sample No.", "Lab Sample ID", "Date Analyzed", and "Time Analyzed" fields blank for the instrument blank (PIBLK), when no instrument blank is analyzed before the PEM. When reporting **all other** PEM analyses, the instrument blank fields shall be completed.

3.14.2.5 In the table, report the RT for each target analyte and surrogate in the PEM, as well as the RT windows.

3.14.2.6 For each target analyte and surrogate in the PEM, enter the amount of the analyte found in the PEM, in nanograms to three decimal places, in the "CALC AMOUNT" column.

3.14.2.7 Enter the nominal amount of each analyte in the PEM in the "NOM AMOUNT" column.

3.14.2.8 Calculate the Percent Difference between the calculated amount and nominal amount for each analyte according to Exhibits D - Analytical Methods for Pesticides and Analytical Methods for Aroclors. Report the values in the "%D" column. If the Percent Difference is greater than 999.9, report as 999.9. If the Percent Difference is less than -99.9, report as -99.9.

3.14.2.9 Calculate the Percent Breakdown (%Breakdown) for Endrin and 4,4'-DDT and the combined %Breakdown in the PEM according to Exhibit D. Enter the values for the %Breakdown of Endrin and 4,4'-DDT in their respective fields immediately under the table.

3.14.2.10 Form VII PEST-2 contains the RT and CF data for Individual Standard Mixtures A and B. FORM VII PEST-3 contains the RT and CF data for Individual Standard Mixture C. FORM VII PEST-4 contains the RT and CF data for the CS3 Toxaphene standard.

Enter the EPA Sample Number, Laboratory Sample Identifier, date, and time of analysis for the instrument blank that preceded the

12-hour sequence (PIBLK). For INDC3 or INDA3 and INDB3 that initiated or terminated a 12-hour sequence and for the CS3 Toxaphene standard that is part of the 72-hour confirmation, enter the EPA Sample Number, Laboratory Sample Identifier, and date and time of analysis in the appropriate fields.

3.14.2.11 Using the appropriate initial calibration, enter the Mean Calibration Factor ($\overline{CF}$) for each target analyte and surrogate in INDC3 on FORM VII PEST-3 or INDA3 and INDB3 on FORM VII PEST-2 and CS3 Toxaphene on FORM VII PEST-4.

3.14.2.12 Enter the CF for each target analyte and surrogate from the calibration verification standards. Calculate the Percent Difference between the calibration verification CF and the $\overline{CF}$ from the initial calibration for each target analyte according to Exhibit D - Analytical Methods for Pesticides. Report the values in the "%D" column. If the Percent Difference is greater than 999.9, report as 999.9. If the Percent Difference is less than -99.9, report as -99.9.

3.14.2.13 On Form VII ARO, enter the EPA Sample Number, Laboratory Sample Identifier, date, and time of analysis for each Aroclor standard (1016 and 1260) in the appropriate fields. If Aroclor 1016 and 1260 are analyzed as a mixture, enter the EPA Sample Number of the mixture in the first "EPA Sample No." field and leave the second field blank.

3.14.2.14 In the table, report the RT for each Aroclor peak and surrogate. If Aroclor 1016 and 1260 are not analyzed as a mixture, report the surrogate information from Aroclor 1016 only. The Contractor shall report the RT window for each Aroclor peak and surrogate as determined from the appropriate initial calibration.

3.14.2.15 Using the appropriate initial calibration, enter the $\overline{CF}$ for each Aroclor peak and surrogate.

3.14.2.16 Enter the CF for each Aroclor peak and surrogate from the calibration verification standard(s).

3.14.2.17 Calculate the Percent Difference for all Aroclor peaks and surrogates. See Exhibit D - Analytical Methods for Aroclors for the equation. If the Percent Difference is greater than 999.9, report as 999.9. If the Percent Difference is less than -99.9, report as -99.9.

3.15 Internal Standard Area and RT Summary (Form VIII VOA, VOA-SIM, and Form VIII SV-1, SV-2, SV-SIM1, SV-SIM2)

3.15.1 Purpose

This form is used to summarize the peak areas and RTs of the internal standards added to all volatile and semivolatile calibration standards and samples, including: dilutions, reanalyses, and blanks. The data are used to determine when changes in internal standard responses will adversely affect quantitation of target compounds. This form shall be completed each time a CCV is performed, or when samples are analyzed under the same GC/MS instrument performance check as an initial calibration.

3.15.2 Instructions

Complete the header information according to Section 3.3. Complete the remainder of the form using the following instructions. If samples are analyzed immediately following an initial calibration, before another instrument performance check and a CCV, Form VIII shall be completed on the basis of the internal standard areas of the 50 µg/L initial calibration standard for volatiles (or the 5 µg/L initial calibration standard if the trace volatiles analysis of water samples at lower CRQLs are requested), and the 20 ng/µL initial calibration standard for semivolatiles (or the 0.40 ng/µL initial calibration if the optional analysis of semivolatiles by the SIM method is requested). Use the date and time of analysis of this standard and the Laboratory File Identifier and areas in place of those of a CCV standard.

3.15.2.1 Enter the date and time of analysis of the continuing calibration standard. The date shall be entered as MM/DD/YYYY. The time shall be reported using military time.

3.15.2.2 For volatiles, enter "Y" of heated purge is performed or "N" of heated purge is not performed. Enter the GC column identifier, internal diameter, and column length. For semivolatiles, enter GC column identifier and internal diameter.

3.15.2.3 From the results of the analysis of the CCV standard, enter the area measured for each internal standard and its RT (in decimal minutes) under the appropriate column in the "12 HOUR STD" row.

3.15.2.4 For each internal standard listed in Tables 7 and 8, calculate the upper and lower limits of the area of the particular standard for Low/Medium Volatiles and Trace Volatiles accordingly. Report these values in the "UPPER LIMIT" and "LOWER LIMIT" rows, respectively. Calculate the upper limit of the RT as the retention of the internal standard, and the lower limit of the RT as the RT in the standard minus 0.50 minutes (30 seconds) for Low/Medium or 0.33 minutes (20 seconds) for Trace Volatiles, respectively.

3.15.2.5 For each sample, including dilutions, reanalyses, blanks, and requested MS/MSDs, analyzed under a given CCV, enter the EPA Sample Number and the area measured for each internal standard and its RT. If the internal standard area is outside the upper or lower limits calculated in Section 3.15.2.4, flag that area with an asterisk ("*"). The asterisk shall be placed in the far right-hand space of the box for each internal standard area, directly under the "#" symbol. Similarly, flag the RT of any internal standard that is outside the limits with an asterisk.

3.15.2.6 Number all pages as described in Section 3.3.

TABLE 7

Volatile Internal Standards

Volatile Internal Standards	CAS Number
IS1: Chlorobenzene-d_5 (CBZ)	3114-55-4
IS2: 1,4-Difluorobenzene (DFB)	540-36-3
IS3: 1,4-Dichlorobenzene-d_4(DCB)	3855-82-1

TABLE 8

Semivolatile Internal Standards

Semivolatile Internal Standards	CAS Number
IS1: 1,4-Dichlorobenzene-d_4 (DCB)	3855-82-1
IS2: Naphthalene-d_8 (NPT)	1146-65-2
IS3: Acenaphthene-d_{10} (ANT)	15067-26-2
IS4: Phenanthrene-d_{10} (PHN)	1517-22-2
IS5: Chrysene-d_{12} (CRY)	1719-03-5
IS6: Perylene-d_{12} (PRY)	1520-96-3

3.16 Pesticide and Analytical Sequence (Form VIII PEST and Form VIII ARO)

3.16.1 Purpose

This form is used to report the analytical sequence for pesticide and Aroclor analyses. At least one form is required for each GC column used for pesticide and Aroclor analyses.

3.16.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.16.2.1 Enter the date(s) of the initial calibration. Give inclusive dates if the initial calibration is performed over more than one day. Dates shall be entered as MM/DD/YYYY.

3.16.2.2 Identify the GC column and internal diameter in the appropriate fields.

3.16.2.3 At the top of the table, report the $\overline{RT}$ for tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) calculated from the initial calibration sequence.

3.16.2.4 For every analysis associated with a particular analytical sequence starting with the initial calibration, enter the EPA Sample Number, Laboratory File Identifier, and date and time of analysis. Each sample analyzed as part of the sequence shall be reported on Form VIII **even** if it is not associated with the SDG. The Contractor shall use ZZZZZ as the EPA Sample Number to distinguish all samples that are not part of the SDG being reported using military time.

3.16.2.5 Report the RT of the surrogates for each analysis in the "TCX RT" and "DCB RT" columns. For pesticides, all sample analyses shall be bracketed by acceptable analyses of instrument blanks, a PEM, and Individual Standard Mixtures A and B or C. Given the fact that the initial calibration for pesticides and Aroclors may remain valid for some time (see Exhibits D - Analytical Methods for Pesticides and Analytical Methods for Aroclors), it is only necessary to report the data from 12-hour periods when samples, dilutions, reanalyses, MS/MSDs, LCSs, or blanks in an SDG were analyzed. All data necessary to demonstrate compliance with the requirements specified in Exhibits D - Analytical Methods for

Pesticides and Analytical Methods for Aroclors must be reported. For pesticides, the Contractor shall submit Form VIII for the initial calibration sequence and forms that include the PEMs and Individual Standard Mixtures that bracket **any** and **all** samples in the SDG. While the data for time periods between the initial calibration and samples in the SDG are not a routine deliverable, the data shall be available as requested (e.g., at on-site evaluations). Non-EPA samples or samples from SDGs not being reported shall be numbered ZZZZZ.

3.16.2.6 Flag all those values which do not meet the contract requirements by entering an asterisk ("*") in the "TCX RT" and "DCB RT"column, under the "#" symbol. If the RT cannot be calculated due to interfering peaks, leave the "RT" column blank for that surrogate, enter an asterisk in the last column, and document the problem in the SDG Narrative.

3.16.2.7 If more than a single copy of Form VIII is required for pesticides or Aroclors, enter the same header information on all subsequent pages for that GC column and instrument, and number each page as described in Section 3.3.

3.17 Pesticide Cleanup Summary (Form IX PEST-1 and PEST-2)

3.17.1 Purpose

This form summarizes the results of the checks performed for both cleanup procedures employed during the preparation of pesticide extracts for analysis. Form IX PEST-1 is used to report the results of the check of the Florisil cartridges used to process all sample extracts, and to associate the lot of cartridges with particular sample results so that problems with a particular cartridge lot may be tracked across all associated samples. Form IX PEST-2 summarizes the results of the calibration verification of the GPC device that shall be used to process all sample extracts for pesticide analyses that require GPC cleanup (mandatory for all soil samples, optional for water samples).

3.17.2 Instructions

Complete the header information according to the instructions in Section 3.3. Enter the Case Number and SDG Number for the current data package, regardless of the original Case for which the cartridge check was performed. Complete the remainder of the form using the following instructions.

3.17.3 FORM IX PEST-1

3.17.3.1 Enter the Florisil cartridge Lot Number.

3.17.3.2 Enter the date the Florisil cartridge check solution was analyzed in the "Date of Analysis" field. The date shall be entered as MM/DD/YYYY.

3.17.3.3 Complete the "GC Column" and "ID" fields for the GC column used to analyze the samples, including blanks, MS/MSDs, and LCSs. Report all results from a single GC column.

3.17.3.4 In the first table, enter the amount of spike added and spike recovered in nanograms (ng) for each analyte.

3.17.3.5 Calculate the Percent Recovery to the nearest whole percent, and enter the number in the "% REC" field. Flag each spike recovery outside the QC limits (shown on the form) with an asterisk ("*"). The asterisk shall be placed in the last space in the "% REC" column, underneath the "#" symbol.

3.17.3.6 In the second table, complete the "EPA Sample No.", the "Lab Sample ID", and "Date Analyzed" fields for each sample and blank that were cleaned up using this lot of Florisil cartridges.

3.17.3.7 Number the pages as described in Section 3.3.

3.17.4 FORM IX PEST-2

3.17.4.1 On Form IX PEST-2, enter an identifier for the GPC column and the analysis date of calibration verification in the appropriate fields.

3.17.4.2 Complete the "GC Column" and "ID" fields as on Form IX PEST-1 for Florisil. Report all results from a single column.

3.17.4.3 For each of the pesticide Matrix Spike compounds listed in the first table, enter the amount of the spike added to the GPC column and the amount recovered, in nanograms (ng).

3.17.4.4 Calculate the Percent Recovery of each analyte, and enter these values on the form, to the nearest percent. Compare the recoveries to the QC limits shown on the form, and flag all those values outside the limits with an asterisk ("*") in the "% REC" column underneath the "#" symbol.

3.17.4.5 For each sample in the data package that was subjected to GPC cleanup under this calibration verification, enter the EPA Sample Number, Laboratory Sample Identifier, and the date the sample was subjected to GPC cleanup in the second table.

3.17.4.6 If more than one copy of Form IX PEST-2 is required, number all pages as described in Section 3.3.

3.18 Identification Summary of Single Component and Multicomponent Analytes (Form X PEST-1, PEST-2 and Form X ARO)

3.18.1 Purpose

This form summarizes the quantitations of all target pesticides and Aroclors detected in a given sample. It reports the RTs of the compound on both columns on which it was analyzed, as well as the RT windows of the standard for that compound on both of these columns. In addition, it is used to report the concentration determined from each GC column, and the Percent Difference between the two quantitative results. Separate forms are used for single component analytes and multicomponent analytes.

Form X is required for each sample, including dilutions and reanalyses, blanks, LCSs, and MS/MSDs in which compounds listed in Exhibit C - Pesticides and Aroclors are detected and reported on Form I. **Do not generate a Form X for pesticide instrument blanks.**

3.18.2 Instructions

Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

3.18.2.1 Enter the date(s) of analysis. Dates shall be entered as MM/DD/YYYY.

3.18.2.2 Enter the GC column and internal diameter for each of the two columns.

3.18.2.3 For each single component pesticide positively identified on both columns, enter the name of the compound in the "ANALYTE" column as it appears on Form I.

3.18.2.4 For Form X PEST-1, enter the RTs on each column of the compounds detected in the sample next to the appropriate column designation (1 or 2).

3.18.2.5 Enter the RT windows on each GC column from the initial calibration standards. These data shall correspond with those on Form VI and shall be entered in a similar manner. The lower value is entered under the "FROM" column, and the upper value under the "TO" column.

3.18.2.6 Enter the concentration calculated from each GC column under the "CONCENTRATION" column. Analyte concentrations must be rounded using the USEPA Rounding Rules to the required number of significant figures. Although the units are the same as those used on Form I, µg/L for water samples and µg/kg for soil samples, do **not** enter any units on Form X.

3.18.2.7 Calculate the Percent Difference between the concentrations entered on this form. See Exhibits D - Analytical Methods for Pesticides and Analytical Methods for Aroclors for equations, and report to a tenth of a percent in the "%D" column. If the Percent Difference is greater than 999.9, report it as 999.9.

3.18.2.8 The **lower** of the two concentrations is reported on Form I for each pesticide compound. The lower concentration is used because, if present, coeluting interferences are likely to increase the calculated concentration of any target compound. If the Percent Difference between the calculated concentrations is greater than 25.0%, flag the concentration on Form I, as described previously. This will alert the data user to the potential problems in quantitating this analyte.

3.18.2.9 If more pesticide compounds are identified in an individual sample than can be reported on one Form X, complete as many additional copies of Form X as necessary, duplicating all header information and numbering the pages as described in Section 3.3.

3.18.2.10 Report Toxaphene detected in samples on Form X PEST-2. Report Aroclors detected in samples on Form X ARO. Complete the header information and GC column fields as described above. For multicomponent analytes (Toxaphene and Aroclors), it is necessary to report the RT and concentration of each peak chosen for quantitation in the target analyte in a fashion similar to that for single component pesticides. The Aroclor peaks used for quantitation must be reported in its proper position (e.g., if peaks 1, 3, and 5 are used, then report the values of these peaks in the 1, 3, and 5 position on Form X). The concentrations of all

peaks quantitated (three are required, up to five may be used) are averaged to determine the mean concentration. The mean concentration must be rounded using the USEPA Rounding Rules to the required number of significant numbers. Report the lower of the two **mean** concentrations on Form I. Flag this value if the mean concentrations from the two GC columns differ by more than 25.0%, as described previously.

3.18.2.11 If more multicomponent compounds, or more than 5 peaks per multicomponent compound, are identified in an individual sample than can be reported on one Form X, complete as many additional copies of Form X as necessary, duplicating all header information and numbering the pages as described in Section 3.3.

3.19 Sample Log-In Sheet (Form DC-1)

3.19.1 Purpose

This form is used to document the receipt and inspection of sample containers and samples. One original Form DC-1 is required for each sample shipping container (only the hardcopy form is required). If the samples in a single sample shipping container are assigned to more than one SDG, the original Form DC-1 shall be placed with the deliverables for the SDG of the lowest alphanumeric number, and a copy of Form DC-1 shall be placed with the deliverables for the other SDGs. The copies shall be identified as "copy(ies)", and the location of the original shall be noted on the copies.

3.19.2 Instructions

3.19.2.1 Sign and date the airbill. If an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company’s electronic tracking information.

3.19.2.2 Complete the header information on the form, including the log-in date.

3.19.2.3 Examine the shipping container and record the presence/absence of custody seals and their condition (e.g., intact, broken) in Item 1.

3.19.2.4 Record the Custody Seal Numbers in Item 2.

3.19.2.5 Open the container, remove the enclosed sample documentation, and record the presence/absence of USEPA forms, SMO forms (i.e., TR/Chain of Custody Records, Packing Lists), and airbills or airbill stickers in Items 3 and 4. Specify if there is an airbill present or an airbill sticker in Item 4. Record the airbill or sticker number in Item 5.

3.19.2.6 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample bottles (e.g., intact, broken, leaking) and presence or absence of sample tags in Items 6 and 7.

3.19.2.7 Record the presence of the cooler temperature indicator bottle in Item 8 and the cooler temperature in Item 9.

3.19.2.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and circle the appropriate answer in Item 10.

3.19.2.9 The log-in date should be recorded at the top of Form DC-1; record the date and time of cooler receipt at the laboratory in Items 11 and 12.

3.19.2.10 If there are no problems observed during receipt, sign and date (include the time) Form DC-1 and the TR/COC, and record the Sample Numbers on Form DC-1 in the "EPA Sample #" column.

3.19.2.11 Record the appropriate Sample Tag Numbers and assigned laboratory numbers, if applicable.

3.19.2.12 Any comments should be made in the "Remarks" column.

3.19.2.13 Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the "Sample Transfer" block located in the bottom left corner of Form DC-1. Sign and date the "Sample Transfer" block.

3.19.2.14 Cross out unused columns and spaces.

3.19.2.15 If there are problems observed during receipt or an answer marked with an asterisk (e.g., "absent*") was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms and note, where appropriate, the resolution of the problem.

3.20 Organics Complete SDG File (CSF) Inventory Sheet (Form DC-2)

3.20.1 Purpose. Form DC-2 is used to record the inventory of documents in the original Sample Data Package sent to the USEPA Region.

3.20.2 Instructions

3.20.2.1 Organize all USEPA CSF documents as described in Section 2.6. Assemble the documents in the order specified on Form DC-2 and Section 2.6, and stamp each page with a consecutive number; however, do not number Form DC-2. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record, in the "Comments" section on Form DC-2, all intentional gaps in the page numbering sequence (e.g., "page numbers not used, XXXX - XXXX, YYYY - YYYY"). If there are no documents for a specific document type, enter "NA" in the empty space.

3.20.2.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly-defined category. The Contractor shall review Form DC-2 to determine if it is most appropriate to place them under categories 8, 9, 10, or 11. Category 11 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category on Form DC-2.

3.20.2.3 If it is necessary to insert new or inadvertently omitted documents, the Contractor shall identify the documents with unique accountable numbers and record the unique accountable numbers and the locations of the documents in the CSF (in the "Other Records" section on Form DC-2).

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

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1A - FORM I VOA-1
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER) _____ Lab Sample ID: _____

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

Level: (TRACE/LOW/MED)_____ Date Received: _____

% Moisture: not dec._____ Date Analyzed: _____

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

Soil Extract Volume: _____(uL) Soil Aliquot Volume: _____(uL)

Purge Volume: _____(mL)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg)_____	Q
75-71-8	Dichlorodifluoromethane		
74-87-3	Chloromethane		
75-01-4	Vinyl chloride		
74-83-9	Bromomethane		
75-00-3	Chloroethane		
75-69-4	Trichlorofluoromethane		
75-35-4	1,1-Dichloroethene		
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane		
67-64-1	Acetone		
75-15-0	Carbon disulfide		
79-20-9	Methyl acetate		
75-09-2	Methylene chloride		
156-60-5	trans-1,2-Dichloroethene		
1634-04-4	Methyl tert-butyl ether		
75-34-3	1,1-Dichloroethane		
156-59-2	cis-1,2-Dichloroethene		
78-93-3	2-Butanone		
74-97-5	Bromochloromethane		
67-66-3	Chloroform		
71-55-6	1,1,1-Trichloroethane		
110-82-7	Cyclohexane		
56-23-5	Carbon tetrachloride		
71-43-2	Benzene		
107-06-2	1,2-Dichloroethane		
123-91-1	1,4-Dioxane		

1B - FORM I VOA-2
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER) _____ Lab Sample ID: _____

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

Level: (TRACE/LOW/MED) _____ Date Received: _____

% Moisture: not dec._____ Date Analyzed: _____

GC Column:_____ ID:_____(mm) Dilution Factor: _____

Soil Extract Volume: _____(uL) Soil Aliquot Volume: _____(uL)

Purge Volume: _____(mL)

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg)_____	Q
79-01-6	Trichloroethene		
108-87-2	Methylcyclohexane		
78-87-5	1,2-Dichloropropane		
75-27-4	Bromodichloromethane		
10061-01-5	cis-1,3-Dichloropropene		
108-10-1	4-Methyl-2-pentanone		
108-88-3	Toluene		
10061-02-6	trans-1,3-Dichloropropene		
79-00-5	1,1,2-Trichloroethane		
127-18-4	Tetrachloroethene		
591-78-6	2-Hexanone		
124-48-1	Dibromochloromethane		
106-93-4	1,2-Dibromoethane		
108-90-7	Chlorobenzene		
100-41-4	Ethylbenzene		
95-47-6	o-Xylene		
179601-23-1	m,p-Xylene		
100-42-5	Styrene		
75-25-2	Bromoform		
98-82-8	Isopropylbenzene		
79-34-5	1,1,2,2-Tetrachloroethane		
541-73-1	1,3-Dichlorobenzene		
106-46-7	1,4-Dichlorobenzene		
95-50-1	1,2-Dichlorobenzene		
96-12-8	1,2-Dibromo-3-chloropropane		
120-82-1	1,2,4-Trichlorobenzene		
87-61-6	1,2,3-Trichlorobenzene		

1C - FORM I VOA-SIM
TRACE VOLATILE ORGANICS SIM ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab Sample ID: _____ Lab File ID: _____

Sample vol:(mL) _____ Date Received: _____

GC Column: _____ ID:_____(mm) Date Analyzed: _____

Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg)_____	Q
123-91-1	1,4-Dioxane		
106-93-4	1,2-Dibromoethane		
96-12-8	1,2-Dibromo-3-chloropropane		

1D - FORM I SV-1 EPA SAMPLE NO.

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER)_____ Lab Sample ID: _____

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

Level: (LOW/MED)_____ Extraction: (Type)_____

% Moisture: _____ Decanted: (Y/N)_____ Date Received: _____

Concentrated Extract Volume: _____(uL) Date Extracted: _____

Injection Volume: _____(uL) GPC Factor: _____ Date Analyzed: _____

GPC Cleanup: (Y/N) ____ pH: _____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg)_____	Q
100-52-7	Benzaldehyde		
108-95-2	Phenol		
111-44-4	Bis(2-chloroethyl)ether		
95-57-8	2-Chlorophenol		
95-48-7	2-Methylphenol		
108-60-1	2,2'-Oxybis(1-chloropropane)		
98-86-2	Acetophenone		
106-44-5	4-Methylphenol		
621-64-7	N-Nitroso-di-n-propylamine		
67-72-1	Hexachloroethane		
98-95-3	Nitrobenzene		
78-59-1	Isophorone		
88-75-5	2-Nitrophenol		
105-67-9	2,4-Dimethylphenol		
111-91-1	Bis(2-chloroethoxy)methane		
120-83-2	2,4-Dichlorophenol		
91-20-3	Naphthalene		
106-47-8	4-Chloroaniline		
87-68-3	Hexachlorobutadiene		
105-60-2	Caprolactam		
59-50-7	4-Chloro-3-methylphenol		
91-57-6	2-Methylnaphthalene		
77-47-4	Hexachlorocyclopentadiene		
88-06-2	2,4,6-Trichlorophenol		
95-95-4	2,4,5-Trichlorophenol		
92-52-4	1,1'-Biphenyl		
91-58-7	2-Chloronaphthalene		
88-74-4	2-Nitroaniline		
131-11-3	Dimethylphthalate		
606-20-2	2,6-Dinitrotoluene		
208-96-8	Acenaphthylene		
99-09-2	3-Nitroaniline		
83-32-9	Acenaphthene		

1E - FORM I SV-2
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER)_____ Lab Sample ID: _____

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

Level: (LOW/MED)_____ Extraction: (Type)_____

% Moisture: _____ Decanted: (Y/N)_____ Date Received: _____

Concentrated Extract Volume: _____(uL) Date Extracted: _____

Injection Volume: _____(uL) GPC Factor: _____ Date Analyzed: _____

GPC Cleanup: (Y/N) ____ pH: _____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg)_____	Q
51-28-5	2,4-Dinitrophenol		
100-02-7	4-Nitrophenol		
132-64-9	Dibenzofuran		
121-14-2	2,4-Dinitrotoluene		
84-66-2	Diethylphthalate		
86-73-7	Fluorene		
7005-72-3	4-Chlorophenyl-phenylether		
100-01-6	4-Nitroaniline		
534-52-1	4,6-Dinitro-2-methylphenol		
86-30-6	N-Nitrosodiphenylamine[1]		
95-94-3	1,2,4,5-Tetrachlorobenzene		
101-55-3	4-Bromophenyl-phenylether		
118-74-1	Hexachlorobenzene		
1912-24-9	Atrazine		
87-86-5	Pentachlorophenol		
85-01-8	Phenanthrene		
120-12-7	Anthracene		
86-74-8	Carbazole		
84-74-2	Di-n-butylphthalate		
206-44-0	Fluoranthene		
129-00-0	Pyrene		
85-68-7	Butylbenzylphthalate		
91-94-1	3,3'-Dichlorobenzidine		
56-55-3	Benzo(a)anthracene		
218-01-9	Chrysene		
117-81-7	Bis(2-ethylhexyl)phthalate		
117-84-0	Di-n-octylphthalate		
205-99-2	Benzo(b)fluoranthene		
207-08-9	Benzo(k)fluoranthene		
50-32-8	Benzo(a)pyrene		
193-39-5	Indeno(1,2,3-cd)pyrene		
53-70-3	Dibenzo(a,h)anthracene		
191-24-2	Benzo(g,h,i)perylene		
58-90-2	2,3,4,6-Tetrachlorophenol		

[1]Cannot be separated from Diphenylamine

1F - FORM I SV-SIM
SEMIVOLATILE SIM ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER)_____ Lab Sample ID: _____

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

Extraction: (Type)_____

% Moisture: _____ Decanted: (Y/N)_____ Date Received: _____

Concentrated Extract Volume: _____(uL) Date Extracted: _____

Injection Volume: _____(uL) GPC Factor: _____ Date Analyzed: _____

GPC Cleanup: (Y/N) ____ pH: _____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg)_____	Q
91-20-3	Naphthalene		
91-57-6	2-Methylnaphthalene		
208-96-8	Acenaphthylene		
83-32-9	Acenaphthene		
86-73-7	Fluorene		
87-86-5	Pentachlorophenol		
85-01-8	Phenanthrene		
120-12-7	Anthracene		
206-44-0	Fluoranthene		
129-00-0	Pyrene		
56-55-3	Benzo(a)anthracene		
218-01-9	Chrysene		
205-99-2	Benzo(b)fluoranthene		
207-08-9	Benzo(k)fluoranthene		
50-32-8	Benzo(a)pyrene		
193-39-5	Indeno(1,2,3-cd)pyrene		
53-70-3	Dibenzo(a,h)anthracene		
191-24-2	Benzo(g,h,i)perylene		

1G - FORM I PEST
PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER)_____ Lab Sample ID: _____

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

% Moisture: _____ Decanted: (Y/N)_____ Date Received: _____

Extraction: (Type)_____ Date Extracted: _____

Concentrated Extract Volume: _____(uL) Date Analyzed: _____

Injection Volume: _____(uL) GPC Factor: _____ Dilution Factor: _____

GPC Cleanup: (Y/N) ____ pH: _____ Sulfur Cleanup: (Y/N) _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg)_____	Q
319-84-6	alpha-BHC		
319-85-7	beta-BHC		
319-86-8	delta-BHC		
58-89-9	gamma-BHC (Lindane)		
76-44-8	Heptachlor		
309-00-2	Aldrin		
1024-57-3	Heptachlor epoxide		
959-98-8	Endosulfan I		
60-57-1	Dieldrin		
72-55-9	4,4'-DDE		
72-20-8	Endrin		
33213-65-9	Endosulfan II		
72-54-8	4,4'-DDD		
1031-07-8	Endosulfan sulfate		
50-29-3	4,4'-DDT		
72-43-5	Methoxychlor		
53494-70-5	Endrin ketone		
7421-93-4	Endrin aldehyde		
5103-71-9	alpha-Chlordane		
5103-74-2	gamma-Chlordane		
8001-35-2	Toxaphene		

1H - FORM I ARO
AROCLOR ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER)_____ Lab Sample ID: _____

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

% Moisture: _____ Decanted: (Y/N)_____ Date Received: _____

Extraction: (Type)_____ Date Extracted: _____

Concentrated Extract Volume: _____(uL) Date Analyzed: _____

Injection Volume: _____(uL) GPC Factor: _____ Dilution Factor: _____

GPC Cleanup: (Y/N) ____ pH: _____ Sulfur Cleanup: (Y/N) _____

Acid Cleanup: (Y/N) ____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/kg)_____	Q
12674-11-2	Aroclor-1016		
11104-28-2	Aroclor-1221		
11141-16-5	Aroclor-1232		
53469-21-9	Aroclor-1242		
12672-29-6	Aroclor-1248		
11097-69-1	Aroclor-1254		
11096-82-5	Aroclor-1260		
37324-23-5	Aroclor-1262		
11100-14-4	Aroclor-1268		

1J - FORM I VOA-TIC
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER)_____ Lab Sample ID: _____

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

Level: (TRACE or LOW/MED)_____ Date Received: _____

% Moisture: not dec._____ Date Analyzed: _____

GC Column: _____ ID:_____(mm) Dilution Factor: _____

Soil Extract Volume: _____(uL) Soil Aliquot Volume: _____(uL)

CONCENTRATION UNITS:(ug/L or ug/kg)____ Purge Volume: _____(mL)

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
	E966796[1]	Total Alkanes	N/A		

[1]EPA-designated Registry Number.

1K - FORM I SV-TIC
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix: (SOIL/SED/WATER)_____ Lab Sample ID: _____

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

Level: (TRACE or LOW/MED)_____ Extraction: (Type)_____

% Moisture: _____ Decanted: (Y/N)___ Date Received: _____

Concentrated Extract Volume: _____(uL) Date Extracted: _____

Injection Volume: _____(uL) GPC Factor: _____ Date Analyzed: _____

GPC Cleanup: (Y/N) ____ pH: _____ Dilution Factor: _____

CONCENTRATION UNITS:(ug/L or ug/kg)____

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
	E966796[2]	Total Alkanes	N/A		

[2]EPA-designated Registry Number.

2A - FORM II VOA-1
WATER VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Level: (TRACE or LOW)_____

	EPA SAMPLE NO.	VDMC1 (VCL) #	VDMC2 (CLA) #	VDMC3 (DCE) #	VDMC4 (BUT) #	VDMC5 (CLF) #	VDMC6 (DCA) #	VDMC7 (BEN) #
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

	QC LIMITS
VDMC1 (VCL) = Vinyl chloride-d_3	(65-131)
VDMC2 (CLA) = Chloroethane-d_5	(71-131)
VDMC3 (DCE) = 1,1-Dichloroethene-d_2	(55-104)
VDMC4 (BUT) = 2-Butanone-d_5	(49-155)
VDMC5 (CLF) = Chloroform-d	(78-121)
VDMC6 (DCA) = 1,2-Dichloroethane-d_4	(78-129)
VDMC7 (BEN) = Benzene-d_6	(77-124)

Column to be used to flag recovery values
* Values outside of contract required QC limits

2B - FORM II VOA-2
WATER VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Level: (TRACE or LOW)_____

	EPA SAMPLE NO.	VDMC8 (DPA) #	VDMC9 (TOL) #	VDMC10 (TDP) #	VDMC11 (HEX) #	VDMC12 (DXE) #	VDMC13 (TCA) #	VDMC14 (DCZ) #	TOT OUT
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

	QC LIMITS
VDMC8 (DPA) = 1,2-Dichloropropane-d_6	(79-124)
VDMC9 (TOL) = Toluene-d_8	(77-121)
VDMC10 (TDP) = trans-1,3-Dichloropropene-d_4	(73-121)
VDMC11 (HEX) = 2-Hexanone-d_5	(28-135)
VDMC12 (DXE) = 1,4-Dioxane-d_8	(50-150)
VDMC13 (TCA) = 1,1,2,2-Tetrachloroethane-d_2	(73-125)
VDMC14 (DCZ) = 1,2-Dichlorobenzene-d_4	(80-131)

Column to be used to flag recovery values
* Values outside of contract required QC limits

2C - FORM II VOA-3
SOIL VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Level: (LOW/MED)_____

	EPA SAMPLE NO.	VDMC1 (VCL) #	VDMC2 (CLA) #	VDMC3 (DCE) #	VDMC4 (BUT) #	VDMC5 (CLF) #	VDMC6 (DCA) #	VDMC7 (BEN) #
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

	QC LIMITS
VDMC1 (VCL) = Vinyl chloride-d_3	(68-122)
VDMC2 (CLA) = Chloroethane-d_5	(61-130)
VDMC3 (DCE) = 1,1-Dichloroethene-d_2	(45-132)
VDMC4 (BUT) = 2-Butanone-d_5	(20-182)
VDMC5 (CLF) = Chloroform-d	(72-123)
VDMC6 (DCA) = 1,2-Dichloroethane-d_4	(79-122)
VDMC7 (BEN) = Benzene-d_6	(80-121)

Column to be used to flag recovery values
* Values outside of contract required QC limits

2D - FORM II VOA-4
SOIL VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Level: (LOW/MED)_____

	EPA SAMPLE NO.	VDMC8 (DPA) #	VDMC9 (TOL) #	VDMC10 (TDP) #	VDMC11 (HEX) #	VDMC12 (DXE) #	VDMC13 (TCA) #	VDMC14 (DCZ) #	TOT OUT
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

	QC LIMITS
VDMC8 (DPA) = 1,2-Dichloropropane-d_6	(74-124)
VDMC9 (TOL) = Toluene-d_8	(78-121)
VDMC10 (TDP) = trans-1,3-Dichloropropene-d_4	(72-130)
VDMC11 (HEX) = 2-Hexanone-d_5	(17-184)
VDMC12 (DXE) = 1,4-Dioxane-d_8	(50-150)
VDMC13 (TCA) = 1,1,2,2-Tetrachloroethane-d_2	(56-161)
VDMC14 (DCZ) = 1,2-Dichlorobenzene-d_4	(70-131)

Column to be used to flag recovery values
* Values outside of contract required QC limits

2E - FORM II VOA-SIM1
TRACE SIM (WATER) VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

	EPA SAMPLE NO.	VDMC1 (VCL) #	VDMC2 (CLA) #	VDMC3 (DCE) #	VDMC4 (BUT) #	VDMC5 (CLF) #	VDMC6 (DCA) #	VDMC7 (BEN) #
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

	QC LIMITS
VDMC1 (VCL) = Vinyl chloride-d_3	(65-131)
VDMC2 (CLA) = Chloroethane-d_5	(71-131)
VDMC3 (DCE) = 1,1-Dichloroethene-d_2	(55-104)
VDMC4 (BUT) = 2-Butanone-d_5	(49-155)
VDMC5 (CLF) = Chloroform-d	(78-121)
VDMC6 (DCA) = 1,2-Dichloroethane-d_4	(78-129)
VDMC7 (BEN) = Benzene-d_6	(77-121)

Column to be used to flag recovery values
* Values outside of contract required QC limits

2F - FORM II VOA-SIM2
TRACE SIM (WATER) VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

	EPA SAMPLE NO.	VDMC8 (DPA) #	VDMC9 (TOL) #	VDMC10 (TDP) #	VDMC11 (HEX) #	VDMC12 (DXE) #	VDMC13 (TCA) #	VDMC14 (DCZ) #	TOT OUT
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

	QC LIMITS
VDMC8 (DPA) = 1,2-Dichloropropane-d_6	(79-124)
VDMC9 (TOL) = Toluene-d_8	(77-121)
VDMC10 (TDP) = trans-1,3-Dichloropropene-d_4	(73-121)
VDMC11 (HEX) = 2-Hexanone-d_5	(28-135)
VDMC12 (DXE) = 1,4-Dioxane-d_8	(50-150)
VDMC13 (TCA) = 1,1,2,2-Tetrachloroethane-d_2	(73-125)
VDMC14 (DCZ) = 1,2-Dichlorobenzene-d_4	(80-131)

Column to be used to flag recovery values
* Values outside of contract required QC limits

2G - FORM II SV-1
WATER SEMIVOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

	EPA SAMPLE NO.	SDMC1 (PHL) #	SDMC2 (BCE) #	SDMC3 (2CP) #	SDMC4 (4MP) #	SDMC5 (NBZ) #	SDMC6 (2NP) #	SDMC7 (DCP) #	SDMC8 (4CA) #
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

	QC LIMITS
SDMC1 (PHL) = Phenol-d_5	(39-106)
SDMC2 (BCE) = Bis(2-chloroethyl)ether-d_8	(40-105)
SDMC3 (2CP) = 2-Chlorophenol-d_4	(41-106)
SDMC4 (4MP) = 4-Methylphenol-d_8	(25-111)
SDMC5 (NBZ) = Nitrobenzene-d_5	(43-108)
SDMC6 (2NP) = 2-Nitrophenol-d_4	(40-108)
SDMC7 (DCP) = 2,4-Dichlorophenol-d_3	(37-105)
SDMC8 (4CA) = 4-Chloroaniline-d_4	(1-145)

\# Column to be used to flag recovery values
* Values outside of contract required QC limits
D DMC diluted out

2H - FORM II SV-2
WATER SEMIVOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

	EPA SAMPLE NO.	SDMC9 (DMP) #	SDMC10 (ACY) #	SDMC11 (4NP) #	SDMC12 (FLR) #	SDMC13 (NMP) #	SDMC14 (ANC) #	SDMC15 (PYR) #	SDMC16 (BAP) #	TOT OUT
01										
02										
03										
04										
05										
06										
07										
08										
09										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										
20										
21										
22										
23										
24										
25										
26										
27										
28										
29										
30										

	QC LIMITS
SDMC9 (DMP) = Dimethylphthalate-d_6	(47-114)
SDMC10 (ACY) = Acenaphthylene-d_8	(41-107)
SDMC11 (4NP) = 4-Nitrophenol-d_4	(33-116)
SDMC12 (FLR) = Fluorene-d_{10}	(42-111)
SDMC13 (NMP) = 4,6-Dinitro-2-methylphenol-d_2	(22-104)
SDMC14 (ANC) = Anthracene-d_{10}	(44-110)
SDMC15 (PYR) = Pyrene-d_{10}	(52-119)
SDMC16 (BAP) = Benzo(a)pyrene-d_{12}	(32-121)

\# Column to be used to flag recovery values
* Values outside of contract required QC limits
D DMC diluted out

2J - FORM II SV-3
SOIL SEMIVOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Level: (LOW/MED)_____

	EPA SAMPLE NO.	SDMC1 (PHL) #	SDMC2 (BCE) #	SDMC3 (2CP) #	SDMC4 (4MP) #	SDMC5 (NBZ) #	SDMC6 (2NP) #	SDMC7 (DCP) #	SDMC8 (4CA) #
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

	QC LIMITS
SDMC1 (PHL) = Phenol-d_5	(17-103)
SDMC2 (BCE) = Bis(2-chloroethyl)ether-d_8	(12-98)
SDMC3 (2CP) = 2-Chlorophenol-d_4	(13-101)
SDMC4 (4MP) = 4-Methylphenol-d_8	(8-100)
SDMC5 (NBZ) = Nitrobenzene-d_5	(16-103)
SDMC6 (2NP) = 2-Nitrophenol-d_4	(16-104)
SDMC7 (DCP) = 2,4-Dichlorophenol-d_3	(23-104)
SDMC8 (4CA) = 4-Chloroaniline-d_4	(1-145)

\# Column to be used to flag recovery values
* Values outside of contract required QC limits
D DMC diluted out

2K - FORM II SV-4
SOIL SEMIVOLATILE DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Level: (LOW/MED)_____

	EPA SAMPLE NO.	SDMC9 (DMP)#	SDMC10 (ACY)#	SDMC11 (4NP) #	SDMC12 (FLR)#	SDMC13 (NMP) #	SDMC14 (ANC) #	SDMC15 (PYR) #	SDMC16 (BAP) #	TOT OUT
01										
02										
03										
04										
05										
06										
07										
08										
09										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										
20										
21										
22										
23										
24										
25										
26										
27										
28										
29										
30										

	QC LIMITS
SDMC9 (DMP) = Dimethylphthalate-d_6	(43-111)
SDMC10 (ACY) = Acenaphthylene-d_8	(20-97)
SDMC11 (4NP) = 4-Nitrophenol-d_4	(16-166)
SDMC12 (FLR) = Fluorene-d_{10}	(40-108)
SDMC13 (NMP) = 4,6-Dinitro-2-methylphenol-d_2	(1-121)
SDMC14 (ANC) = Anthracene-d_{10}	(22-98)
SDMC15 (PYR) = Pyrene-d_{10}	(51-120)
SDMC16 (BAP) = Benzo(a)pyrene-d_{12}	(43-111)

\# Column to be used to flag recovery values
* Values outside of contract required QC limits
D DMC diluted out

2L - FORM II SV-SIM1
WATER SEMIVOLATILE SIM DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

	EPA SAMPLE NO.	SDMC17 (FLN) #	SDMC18 (2MN) #	TOT OUT
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

	QC LIMITS
SDMC17 (FLN) = Fluoranthene-d_{10}	(50-150)
SDMC18 (2MN) = 2-Methylnapthalene-d_{10}	(50-150)

\# Column to be used to flag recovery values
* Values outside of contract required QC limits
D DMC diluted out

2M - FORM II SV-SIM2
SOIL SEMIVOLATILE SIM DEUTERATED MONITORING COMPOUND RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

	EPA SAMPLE NO.	SDMC17 (FLN) #	SDMC18 (2MN) #	TOT OUT
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

	QC LIMITS
SDMC17 (FLN) = Fluoranthene-d_{10}	(50-150)
SDMC18 (2MN) = 2-Methylnapthalene-d_{10}	(50-150)

\# Column to be used to flag recovery values
* Values outside of contract required QC limits
D DMC diluted out

2N - FORM II PEST-1
WATER PESTICIDE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column(1): _____ ID: ____(mm) GC Column(2): _____ ID: ____(mm)

	EPA SAMPLE NO.	TCX 1 %REC #	TCX 2 %REC #	DCB 1 %REC #	DCB 2 %REC #	OTHER (1)	OTHER (2)	TOT OUT
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

	QC LIMITS
TCX = Tetrachloro-m-xylene	(30-150)
DCB = Decachlorobiphenyl	(30-150)

\# Column to be used to flag recovery values
* Values outside of QC limits
D Surrogate diluted out

2P - FORM II PEST-2
SOIL PESTICIDE SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column(1): _____ ID: ____(mm) GC Column(2): _____ ID: ____(mm)

	EPA SAMPLE NO.	TCX 1 %REC #	TCX 2 %REC #	DCB 1 %REC #	DCB 2 %REC #	OTHER (1)	OTHER (2)	TOT OUT
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

	QC LIMITS
TCX = Tetrachloro-m-xylene	(30-150)
DCB = Decachlorobiphenyl	(30-150)

\# Column to be used to flag recovery values
* Values outside of QC limits
D Surrogate diluted out

2Q - FORM II ARO-1
WATER AROCLOR SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column(1): _____ ID: ____(mm) GC Column(2): _____ ID: ____(mm)

	EPA SAMPLE NO.	TCX 1 %REC #	TCX 2 %REC #	DCB 1 %REC #	DCB 2 %REC #	OTHER (1)	OTHER (2)	TOT OUT
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

	QC LIMITS
TCX = Tetrachloro-m-xylene	(30-150)
DCB = Decachlorobiphenyl	(30-150)

\# Column to be used to flag recovery values
* Values outside of QC limits
D Surrogate diluted out

2R - FORM II ARO-2
SOIL AROCLOR SURROGATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column(1): _____ ID: ____(mm) GC Column(2): _____ ID: ____(mm)

	EPA SAMPLE NO.	TCX 1 %REC #	TCX 2 %REC #	DCB 1 %REC #	DCB 2 %REC #	OTHER (1)	OTHER (2)	TOT OUT
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

	QC LIMITS
TCX = Tetrachloro-m-xylene	(30-150)
DCB = Decachlorobiphenyl	(30-150)

\# Column to be used to flag recovery values
* Values outside of QC limits
D Surrogate diluted out

3A - FORM III VOA-1
WATER VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____ Level: (TRACE or LOW)_____

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS %REC #	QC LIMITS REC.
1,1-Dichloroethene					61-145
Trichloroethene					71-120
Benzene					76-127
Toluene					76-125
Chlorobenzene					75-130

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD %REC #	%RPD #	QC LIMITS	
					RPD	REC.
1,1-Dichloroethene					0-14	61-145
Trichloroethene					0-14	71-120
Benzene					0-11	76-127
Toluene					0-13	76-125
Chlorobenzene					0-13	75-130

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____
_____

3B - FORM III VOA-2
SOIL VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____ Level: (LOW/MED)_____

COMPOUND	SPIKE ADDED (ug/kg)	SAMPLE CONCENTRATION (ug/kg)	MS CONCENTRATION (ug/kg)	MS %REC #	QC LIMITS REC.
1,1-Dichloroethene					59-172
Trichloroethene					62-137
Benzene					66-142
Toluene					59-139
Chlorobenzene					60-133

COMPOUND	SPIKE ADDED (ug/kg)	MSD CONCENTRATION (ug/kg)	MSD %REC #	%RPD #	QC LIMITS	
					RPD	REC.
1,1-Dichloroethene					0-22	59-172
Trichloroethene					0-24	62-137
Benzene					0-21	66-142
Toluene					0-21	59-139
Chlorobenzene					0-21	60-133

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____
_____

3C - FORM III SV-1
WATER SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS %REC #	QC LIMITS REC.
Phenol					12-110
2-Chlorophenol					27-123
N-Nitroso-di-n-propylamine					41-116
4-Chloro-3-methylphenol					23-97
Acenaphthene					46-118
4-Nitrophenol					10-80
2,4-Dinitrotoluene					24-96
Pentachlorophenol					9-103
Pyrene					26-127

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD %REC #	%RPD #	QC LIMITS	
					RPD	REC.
Phenol					0-42	12-110
2-Chlorophenol					0-40	27-123
N-Nitroso-di-n-propylamine					0-38	41-116
4-Chloro-3-methylphenol					0-42	23-97
Acenaphthene					0-31	46-118
4-Nitrophenol					0-50	10-80
2,4-Dinitrotoluene					0-38	24-96
Pentachlorophenol					0-50	9-103
Pyrene					0-31	26-127

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____

3D - FORM III SV-2
SOIL SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____ Level: (LOW/MED)_____

COMPOUND	SPIKE ADDED (ug/kg)	SAMPLE CONCENTRATION (ug/kg)	MS CONCENTRATION (ug/kg)	MS %REC #	QC LIMITS REC.
Phenol					26-90
2-Chlorophenol					25-102
N-Nitroso-di-n-propylamine					41-126
4-Chloro-3-methylphenol					26-103
Acenaphthene					31-137
4-Nitrophenol					11-114
2,4-Dinitrotoluene					28-89
Pentachlorophenol					17-109
Pyrene					35-142

COMPOUND	SPIKE ADDED (ug/kg)	MSD CONCENTRATION (ug/kg)	MSD %REC #	%RPD #	QC LIMITS	
					RPD	REC.
Phenol					0-35	26-90
2-Chlorophenol					0-50	25-102
N-Nitroso-di-n-propylamine					0-38	41-126
4-Chloro-3-methylphenol					0-33	26-103
Acenaphthene					0-19	31-137
4-Nitrophenol					0-50	11-114
2,4-Dinitrotoluene					0-47	28-89
Pentachlorophenol					0-47	17-109
Pyrene					0-36	35-142

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____
_____

3E - FORM III SV-SIM1
WATER SEMIVOLATILE SIM MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS %REC #	QC LIMITS REC.
Acenaphthene					46-118
Pentachlorophenol					9-103
Pyrene					26-127

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD %REC #	%RPD #	QC LIMITS	
					RPD	REC.
Acenaphthene					0-31	46-118
Pentachlorophenol					0-50	9-103
Pyrene					0-31	26-127

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____

3F - FORM III SV-SIM2
SOIL SEMIVOLATILE SIM MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/kg)	SAMPLE CONCENTRATION (ug/kg)	MS CONCENTRATION (ug/kg)	MS %REC #	QC LIMITS REC.
Acenaphthene					31-137
Pentachlorophenol					17-109
Pyrene					35-142

COMPOUND	SPIKE ADDED (ug/kg)	MSD CONCENTRATION (ug/kg)	MSD %REC #	%RPD #	QC LIMITS	
					RPD	REC.
Acenaphthene					0-19	31-137
Pentachlorophenol					0-47	17-109
Pyrene					0-36	35-142

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____

3G - FORM III PEST-1
WATER PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

Instrument ID: _____ GC Column:_____ ID: _____(mm)

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS %REC #	QC LIMITS REC.
gamma-BHC (Lindane)					56-123
Heptachlor					40-131
Aldrin					40-120
Dieldrin					52-126
Endrin					56-121
4,4'-DDT					38-127

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD %REC #	%RPD #	QC LIMITS	
					RPD	REC.
gamma-BHC (Lindane)					0-15	56-123
Heptachlor					0-20	40-131
Aldrin					0-22	40-120
Dieldrin					0-18	52-126
Endrin					0-21	56-121
4,4'-DDT					0-27	38-127

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____
_____

3H - FORM III PEST-2
SOIL PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

Instrument ID: _____ GC Column:_____ ID: _____(mm)

COMPOUND	SPIKE ADDED (ug/kg)	SAMPLE CONCENTRATION (ug/kg)	MS CONCENTRATION (ug/kg)	MS %REC #	QC LIMITS REC.
gamma-BHC (Lindane)					46-127
Heptachlor					35-130
Aldrin					34-132
Dieldrin					31-134
Endrin					42-139
4,4'-DDT					23-134

COMPOUND	SPIKE ADDED (ug/kg)	MSD CONCENTRATION (ug/kg)	MSD %REC #	%RPD #	QC LIMITS	
					RPD	REC.
gamma-BHC (Lindane)					0-50	46-127
Heptachlor					0-31	35-130
Aldrin					0-43	34-132
Dieldrin					0-38	31-134
Endrin					0-45	42-139
4,4'-DDT					0-50	23-134

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____

_____

3J - FORM III ARO-1
WATER AROCLOR MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

Instrument ID: _____ GC Column:_____ ID: _____(mm)

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC LIMITS REC.
AR1016					29-135
AR1260					29-135

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	%RPD #	QC LIMITS	
					RPD	REC.
AR1016					0-15	29-135
AR1260					0-20	29-135

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____

3K - FORM III ARO-2
SOIL AROCLOR MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

Instrument ID: _____ GC Column:_____ ID: _____(mm)

COMPOUND	SPIKE ADDED (ug/kg)	SAMPLE CONCENTRATION (ug/kg)	MS CONCENTRATION (ug/kg)	MS % REC #	QC LIMITS REC.
AR1016					29-135
AR1260					29-135

COMPOUND	SPIKE ADDED (ug/kg)	MSD CONCENTRATION (ug/kg)	MSD % REC #	% RPD #	QC LIMITS	
					RPD	REC.
AR1016					0-15	29-135
AR1260					0-20	29-135

Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: ___ out of ___ outside limits
Spike Recovery: ___ out of ___ outside limits

COMMENTS: _____
_____

3L - FORM III PEST-3
WATER PESTICIDE LABORATORY CONTROL
SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab Sample ID: _____ LCS Lot No.: _____

Date Extracted: _____ Date Analyzed (1): _____

Instrument ID (1): _____ GC Column (1):_____ ID: _____(mm)

COMPOUND	AMOUNT ADDED (ug/L)	AMOUNT RECOVERED (ug/L)	%REC #	QC LIMITS
gamma-BHC (Lindane)				50-120
Heptachlor epoxide				50-150
Dieldrin				30-130
4,4'-DDE				50-150
Endrin				50-120
Endosulfan sulfate				50-120
gamma-Chlordane				30-130

Instrument ID (2): _____ GC Column (2):_____ ID: _____(mm)

Date Analyzed (2): _____

COMPOUND	AMOUNT ADDED (ug/L)	AMOUNT RECOVERED (ug/L)	%REC #	QC LIMITS
gamma-BHC (Lindane)				50-120
Heptachlor epoxide				50-150
Dieldrin				30-130
4,4'-DDE				50-150
Endrin				50-120
Endosulfan sulfate				50-120
gamma-Chlordane				30-130

Column to be used to flag recovery values with an asterisk
* Values outside of QC limits

LCS Recovery:_____ out of _____ outside limits.

COMMENTS: _____

_____

3M - FORM III PEST-4
SOIL PESTICIDE LABORATORY CONTROL
SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab Sample ID: _____ LCS Lot No.: _____

Date Extracted: _____ Date Analyzed (1): _____

Instrument ID (1): _____ GC Column (1):_____ ID: _____(mm)

COMPOUND	AMOUNT ADDED (ug/kg)	AMOUNT RECOVERED (ug/kg)	%REC #	QC LIMITS
gamma-BHC (Lindane)				50-120
Heptachlor epoxide				50-150
Dieldrin				30-130
4,4'-DDE				50-150
Endrin				50-120
Endosulfan sulfate				50-120
gamma-Chlordane				30-130

Instrument ID (2): _____ GC Column (2):_____ ID: _____(mm)

Date Analyzed (2): _____

COMPOUND	AMOUNT ADDED (ug/kg)	AMOUNT RECOVERED (ug/kg)	%REC #	QC LIMITS
gamma-BHC (Lindane)				50-120
Heptachlor epoxide				50-150
Dieldrin				30-130
4,4'-DDE				50-150
Endrin				50-120
Endosulfan sulfate				50-120
gamma-Chlordane				30-130

Column to be used to flag recovery values with an asterisk
* Values outside of QC limits

LCS Recovery:_____ out of _____ outside limits.

COMMENTS: _____

_____

3N - FORM III ARO-3
WATER AROCLOR LABORATORY CONTROL
SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab Sample ID: _____ LCS Lot No.: _____

Date Extracted: _____ Date Analyzed (1): _____

Instrument ID (1): _____ GC Column (1):_____ ID: _____(mm)

COMPOUND	AMOUNT ADDED (ug/L)	AMOUNT RECOVERED (ug/L)	%REC #	QC LIMITS
AR1016				50-150
AR1260				50-150

Instrument ID (2): _____ GC Column (2):_____ ID: _____(mm)

Date Analyzed (2): _____

COMPOUND	AMOUNT ADDED (ug/L)	AMOUNT RECOVERED (ug/L)	%REC #	QC LIMITS
AR1016				50-150
AR1260				50-150

Column to be used to flag recovery values with an asterisk
* Values outside of QC limits

LCS Recovery:_____ out of _____ outside limits.

COMMENTS: _____

_____

3P - FORM III ARO-4
SOIL AROCLOR LABORATORY CONTROL
SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab Sample ID: _____ LCS Lot No.: _____

Date Extracted: _____ Date Analyzed (1): _____

Instrument ID (1): _____ GC Column (1):_____ ID: _____(mm)

COMPOUND	AMOUNT ADDED (ug/kg)	AMOUNT RECOVERED (ug/kg)	%REC #	QC LIMITS
AR1016				50-150
AR1260				50-150

Instrument ID (2): _____ GC Column (2):_____ ID: _____(mm)

Date Analyzed (2): _____

COMPOUND	AMOUNT ADDED (ug/kg)	AMOUNT RECOVERED (ug/kg)	%REC #	QC LIMITS
AR1016				50-150
AR1260				50-150

Column to be used to flag recovery values with an asterisk
* Values outside of QC limits

LCS Recovery:_____ out of _____ outside limits.

COMMENTS: _____

_____

4A - FORM IV VOA
VOLATILE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Instrument ID: _____

Matrix: (SOIL/SED/WATER) _____ Date Analyzed: _____

Level: (TRACE or LOW/MED)_____ Time Analyzed: _____

GC Column: _____ ID: _____(mm) Heated Purge: (Y/N) _____

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: _____

4B - FORM IV VOA-SIM
TRACE VOLATILE (WATER) SIM METHOD
BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Instrument ID: _____ Date Analyzed: _____

GC Column: _____ ID: _____(mm) Time Analyzed: _____

Heated Purge: (Y/N) _____

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: _____

4C - FORM IV SV
SEMIVOLATILE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Instrument ID: _____ Date Extracted: _____

Matrix: (SOIL/SED/WATER) _____ Date Analyzed: _____

Level: (LOW/MED)_____ Time Analyzed: _____

Extraction: (Type) _____ GPC Cleanup: (Y/N)_____

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: _____

4D - FORM IV SV-SIM
SEMIVOLATILE SIM METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Instrument ID: _____ Date Extracted: _____

Matrix: (SOIL/SED/WATER): _____ Date Analyzed: _____

Time Analyzed: _____ Extraction: (Type)_____ GPC Cleanup: (Y/N)_____

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: _____

4E - FORM IV PEST
PESTICIDE METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Matrix: (SOIL/SED/WATER) _____ Extraction: (Type)_____ Date Extracted: _____

Sulfur Cleanup: (Y/N)_____ GPC Cleanup: (Y/N)_____

Date Analyzed (1): _____ Date Analyzed (2): _____

Time Analyzed (1): _____ Time Analyzed (2): _____

Instrument ID (1): _____ Instrument ID (2): _____

GC Column(1): _____ ID: ____(mm) GC Column(2): _____ ID: ____(mm)

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED (1)	DATE ANALYZED (2)
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				

COMMENTS: _____

4F - FORM IV ARO
AROCLOR METHOD BLANK SUMMARY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab File ID: _____ Lab Sample ID: _____

Matrix: (SOIL/SED/WATER) _____ Extraction: (Type)_____ Date Extracted: _____

Sulfur Cleanup: (Y/N)_____ GPC Cleanup: (Y/N)_____

Acid Cleanup: (Y/N)_____

Date Analyzed (1): _____ Date Analyzed (2): _____

Time Analyzed (1): _____ Time Analyzed (2): _____

Instrument ID (1): _____ Instrument ID (2): _____

GC Column(1): _____ ID: ____(mm) GC Column(2): _____ ID: ____(mm)

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED (1)	DATE ANALYZED (2)
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				

COMMENTS: _____

5A - FORM V VOA
VOLATILE ORGANIC INSTRUMENT
PERFORMANCE CHECK
BROMOFLUOROBENZENE (BFB)

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab File ID: _____ BFB Injection Date: _____

Instrument ID: _____ BFB Injection Time: _____

GC Column: _____ ID: _____ (mm)

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	
75	30.0 - 80.0% of mass 95	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of mass 95	
173	Less than 2.0% of mass 174	( )1
174	50.0 - 120% of mass 95	
175	5.0 - 9.0% of mass 174	( )1
176	95.0 - 101% of mass 174	( )1
177	5.0 - 9.0% of mass 176	( )2

1 - Value is %mass 174 2 - Value is %mass 176

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

5B - FORM V SV
SEMIVOLATILE ORGANIC INSTRUMENT
PERFORMANCE CHECK
DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab File ID: _____ DFTPP Injection Date: _____

Instrument ID: _____ DFTPP Injection Time: _____

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	10.0 - 80.0% of mass 198	
68	Less than 2.0% of mass 69	( )1
69	Mass 69 relative abundance	
70	Less than 2.0% of mass 69	( )1
127	10.0 - 80.0% of mass 198	
197	Less than 2.0% of mass 198	
198	Base Peak, 100% relative abundance	
199	5.0 to 9.0% of mass 198	
275	10.0 - 60.0% of mass 198	
365	Greater than 1.0% of mass 198	
441	Present, but less than mass 443	
442	50.0 - 100% of mass 198	
443	15.0 - 24.0% of mass 442	( )2

1 - Value is %mass 69 2 - Value is% mass 442

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					

6A - FORM VI VOA-1
VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Heated Purge: (Y/N) _____ Calibration Time(s): _____ _____

Purge Volume: _____ (mL)

GC Column: _____ ID: _____ (mm) Length: _____ (m)

LAB FILE ID: _____ RRF___ = _____ RRF___ = _____

RRF___ = _____ RRF___ = _____ RRF___ = _____

COMPOUND	RRF___	RRF___	RRF___	RRF___	RRF___	$\overline{RRF}$	%RSD
Dichlorodifluoromethane							
Chloromethane							
Vinyl chloride							
Bromomethane							
Chloroethane							
Trichlorofluoromethane							
1,1-Dichloroethene							
1,1,2-Trichloro-1,2,2-trifluoroethane							
Acetone							
Carbon disulfide							
Methyl acetate							
Methylene chloride							
trans-1,2-Dichloroethene							
Methyl tert-butyl ether							
1,1-Dichloroethane							
cis-1,2-Dichloroethene							
2-Butanone							
Bromochloromethane							
Chloroform							
1,1,1-Trichloroethane							
Cyclohexane							
Carbon tetrachloride							
Benzene							
1,2-Dichloroethane							
1,4-Dioxane							
Trichloroethene							
Methylcyclohexane							

6B - FORM VI VOA-2
VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Heated Purge: (Y/N) _____ Calibration Time(s): _____ _____

Purge Volume: _____ (mL)

GC Column: _____ ID: _____ (mm) Length: _____ (m)

LAB FILE ID: _____ RRF___ = _____ RRF___ = _____

RRF___= _____ RRF___ = _____ RRF___ = _____

COMPOUND	RRF___	RRF___	RRF___	RRF___	RRF___	$\overline{RRF}$	% RSD
1,2-Dichloropropane							
Bromodichloromethane							
cis-1,3-Dichloropropene							
4-Methyl-2-pentanone							
Toluene							
trans-1,3-Dichloropropene							
1,1,2-Trichloroethane							
Tetrachloroethene							
2-Hexanone							
Dibromochloromethane							
1,2-Dibromoethane							
Chlorobenzene							
Ethylbenzene							
o-Xylene							
m,p-Xylene							
Styrene							
Bromoform							
Isopropylbenzene							
1,1,2,2-Tetrachloroethane							
1,3-Dichlorobenzene							
1,4-Dichlorobenzene							
1,2-Dichlorobenzene							
1,2-Dibromo-3-chloropropane							
1,2,4-Trichlorobenzene							
1,2,3-Trichlorobenzene							

6C - FORM VI VOA-3
VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Heated Purge: (Y/N) _____ Calibration Time(s): _____ _____

Purge Volume: _____ (mL)

GC Column: _____ ID: _____ (mm) Length: _____ (m)

LAB FILE ID: _____ RRF___ = _____ RRF___ = _____

RRF___= _____ RRF___ = _____ RRF___ = _____

COMPOUND	RRF___	RRF___	RRF___	RRF___	RRF___	$\overline{RRF}$	% RSD
Vinyl chloride-d_3							
Chloroethane-d_5							
1,1-Dichloroethene-d_2							
2-Butanone-d_5							
Chloroform-d							
1,2-Dichloroethane-d_4							
Benzene-d_6							
1,2-Dichloropropane-d_6							
Toluene-d_8							
trans-1,3-Dichloropropene-d_4							
2-Hexanone-d_5							
1,4-Dioxane-d_8							
1,1,2,2-Tetrachloroethane-d_2							
1,2-Dichlorobenzene-d_4							

6D - FORM VI VOA-SIM
TRACE VOLATILE (WATER) ORGANICS SIM INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Heated Purge: (Y/N) _____ Calibration Time(s): _____ _____

GC Column: _____ ID: _____ (mm) Length: _____ (m)

LAB FILE ID: _____ RRF___ = _____ RRF___ = _____

RRF___= _____ RRF___ = _____ RRF___ = _____

COMPOUND	RRF___	RRF___	RRF___	RRF___	RRF___	$\overline{RRF}$	% RSD
1,4-Dioxane							
1,2-Dibromoethane							
1,2-Dibromo-3-chloropropane							
1,2-Dichloroethane-d_4							
1,4-Dioxane-d_8							
1,1,2,2-Tetrachloroethane-d_2							

6E - FORM VI SV-1
SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Time(s): _____ _____

LAB FILE ID: _____ RRF___ = _____ RRF___ = _____

RRF___= _____ RRF___ = _____ RRF___ = _____

COMPOUND	RRF___	RRF___	RRF___	RRF___	RRF___	$\overline{RRF}$	% RSD
Benzaldehyde							
Phenol							
Bis(2-chloroethyl)ether							
2-Chlorophenol							
2-Methylphenol							
2,2'-Oxybis(1-chloropropane)							
Acetophenone							
4-Methylphenol							
N-Nitroso-di-n-propylamine							
Hexachloroethane							
Nitrobenzene							
Isophorone							
2-Nitrophenol							
2,4-Dimethylphenol							
Bis(2-chloroethoxy)methane							
2,4-Dichlorophenol							
Naphthalene							
4-Chloroaniline							
Hexachlorobutadiene							
Caprolactam							
4-Chloro-3-methylphenol							
2-Methylnaphthalene							
Hexachlorocyclopentadiene							
2,4,6-Trichlorophenol							
2,4,5-Trichlorophenol							
1,1'-Biphenyl							
2-Chloronaphthalene							
2-Nitroaniline							
Dimethylphthalate							
2,6-Dinitrotoluene							
Acenaphthylene							
3-Nitroaniline							
Acenaphthene							
2,4-Dinitrophenol							
4-Nitrophenol							
Dibenzofuran							

6F - FORM VI SV-2
SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Time(s): _____ _____

LAB FILE ID: _____ RRF___ = _____ RRF___ = _____

RRF___= _____ RRF___ = _____ RRF___ = _____

COMPOUND	RRF___	RRF___	RRF___	RRF___	RRF___	$\overline{RRF}$	% RSD
2,4-Dinitrotoluene							
Diethylphthalate							
Fluorene							
4-Chlorophenyl-phenylether							
4-Nitroaniline							
4,6-Dinitro-2-methylphenol							
N-Nitrosodiphenylamine[1]							
1,2,4,5-Tetrachlorobenzene							
4-Bromophenyl-phenylether							
Hexachlorobenzene							
Atrazine							
Pentachlorophenol							
Phenanthrene							
Anthracene							
Carbazole							
Di-n-butylphthalate							
Fluoranthene							
Pyrene							
Butylbenzylphthalate							
3,3'-Dichlorobenzidine							
Benzo(a)anthracene							
Chrysene							
Bis(2-ethylhexyl)phthalate							
Di-n-octylphthalate							
Benzo(b)fluoranthene							
Benzo(k)fluoranthene							
Benzo(a)pyrene							
Indeno(1,2,3-cd)pyrene							
Dibenzo(a,h)anthracene							
Benzo(g,h,i)perylene							
2,3,4,6-Tetrachlorophenol							

[1]Cannot be separated from Diphenylamine

6G - FORM VI SV-3
SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Time(s): _____ _____

LAB FILE ID: _____ RRF___ = _____ RRF___ = _____

RRF___= _____ RRF___ = _____ RRF___ = _____

COMPOUND	RRF___	RRF___	RRF___	RRF___	RRF___	$\overline{RRF}$	% RSD
Phenol-d_5							
Bis(2-chloroethyl)ether-d_8							
2-Chlorophenol-d_4							
4-Methylphenol-d_8							
Nitrobenzene-d_5							
2-Nitrophenol-d_4							
2,4-Dichlorophenol-d_3							
4-Chloroaniline-d_4							
Dimethylphthalate-d_6							
Acenaphthylene-d_8							
4-Nitrophenol-d_4							
Fluorene-d_{10}							
4,6-Dinitro-methylphenol-d_2							
Anthracene-d_{10}							
Pyrene-d_{10}							
Benzo(a)pyrene-d_{12}							

6H - FORM VI SV-SIM
SEMIVOLATILE ORGANICS SIM INITIAL CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Calibration Date(s): _____ _____

Calibration Time(s): _____ _____

LAB FILE ID: _____ RRF___ = _____ RRF___ = _____

RRF___= _____ RRF___ = _____ RRF___ = _____

COMPOUND	RRF___	RRF___	RRF___	RRF___	RRF___	$\overline{RRF}$	% RSD
Naphthalene							
2-Methylnaphthalene							
Acenaphthylene							
Acenaphthene							
Fluorene							
Pentachlorophenol							
Phenanthrene							
Anthracene							
Fluoranthene							
Pyrene							
Benzo(a)anthracene							
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							
Fluoranthene-d_{10}							
2-Methylnaphthalene-d_{10}							

6J - FORM VI PEST-1
PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____

Level (x CS1): CS1 ___ CS2 ___ CS3___ CS4 ___ CS5 ___

GC Column: _____ ID: _____ (mm) Date(s) Analyzed:_____ _____

COMPOUND	RT OF STANDARDS					$\overline{RT}$	RT WINDOW*	
	CS1	CS2	CS3	CS4	CS5		FROM	TO
alpha-BHC								
beta-BHC								
delta-BHC								
gamma-BHC (Lindane)								
Heptachlor								
Aldrin								
Heptachlor epoxide								
Endosulfan I								
Dieldrin								
4,4'-DDE								
Endrin								
Endosulfan II								
4,4'-DDD								
Endosulfan sulfate								
4,4'-DDT								
Methoxychlor								
Endrin ketone								
Endrin aldehyde								
alpha-Chlordane								
gamma-Chlordane								
TCX (A)								
DCB (A)								
TCX (B)								
DCB (B)								

(A) Surrogate RTs are measured from Standard Mixture A if two mixtures are used or from Standard Mixture C if one mixture is used.

(B) Surrogate RTs are measured from Standard Mixture B if two mixtures are used. Leave entries blank if Standard Mixture C is used.

* RT windows are ± 0.05 minutes for all compounds that elute before Heptachlor epoxide; ± 0.07 minutes for all other compounds (except ± 0.10 minutes for DCB).

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

6K - FORM VI PEST-2
PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____

Level (x CS1): CS1 ___ CS2 ___ CS3___ CS4 ___ CS5 ___

GC Column: _____ ID: _____ (mm) Date(s) Analyzed:_____ _____

COMPOUND	CALIBRATION FACTORS (CFs)					% RSD
	CS1	CS2	CS3	CS4	CS5	
alpha-BHC						
beta-BHC						
delta-BHC						
gamma-BHC (Lindane)						
Heptachlor						
Aldrin						
Heptachlor epoxide						
Endosulfan I						
Dieldrin						
4,4'-DDE						
Endrin						
Endosulfan II						
4,4'-DDD						
Endosulfan sulfate						
4,4'-DDT						
Methoxychlor						
Endrin ketone						
Endrin aldehyde						
alpha-Chlordane						
gamma-Chlordane						
TCX (A)						
DCB (A)						
TCX (B)						
DCB (B)						

(A) Surrogate CFs and %RSD are measured from Standard Mixture A if two mixtures are used or from Standard Mixture C if one mixture is used.

(B) Surrogate CFs and %RSD are measured from Standard Mixture B if two mixtures are used. Leave entries blank if Standard Mixture C is used.

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

6L - FORM VI PEST-3
TOXAPHENE INITIAL CALIBRATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID (1): _____ Date(s) Analyzed (1): _____

GC Column (1): _____ ID: _____ (mm)

Level (x CS1): CS1 ___ CS2 ___ CS3___ CS4 ___ CS5 ___

COMPOUND	PEAK[1]	RT OF STANDARDS						RT WINDOW	
		CS1	CS2	CS3	CS4	CS5	$\overline{RT}$	FROM	TO
Toxaphene	1								
	2								
	3								
	4								
	5								

Instrument ID (2): _____ Date(s) Analyzed (2): _____

GC Column (2): _____ ID: _____ (mm)

Level (x CS1): CS1 ___ CS2 ___ CS3___ CS4 ___ CS5 ___

COMPOUND	PEAK[1]	RT OF STANDARDS						RT WINDOW	
		CS1	CS2	CS3	CS4	CS5	$\overline{RT}$	FROM	TO
Toxaphene	1								
	2								
	3								
	4								
	5								

[1]At least three peaks for each column are required for identification of Toxaphene.

6M - FORM VI PEST-4
TOXAPHENE INITIAL CALIBRATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID (1): _____ Date(s) Analyzed (1): _____

GC Column (1): _____ ID: _____ (mm)

Level (x CS1): CS1 ___ CS2 ___ CS3___ CS4 ___ CS5 ___

COMP-OUND	PEAK[1]	CALIBRATION FACTORS (CFs) STANDARDS					%RSD
		CS1	CS2	CS3	CS4	CS5	
Toxaphene	1						
	2						
	3						
	4						
	5						

Instrument ID (2): _____ Date(s) Analyzed (2): _____

GC Column (2): _____ ID: _____ (mm)

Level (x CS1): CS1 ___ CS2 ___ CS3___ CS4 ___ CS5 ___

COMP-OUND	PEAK[1]	CALIBRATION FACTORS (CFs) STANDARDS					%RSD
		CS1	CS2	CS3	CS4	CS5	
Toxaphene	1						
	2						
	3						
	4						
	5						

[1]At least three peaks for each column are required for identification of Toxaphene.

6N - FORM VI ARO-1
AROCLORS INITIAL CALIBRATION (MULTIPOINT)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____

Level (x CS1): CS1 ___ CS2 ___ CS3___ CS4 ___ CS5 ___

GC Column: _____ ID: _____ (mm) Date(s) Analyzed:_____ _____

COMPOUND	PEAK*	RT OF STANDARDS					$\overline{RT}$	RT WINDOW**	
		CS1	CS2	CS3	CS4	CS5		FROM	TO
AR1016	1								
	2								
	3								
	4								
	5								
TCX									
DCB									
AR1260	1								
	2								
	3								
	4								
	5								
TCX									
DCB									
AR_____	1								
	2								
	3								
	4								
	5								
TCX									
DCB									

*At least three peaks for each column are required for identification of Aroclors.

**Retention Time windows are ± 0.07 minutes for each Aroclor peak; ± 0.05 minutes for TCX; and ± 0.10 minutes for DCB.

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

6P - FORM VI ARO-2
AROCLORS INITIAL CALIBRATION (MULTIPOINT)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____

Level (x CS1): CS1 ___ CS2 ___ CS3___ CS4 ___ CS5 ___

GC Column: _____ ID: _____ (mm) Date(s) Analyzed:_____ _____

COMP-OUND	PEAK[1]	CALIBRATION FACTORS (CFs)					%RSD
		CS1	CS2	CS3	CS4	CS5	
AR1016	1						
	2						
	3						
	4						
	5						
TCX							
DCB							
AR1260	1						
	2						
	3						
	4						
	5						
TCX							
DCB							
AR____	1						
	2						
	3						
	4						
	5						
TCX							
DCB							

[1]At least three peaks for each column are required for identification of Aroclors.

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

6Q - FORM VI ARO-3
AROCLOR INITIAL CALIBRATION (SINGLE POINT)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID: _____ Date(s) Analyzed: _____ _____

GC Column: _____ ID: _____ (mm)

COMPOUND	AMOUNT (ng)	PEAK[1]	RT	RT WINDOW		CALIBRATION FACTOR
				FROM	TO	
Aroclor 1221		1				
		2				
		3				
		4				
		5				
Aroclor 1232		1				
		2				
		3				
		4				
		5				
Aroclor 1242		1				
		2				
		3				
		4				
		5				
Aroclor 1248		1				
		2				
		3				
		4				
		5				
Aroclor 1254		1				
		2				
		3				
		4				
		5				
Aroclor 1262		1				
		2				
		3				
		4				
		5				
Aroclor 1268		1				
		2				
		3				
		4				
		5				

[1]At least three peaks for each column are required for identification of multicomponent analytes.

6R - FORM VI PEST-5
PESTICIDE RESOLUTION CHECK SUMMARY
COLUMN 1

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column (1): _____ ID: _____ (mm) Instrument ID (1): _____

EPA Sample No. (RESC##): _____ Lab Sample ID (1): _____

Date Analyzed (1):_____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			

6T - FORM VI PEST-5
PESTICIDE RESOLUTION CHECK SUMMARY
COLUMN 2

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column (2): _____ ID: _____ (mm) Instrument ID (2): _____

EPA Sample No. (RESC##): _____ Lab Sample ID (2): _____

Date Analyzed (2):_____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			

6U - FORM VI PEST-6
PERFORMANCE EVALUATION MIXTURE (PEM)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column (1): _____ ID: _____ (mm) Instrument ID (1): _____

EPA Sample No. (PEM##): _____ Lab Sample ID (1): _____

Date Analyzed (1):_____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			

GC Column (2):_____ ID:_____(mm) Instrument ID (2): _____

EPA Sample No. (PEM##): _____ Lab Sample ID (2): _____

Date Analyzed (2): _____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			

6V - FORM VI PEST-7
INDIVIDUAL STANDARD MIXTURE A

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column (1):_____ ID:_____(mm) Instrument ID (1): _____

EPA Sample No. (INDA3##): _____ Lab Sample ID (1): _____

Date Analyzed (1): _____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			

GC Column (2):_____ ID:_____(mm) Instrument ID (2): _____

EPA Sample No. (INDA3##): _____ Lab Sample ID (2): _____

Date Analyzed (2): _____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			

6W - FORM VI PEST-8
INDIVIDUAL STANDARD MIXTURE B

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column (1):_____ ID:_____(mm) Instrument ID (1): _____

EPA Sample No. (INDB3##): _____ Lab Sample ID (1): _____

Date Analyzed (1): _____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			

GC Column (2):_____ ID:_____(mm) Instrument ID (2): _____

EPA Sample No. (INDB3##): _____ Lab Sample ID (2): _____

Date Analyzed (2): _____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			

6X - FORM VI PEST-9
INDIVIDUAL STANDARD MIXTURE C

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column (1):_____ ID:_____(mm) Instrument ID (1): _____

EPA Sample No. (INDC3##): _____ Lab Sample ID (1): _____

Date Analyzed (1): _____ Time Analyzed (1): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			

6Y - FORM VI PEST-10
INDIVIDUAL STANDARD MIXTURE C

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column (2):_____ ID:_____(mm) Instrument ID (2): _____

EPA Sample No. (INDC3##): _____ Lab Sample ID (2): _____

Date Analyzed (2): _____ Time Analyzed (2): _____

	ANALYTE	RT	RESOLUTION (%)
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			

7A - FORM VII VOA-1
VOLATILE CONTINUING CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID:_____ Calibration Date:_____ Time:_____

Lab File ID:_____ Init. Calib. Date(s):_____ _____

EPA Sample No.(VSTD#####):_____ Init. Calib. Time(s):_____ _____

Heated Purge: (Y/N) _____ GC Column:_____ ID:_____(mm) Length: _____(m)

Purge Volume: _____(mL)

COMPOUND	$\overline{RRF}$	RRF__	MIN RRF	%D	MAX %D
Dichlorodifluoromethane					
Chloromethane					
Vinyl chloride					
Bromomethane					
Chloroethane					
Trichlorofluoromethane					
1,1-Dichloroethene					
1,1,2-Trichloro-1,2,2-trifluoroethane					
Acetone					
Carbon disulfide					
Methyl acetate					
Methylene chloride					
trans-1,2-Dichloroethene					
Methyl tert-butyl ether					
1,1-Dichloroethane					
cis-1,2-Dichloroethene					
2-Butanone					
Bromochloromethane					
Chloroform					
1,1,1-Trichloroethane					
Cyclohexane					
Carbon tetrachloride					
Benzene					
1,2-Dichloroethane					
1,4-Dioxane					
Trichloroethene					
Methylcyclohexane					

7B - FORM VII VOA-2
VOLATILE CONTINUING CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID:_____ Calibration Date:_____ Time:_____

Lab File ID:_____ Init. Calib. Date(s):_____ _____

EPA Sample No.(VSTD#####):_____ Init. Calib. Time(s):_____ _____

Heated Purge: (Y/N) _____ GC Column:_____ ID:_____(mm) Length: _____(m)

Purge Volume: _____(mL)

COMPOUND	$\overline{RRF}$	RRF__	MIN RRF	%D	MAX %D
1,2-Dichloropropane					
Bromodichloromethane					
cis-1,3-Dichloropropene					
4-Methyl-2-pentanone					
Toluene					
trans-1,3-Dichloropropene					
1,1,2-Trichloroethane					
Tetrachloroethene					
2-Hexanone					
Dibromochloromethane					
1,2-Dibromoethane					
Chlorobenzene					
Ethylbenzene					
o-Xylene					
m,p-Xylene					
Styrene					
Bromoform					
Isopropylbenzene					
1,1,2,2-Tetrachloroethane					
1,3-Dichlorobenzene					
1,4-Dichlorobenzene					
1,2-Dichlorobenzene					
1,2-Dibromo-3-chloropropane					
1,2,4-Trichlorobenzene					
1,2,3-Trichlorobenzene					

7C - FORM VII VOA-3
VOLATILE CONTINUING CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID:_____ Calibration Date:_____ Time:_____

Lab File ID:_____ Init. Calib. Date(s):_____ _____

EPA Sample No.(VSTD#####):_____ Init. Calib. Time(s):_____ _____

Heated Purge: (Y/N) _____ GC Column:_____ ID:_____(mm) Length: _____(m)

Purge Volume: _____(mL)

COMPOUND	$\overline{RRF}$	RRF__	MIN RRF	%D	MAX %D
Vinyl chloride-d_3					
Chloroethane-d_5					
1,1-Dichloroethene-d_2					
2-Butanone-d_5					
Chloroform-d					
1,2-Dichloroethane-d_4					
Benzene-d_6					
1,2-Dichloropropane-d_6					
Toluene-d_8					
trans-1,3-Dichloropropene-d_4					
2-Hexanone-d_5					
1,4-Dioxane-d_8					
1,1,2,2-Tetrachloroethane-d_2					
1,2-Dichlorobenzene-d_4					

7D - FORM VII VOA-SIM
TRACE VOLATILE (WATER) SIM CONTINUING CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID:_____ Calibration Date:_____ Time:_____

Lab File ID:_____ Init. Calib. Date(s):_____ _____

EPA Sample No.(VSTD#####):_____ Init. Calib. Time(s):_____ _____

Heated Purge: (Y/N) _____ GC Column:_____ ID:_____(mm) Length: _____(m)

COMPOUND	$\overline{RRF}$	RRF__	MIN RRF	%D	MAX %D
1,4-Dioxane					
1,2-Dibromoethane					
1,2-Dibromo-3-chloropropane					
1,2-Dichloroethane-d_4					
1,4-Dioxane-d_8					
1,1,2,2-Tetrachloroethane-d_2					

7E - FORM VII SV-1
SEMIVOLATILE CONTINUING CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID:_____ Calibration Date:_____ Time:_____

Lab File ID:_____ Init. Calib. Date(s):_____ _____

EPA Sample No.(SSTD020##):_____ Init. Calib. Time(s):_____ _____

GC Column:_____ ID:_____(mm)

COMPOUND	$\overline{\text{RRF}}$	RRF__	MIN RRF	%D	MAX %D
Benzaldehyde					
Phenol					
Bis(2-chloroethyl)ether					
2-Chlorophenol					
2-Methylphenol					
2,2'-Oxybis(1-chloropropane)					
Acetophenone					
4-Methylphenol					
N-Nitroso-di-n-propylamine					
Hexachloroethane					
Nitrobenzene					
Isophorone					
2-Nitrophenol					
2,4-Dimethylphenol					
Bis(2-chloroethoxy)methane					
2,4-Dichlorophenol					
Naphthalene					
4-Chloroaniline					
Hexachlorobutadiene					
Caprolactam					
4-Chloro-3-methylphenol					
2-Methylnaphthalene					
Hexachlorocyclopentadiene					
2,4,6-Trichlorophenol					
2,4,5-Trichlorophenol					
1,1'-Biphenyl					
2-Chloronaphthalene					
2-Nitroaniline					
Dimethylphthalate					
2,6-Dinitrotoluene					
Acenaphthylene					
3-Nitroaniline					
Acenaphthene					

7F - FORM VII SV-2
SEMIVOLATILE CONTINUING CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID:_____ Calibration Date:_____ Time:_____

Lab File ID:_____ Init. Calib. Date(s):_____ _____

EPA Sample No.(SSTD020##):_____ Init. Calib. Time(s):_____ _____

GC Column:_____ ID:_____(mm)

COMPOUND	$\overline{RRF}$	RRF__	MIN RRF	%D	MAX %D
2,4-Dinitrotoluene					
Diethylphthalate					
Fluorene					
4-Chlorophenyl-phenylether					
4-Nitroaniline					
4,6-Dinitro-2-methylphenol					
N-Nitrosodiphenylamine (1)					
1,2,4,5-Tetrachlorobenzene					
4-Bromophenyl-phenylether					
Hexachlorobenzene					
Atrazine					
Pentachlorophenol					
Phenanthrene					
Anthracene					
Carbazole					
Di-n-butylphthalate					
Fluoranthene					
Pyrene					
Butylbenzylphthalate					
3,3'-Dichlorobenzidine					
Benzo(a)anthracene					
Chrysene					
Bis(2-ethylhexyl)phthalate					
Di-n-octylphthalate					
Benzo(b)fluoranthene					
Benzo(k)fluoranthene					
Benzo(a)pyrene					
Indeno(1,2,3-cd)pyrene					
Dibenzo(a,h)anthracene					
Benzo(g,h,i)perylene					
2,3,4,6-Tetrachlorophenol					

(1) Cannot be separated from Diphenylamine

7G - FORM VII SV-3
SEMIVOLATILE CONTINUING CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID:_____ Calibration Date:_____ Time:_____

Lab File ID:_____ Init. Calib. Date(s):_____ _____

EPA Sample No.(SSTD020##):_____ Init. Calib. Time(s):_____ _____

GC Column:_____ ID:_____(mm)

COMPOUND	$\overline{RRF}$	RRF__	MIN RRF	%D	MAX %D
Phenol-d_5					
Bis(2-chloroethyl)ether-d_8					
2-Chlorophenol-d_4					
4-Methylphenol-d_8					
Nitrobenzene-d_5					
2-Nitrophenol-d_4					
2,4-Dichlorophenol-d_3					
4-Chloroaniline-d_4					
Dimethylphthalate-d_6					
Acenaphthylene-d_8					
4-Nitrophenol-d_4					
Fluorene-d_{10}					
4,6-Dinitro-methylphenol-d_2					
Anthracene-d_{10}					
Pyrene-d_{10}					
Benzo(a)pyrene-d_{12}					

7H - FORM VII SV-SIM
SEMIVOLATILE SIM CONTINUING CALIBRATION DATA

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Instrument ID:_____ Calibration Date:_____ Time:_____

Lab File ID:_____ Init. Calib. Date(s):_____ _____

EPA Sample No.(SSTD0.4##):_____ Init. Calib. Time(s):_____ _____

GC Column:_____ ID:_____(mm)

COMPOUND	$\overline{\text{RRF}}$	RRF__	MIN RRF	%D	MAX %D
Naphthalene					
2-Methylnaphthalene					
Acenaphthylene					
Acenaphthene					
Fluorene					
Pentachlorophenol					
Phenanthrene					
Anthracene					
Fluoranthene					
Pyrene					
Benzo(a)anthracene					
Chrysene					
Benzo(b)fluoranthracene					
Benzo(k)fluoranthracene					
Benzo(a)pyrene					
Indeno(1,2,3-cd)pyrene					
Dibenzo(a,h)anthracene					
Benzo(g,h,i)perylene					
Fluoranthene-d_{10}					
2-Methylnapthalene-d_{10}					

7J - FORM VII PEST-1
PESTICIDE CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column:_____ ID:_____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No. (PIBLK##): _____ Date Analyzed: _____

Lab Sample ID (PIBLK): _____ Time Analyzed: _____

EPA Sample No. (PEM##): _____ Date Analyzed: _____

Lab Sample ID (PEM): _____ Time Analyzed: _____

PEM COMPOUND	RT	RT WINDOW		CALC AMOUNT (ng)	NOM AMOUNT (ng)	%D
		FROM	TO			
alpha-BHC						
beta-BHC						
gamma-BHC (Lindane)						
Endrin						
4,4'-DDT						
Methoxychlor						
TCX						
DCB						

4,4'-DDT %Breakdown (1): _____ Endrin %Breakdown (1):_____

Combined %Breakdown (1): _____

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

7K - FORM VII PEST-2
PESTICIDE CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column:_____ ID:_____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No. (PIBLK##): _____ Date Analyzed: _____

Lab Sample ID (PIBLK): _____ Time Analyzed: _____

EPA Sample No. (INDA3##): _____ Date Analyzed: _____

Lab Sample ID (INDA3): _____ Time Analyzed: _____

INDIVIDUAL MIX A COMPOUND	RT	RT WINDOW		$\overline{CF}$	CF	%D
		FROM	TO			
alpha-BHC						
gamma-BHC (Lindane)						
Heptachlor						
Endosulfan I						
Dieldrin						
Endrin						
4,4'-DDD						
4,4'-DDT						
Methoxychlor						
TCX						
DCB						

EPA Sample No. (INDB3##): _____ Date Analyzed: _____

Lab Sample ID (INDB3): _____ Time Analyzed: _____

INDIVIDUAL MIX B COMPOUND	RT	RT WINDOW		$\overline{CF}$	CF	%D
		FROM	TO			
beta-BHC						
delta-BHC						
Aldrin						
Heptachlor epoxide						
4,4'-DDE						
Endosulfan II						
Endosulfan sulfate						
Endrin ketone						
Endrin aldehyde						
alpha-Chlordane						
gamma-Chlordane						
TCX						
DCB						

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

7L - FORM VII PEST-3
PESTICIDE CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column:_____ ID:_____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No. (PIBLK##): _____ Date Analyzed: _____

Lab Sample ID (PIBLK): _____ Time Analyzed: _____

EPA Sample No. (INDC3##): _____ Date Analyzed: _____

Lab Sample ID (INDC3): _____ Time Analyzed: _____

INDIVIDUAL MIX C COMPOUND	RT	RT WINDOW		$\overline{CF}$	CF	%D
		FROM	TO			
alpha-BHC						
gamma-BHC (Lindane)						
Heptachlor						
Endosulfan I						
Dieldrin						
Endrin						
4,4'-DDD						
4,4'-DDT						
Methoxychlor						
beta-BHC						
delta-BHC						
Aldrin						
Heptachlor epoxide						
4,4'-DDE						
Endosulfan II						
Endosulfan sulfate						
Endrin ketone						
Endrin aldehyde						
alpha-Chlordane						
gamma-Chlordane						
TCX						
DCB						

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

7M - FORM VII PEST-4
TOXAPHENE CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column:_____ ID:_____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No. (PIBLK##): _____ Date Analyzed: _____

Lab Sample ID (PIBLK): _____ Time Analyzed: _____

EPA Sample No. (TOXAPH3##): _____ Date Analyzed: _____

Lab Sample ID (TOXAPH3): _____ Time Analyzed: _____

COMPOUND	PEAK	RT	RT WINDOW		$\overline{CF}$	CF	%D
			FROM	TO			
TOXAPHENE	1						
	2						
	3						
	4						
	5						
TCX							
DCB							

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

7N - FORM VII ARO
AROCLOR CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column:_____ ID:_____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No. (AR####3##): _____ Date Analyzed: _____

Lab Sample ID: _____ Time Analyzed: _____

EPA Sample No. (AR####3##): _____ Date Analyzed: _____

Lab Sample ID: _____ Time Analyzed: _____

AROCLOR COMPOUND	PEAK	RETENTION	RT WINDOW		$\overline{CF}$	CF	%D
		RT	FROM	TO			
AR1016	1						
	2						
	3						
	4						
	5						
TCX							
DCB							
AR1260	1						
	2						
	3						
	4						
	5						
TCX							
DCB							
AR____	1						
	2						
	3						
	4						
	5						
TCX							
DCB							

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

8A - FORM VIII VOA
VOLATILE INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column:_____ ID:_____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No.(VSTD#####): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____ Heated Purge: (Y/N)_____

		IS1 (CBZ) AREA#	RT #	IS2 (DFB) AREA #	RT #	IS3 (DCB) AREA #	RT #
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
	EPA SAMPLE NO.						
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

IS1 (CBZ) = Chlorobenzene-d_5
IS2 (DFB) = 1,4-Difluorobenzene
IS3 (DCB) = 1,4-Dichlorobenzene-d_4

AREA UPPER LIMIT = 200% (Low-Medium Volatiles) and 140% (Trace Volatiles) of internal standard area
AREA LOWER LIMIT = 50% (Low-Medium Volatiles) and 60% (Trace Volatiles) of internal standard area
RT UPPER LIMIT = + 0.50 (Low-Medium Volatiles) and + 0.33 (Trace Volatiles) minutes of internal standard RT
RT LOWER LIMIT = - 0.50 (Low-Medium Volatiles) and - 0.33 (Trace Volatiles) minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.

8B - FORM VIII VOA-SIM
TRACE VOLATILE (WATER) SIM INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column: _____ ID: _____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No.(VSTD0.5##): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____ Heated Purge: (Y/N)_____

		IS1 (CBZ) AREA#	RT #	IS2 (DFB) AREA #	RT #	IS3 (DCB) AREA #	RT #
	12 HOUR STD						
	UPPER LIMIT						
	LOWER LIMIT						
	EPA SAMPLE NO.						
01							
02							
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

IS1 (CBZ) = Chlorobenzene-d_5
IS2 (DFB) = 1,4-Difluorobenzene
IS3 (DCB) = 1,4-Dichlorobenzene-d_4

AREA UPPER LIMIT = 140% of internal standard area
AREA LOWER LIMIT = 60% of internal standard area
RT UPPER LIMIT = + 0.33 minutes of internal standard RT
RT LOWER LIMIT = - 0.33 minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.

8C - FORM VIII SV-1
SEMIVOLATILE INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column: _____ ID: _____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No.(SSTD020##): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____

		IS1 (DCB) AREA	#	RT	#	IS2 (NPT) AREA	#	RT	#	IS3 (ANT) AREA	#	RT	#
	12 HOUR STD												
	UPPER LIMIT												
	LOWER LIMIT												
	EPA SAMPLE NO.												
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													

IS1 (DCB) = 1,4-Dichlorobenzene-d_4
IS2 (NPT) = Naphthalene-d_8
IS3 (ANT) = Acenaphthene-d_{10}

AREA UPPER LIMIT = 200% of internal standard area
AREA LOWER LIMIT = 50% of internal standard area
RT UPPER LIMIT = + 0.50 minutes of internal standard RT
RT LOWER LIMIT = - 0.50 minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.

8D - FORM VIII SV-2
SEMIVOLATILE INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

EPA Sample No.(SSTD020##): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____ GC Column:_____ ID:_____(mm)

		IS4 (PHN) AREA	#	RT	#	IS5 (CRY) AREA	#	RT	#	IS6 (PRY) AREA	#	RT	#
	12 HOUR STD												
	UPPER LIMIT												
	LOWER LIMIT												
	EPA SAMPLE NO.												
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													

IS4 (PHN) = Phenanthrene-d_{10}
IS5 (CRY) = Chrysene-d_{12}
IS6 (PRY) = Perylene-d_{12}

AREA UPPER LIMIT = 200% of internal standard area
AREA LOWER LIMIT = 50% of internal standard area
RT UPPER LIMIT = + 0.50 minutes of internal standard RT
RT LOWER LIMIT = - 0.50 minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.

8E - FORM VIII SV-SIM1
SEMIVOLATILE SIM INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column: _____ ID: _____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No.(SSTD0.4##): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____

		IS1 (DCB) AREA	#	RT	#	IS2 (NPT) AREA	#	RT	#	IS3 (ANT) AREA	#	RT	#
	12 HOUR STD												
	UPPER LIMIT												
	LOWER LIMIT												
	EPA SAMPLE NO.												
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													

IS1 (DCB) = 1,4-Dichlorobenzene-d_4
IS2 (NPT) = Naphthalene-d_8
IS3 (ANT) = Acenaphthene-d_{10}

AREA UPPER LIMIT = 200% of internal standard area
AREA LOWER LIMIT = 50% of internal standard area
RT UPPER LIMIT = + 0.50 minutes of internal standard RT
RT LOWER LIMIT = - 0.50 minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.

8F - FORM VIII SV-SIM2
SEMIVOLATILE SIM INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

EPA Sample No.(SSTD0.4##): _____ Date Analyzed: _____

Lab File ID (Standard): _____ Time Analyzed: _____

Instrument ID: _____ GC Column:_____ ID:_____(mm)

		IS4 (PHN) AREA	#	RT	#	IS5 (CRY) AREA	#	RT	#	IS6 (PRY) AREA	#	RT	#
	12 HOUR STD												
	UPPER LIMIT												
	LOWER LIMIT												
	EPA SAMPLE NO.												
01													
02													
03													
04													
05													
06													
07													
08													
09													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													

IS4 (PHN) = Phenanthrene-d_{10}
IS5 (CRY) = Chrysene-d_{12}
IS6 (PRY) = Perylene-d_{12}

AREA UPPER LIMIT = 200% of internal standard area
AREA LOWER LIMIT = 50% of internal standard area
RT UPPER LIMIT = + 0.50 minutes of internal standard RT
RT LOWER LIMIT = - 0.50 minutes of internal standard RT

Column used to flag values outside QC limits with an asterisk.

8G - FORM VIII PEST
PESTICIDE ANALYTICAL SEQUENCE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column:_____ ID:_____(mm) Init. Calib. Date(s): _____ _____

Instrument ID: _____

THE ANALYTICAL SEQUENCE OF BLANKS, SAMPLES, STANDARDS, MS/MSDs, and LCSs IS GIVEN BELOW:

	MEAN SURROGATE RT FROM INITIAL CALIBRATION TCX:_____ DCB:_____					
	EPA SAMPLE NO.	LAB File ID	DATE ANALYZED	TIME ANALYZED	TCX RT #	DCB RT #
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						

	QC LIMITS
TCX = Tetrachloro-m-xylene	(± 0.05 MINUTES)
DCB = Decachlorobiphenyl	(± 0.10 MINUTES)

Column used to flag RT values with an asterisk.

8H - FORM VIII ARO
AROCLOR ANALYTICAL SEQUENCE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GC Column:_____ ID:_____(mm) Init. Calib. Date(s): _____ _____

Instrument ID: _____

THE ANALYTICAL SEQUENCE OF BLANKS, SAMPLES, STANDARDS, MS/MSDs, and LCSs IS GIVEN BELOW:

	MEAN SURROGATE RT FROM INITIAL CALIBRATION TCX: _____ DCB: _____					
	EPA SAMPLE NO.	LAB FILE ID	DATE ANALYZED	TIME ANALYZED	TCX RT #	DCB RT #
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						

QC LIMITS

TCX = Tetrachloro-m-xylene (± 0.05 MINUTES)
DCB = Decachlorobiphenyl (± 0.10 MINUTES)

Column used to flag RT values with an asterisk.

9A - FORM IX PEST-1
PESTICIDE FLORISIL CARTRIDGE CHECK

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Florisil Cartridge Lot Number: _____ Date of Analysis: _____

GC Column: _____ ID: _____ (mm)

COMPOUND	SPIKE ADDED (ng)	SPIKE RECOVERED (ng)	%REC #	QC LIMITS
alpha-BHC				80-120
gamma-BHC (Lindane)				80-120
Heptachlor				80-120
Endosulfan I				80-120
Dieldrin				80-120
Endrin				80-120
4,4'-DDD				80-120
4,4'-DDT				80-120
Methoxychlor				80-120
TCX				80-120
DCB				80-120
2,4,5-Trichlorophenol				<5

Column to be used to flag recovery with an asterisk.

THIS CARTRIDGE LOT APPLIES TO THE FOLLOWING SAMPLES, BLANKS, LCSs, AND MS/MSDs:

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	DATE ANALYZED 2
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				

TCX = Tetrachloro-m-xylene
DCB = Decachlorobiphenyl

9B - FORM IX PEST-2
PESTICIDE GPC CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

GPC Column: _____ Calibration Verification Date: _____

GC Column: _____ ID: _____ (mm)

COMPOUND	SPIKE ADDED (ng)	SPIKE RECOVERED (ng)	%REC #	QC LIMITS
gamma-BHC (Lindane)				80-110
Heptachlor				80-110
Aldrin				80-110
Dieldrin				80-110
Endrin				80-110
4,4'-DDT				80-110

Column to be used to flag recovery with an asterisk.

THIS GPC CALIBRATION VERIFICATION APPLIES TO THE FOLLOWING SAMPLES, BLANKS, LCSs, AND MS/MSDs:

	EPA SAMPLE NO.	LAB SAMPLE ID	GPC CLEANUP DATE
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			

10A - FORM X PEST-1
IDENTIFICATION SUMMARY
FOR SINGLE COMPONENT ANALYTES

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab Sample ID: _____ Date(s) Analyzed: _____

Instrument ID (1): _____ Instrument ID (2):_____

GC Column (1): _____ ID: _____(mm) GC Column (2): _____ ID: _____(mm)

ANALYTE	COL	RT	RT WINDOW		CONCENTRATION	%D
			FROM	TO		
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					
	1					
	2					

10B - FORM X PEST-2
IDENTIFICATION SUMMARY
FOR TOXAPHENE

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref No.: _____ SDG No.: _____

Lab Sample ID: _____ Date(s) Analyzed: _____

Instrument ID (1): _____ Instrument ID (2):_____

GC Column (1): _____ ID: _____(mm) GC Column (2): _____ ID: _____(mm)

ANALYTE	PEAK	RT	RT WINDOW FROM	RT WINDOW TO	CONCENTRATION PEAK	CONCENTRATION MEAN	%D
COLUMN 1	1						
	2						
	3						
	4						
	5						
COLUMN 2	1						
	2						
	3						
	4						
	5						

At least three peaks for each column are required for identification of multicomponent analytes.

10C - FORM X ARO
IDENTIFICATION SUMMARY
FOR MULTICOMPONENT ANALYTES

EPA SAMPLE NO.

Lab Name: ____________________________ Contract: _________________________

Lab Code: _______ Case No.: ______ Mod. Ref No.: _________ SDG No.: ____________

Lab Sample ID: _______________________ Date(s) Analyzed: _________________

Instrument ID (1): ___________________ Instrument ID (2): ________________

GC Column (1): ________ ID: _______(mm) GC Column (2): ______ ID: _____(mm)

			RT WINDOW		CONCENTRATION		
ANALYTE	PEAK	RT	FROM	TO	PEAK	MEAN	%D
______________	1						
	2						
COLUMN 1	3						
	4						
	5						
	1						
	2						
COLUMN 2	3						
	4						
	5						
______________	1						
	2						
COLUMN 1	3						
	4						
	5						
	1						
	2						
COLUMN 2	3						
	4						
	5						
______________	1						
	2						
COLUMN 1	3						
	4						
	5						
	1						
	2						
COLUMN 2	3						
	4						
	5						

At least three peaks for each column are required for identification of multicomponent analytes.

SAMPLE LOG-IN SHEET
FORM DC-1

Lab Name					Page __ of __
Received By (Print Name)					Log-in Date
Received By (Signature)					
Case Number		Sample Delivery Group No.			Mod. Ref. No.
Remarks:			Corresponding		
		EPA Sample #	Sample Tag #	Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____				
	_____				
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists	Present/Absent*				
4. Airbill	Airbill/Sticker Present/Absent*				
5. Airbill No.	_____				
	_____				
6. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody				
7. Sample Condition	Intact/Broken*/ Leaking				
8. Cooler Temperature Indicator Bottle	Present/Absent				
9. Cooler Temperature	_____				
10. Does information on TR/COCs and sample tags agree?	Yes/No*				
11. Date Received at Laboratory	_____				
12. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution.

Reviewed By	Logbook No.
Date	Logbook Page No.

ORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET
FORM DC-2

LABORATORY NAME _____

CITY/STATE _____

CASE NO. _____ SDG NO. _____

SDG NOs. TO FOLLOW _____ _____ _____

MOD. REF. NO. _____

CONTRACT NO. _____

SOW NO. _____

All documents delivered in the Complete SDG File (CSF) must be original documents where possible.

	PAGE NOs		CHECK	
	FROM	TO	LAB	USEPA
1. **Inventory Sheet** (Form DC-2) (Do not number)				
2. **SDG Case Narrative**				
3. **SDG Cover Sheet/Traffic Report**				
4. **Trace Volatiles Data**				
a. QC Summary				
Deuterated Monitoring Compound Recovery (Form II VOA-1 and VOA-2)				
Matrix Spike/Matrix Spike Duplicate Recovery (Form III VOA) (if requested by USEPA Region)				
Method Blank Summary (Form IV VOA)				
GC/MS Instrument Performance Check (Form V VOA)				
Internal Standard Area and RT Summary (Form VIII VOA)				
b. Sample Data				
TCL Results - Organics Analysis Data Sheet (Form I VOA-1 and VOA-2)				
Tentatively Identified Compounds (Form I VOA-TIC)				
Reconstructed total ion chromatograms (RIC) for each sample				
For each sample:				
Raw Spectra and background-subtracted mass spectra of target compounds identified				
Quantitation reports				
Mass Spectra of all reported TICs with three best library matches				
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI VOA-1, VOA-2, VOA-3)				
RICs and Quantitation Reports for all Standards				
Continuing Calibration Data (Form VII VOA-1, VOA-2, VOA-3)				
RICs and Quantitation Reports for all Standards				
d. Raw/Quality Control (QC) Data				
BFB				
Blank Data				

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ MOD. REF. NO. _____

	PAGE NOs		CHECK	
	FROM	TO	LAB	USEPA
Matrix Spike/Matrix Spike Duplicate Data (if requested by USEPA Region)				
e. Trace SIM Data (Place at the end of the Trace Volatiles Section)				
[Form I VOA-SIM; Form II VOA-SIM1 and VOA-SIM2; Form IV-VOA-SIM; Form VI VOA-SIM; Form VII VOA-SIM; Form VIII VOA-SIM; and all raw data for QC, Samples, and Standards.]				
5. **Low/Med Volatiles Data**				
a. QC Summary				
Deuterated Monitoring Compound Recovery (Form II VOA-1, VOA-2, VOA-3, VOA-4)				
Matrix Spike/Matrix Spike Duplicate Recovery (Form III VOA-1 and VOA-2) (if requested by USEPA Region)				
Method Blank Summary (Form IV VOA)				
GC/MS Instrument Performance Check (Form V VOA)				
Internal Standard Area and RT Summary (Form VIII VOA)				
b. Sample Data				
TCL Results - Organics Analysis Data Sheet (Form I VOA-1 and VOA-2)				
Tentatively Identified Compounds (Form I VOA-TIC)				
Reconstructed total ion chromatograms (RIC) for each sample				
For each sample:				
Raw Spectra and background-subtracted mass spectra of target compounds identified				
Quantitation reports				
Mass Spectra of all reported TICs with three best library matches				
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI VOA-1, VOA-2, VOA-3)				
RICs and Quantitation Reports for all Standards				
Continuing Calibration Data (Form VII VOA-1, VOA-2, VOA-3)				
RICs and Quantitation Reports for all Standards				
d. Raw/Quality Control (QC) Data				
BFB				
Blank Data				

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ MOD. REF. NO. _____

	PAGE NOs		CHECK	
	FROM	TO	LAB	USEPA
Martix Spike/Matrix Spike Duplicate Data (if requested by USEPA Region)				
6. **Semivolatiles Data**				
a. QC Summary				
Deuterated Monitoring Compound Recovery (Form II SV-1, SV-2, SV-3, SV-4)				
Matrix Spike/Matrix Spike Duplicate Recovery Summary (Form III SV-1 and SV-2) (if requested by USEPA Region)				
Method Blank Summary (Form IV SV)				
GC/MS Instrument Performance Check (Form V SV)				
Internal Standard Area and RT Summary (Form VIII SV-1 and SV-2)				
b. Sample Data				
TCL Results - Organics Analysis Data Sheet (Form I SV-1 and SV-2)				
Tentatively Identified Compounds (Form I SV-TIC)				
Reconstructed total ion chromatograms (RICs) for each sample				
For each sample:				
Raw Spectra and background-subtracted mass spectra of target compounds				
Quantitation reports				
Mass Spectra of TICs with three best library matches				
GPC chromatograms (if GPC is required)				
c. Standards Data (All Instruments)				
Initial Calibration Data (Form VI SV-1, SV-2, SV-3)				
RICs and Quantitation Reports for all Standards				
Continuing Calibration Data (Form VII SV-1, SV-2, SV-3)				
RICs and Quantitation Reports for all Standards				
d. Raw QC Data				
DFTPP				
Blank Data				
MS/MSD Data (if requested by USEPA Region)				
e. Raw GPC Data				

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ MOD. REF. NO. _____

	PAGE NOs		CHECK	
	FROM	TO	LAB	USEPA
f. Semivolatile SIM Data				
[Form I SV-SIM; Form II SV-SIM1 and SV-SIM2; Form III SV-SIM1 and SV-SIM2 (if required); Form IV SV-SIM; Form VI SV-SIM; Form VII SV-SIM; Form VIII SV-SIM1 and SV-SIM2; and all raw data for QC, Samples, and Standards.]				
7. **Pesticides Data**				
a. QC Summary				
Surrogate Recovery Summary (Form II PEST-1 and PEST-2)				
Matrix Spike/Matrix Spike Duplicate Recovery Summary (Form III PEST-1 and PEST-2)				
Laboratory Control Sample Recovery (Form III PEST-3 and PEST-4)				
Method Blank Summary (Form IV PEST)				
b. Sample Data				
TCL Results - Organics Analysis Data Sheet (Form I PEST)				
Chromatograms (Primary Column)				
Chromatograms from second GC column confirmation				
GC Integration report or data system printout				
Manual work sheets				
For pesticides by GC/MS				
Copies of raw spectra and copies of background-subtracted mass spectra of target compounds (samples & standards)				
c. Standards Data				
Initial Calibration of Single Component Analytes (Form VI PEST-1 and PEST-2)				
Toxaphene Initial Calibration (Form VI PEST-3 and PEST-4)				
Analyte Resolution Summary (Form VI PEST-5, per column)				
Performance Evaluation Mixture (Form VI PEST-6)				
Individual Standard Mixture A (Form VI PEST-7)				
Individual Standard Mixture B (Form VI PEST-8)				
Individual Standard Mixture C (Form VI PEST-9 and PEST-10)				
Calibration Verification Summary (Form VII PEST-1)				
Calibration Verification Summary (Form VII PEST-2)				
Calibration Verification Summary (Form VII PEST-3)				

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ MOD. REF. NO. _____

	PAGE NOs		CHECK	
	FROM	TO	LAB	USEPA
Calibration Verification Summary (Form VII PEST-4)				
Analytical Sequence (Form VIII PEST)				
Florisil Cartridge Check (Form IX PEST-1)				
Pesticide GPC Calibration (Form IX PEST-2)				
Identification Summary for Single Component Analytes (Form X PEST-1)				
Identification Summary for Toxaphene (Form X PEST-2)				
Chromatograms and data system printouts A printout of Retention Times and corresponding peak areas or peak heights				
d. Raw QC Data				
Blank Data				
Matrix Spike/Matrix Spike Duplicate Data				
Laboratory Control Sample Data				
e. Raw GPC Data				
f. Raw Florisil Data				
8. **Aroclor Data**				
a. QC Summary				
Surrogate Recovery Summary (Form II ARO-1 and ARO-2)				
Matrix Spike/Matrix Spike Duplicate Summary (Form III ARO-1 and ARO-2)				
Laboratory Control Sample Recovery(Form III ARO-3 and ARO-4)				
Method Blank Summary (Form IV ARO)				
b. Sample Data				
TCL Results - Organics Analysis Data Sheet (Form I ARO)				
Chromatograms (Primary Column)				
Chromatograms from second GC column confirmation				
GC Integration report or data system printout				
Manual work sheets				
For Aroclors by GC/MS				
Copies of raw spectra and copies of background-subtracted mass spectra of target compounds (samples & standards)				

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ MOD. REF. NO. _____

	PAGE NOs		CHECK	
	FROM	TO	LAB	USEPA
c. Standards Data				
Aroclors Initial Calibration (Form VI ARO-1, ARO-2, and ARO-3)				
Calibration Verification Summary (Form VII ARO-1)				
Analytical Sequence (Form VIII ARO)				
Identification Summary for Multicomponent Analytes (Form X ARO)				
Chromatograms and data system printouts A printout of Retention Times and corresponding peak areas or peak heights				
d. Raw QC Data				
Blank Data				
Matrix Spike/Matrix Spike Duplicate Data				
Laboratory Control Sample (LCS) Data				
e. Raw GPC Data (if performed)				
9. **Miscellaneous Data**				
Original preparation and analysis forms or copies of preparation and analysis logbook pages				
Internal sample and sample extract transfer chain-of-custody records				
Screening records				
All instrument output, including strip charts from screening activities (describe or list)				
_____				
_____				
10. **EPA Shipping/Receiving Documents**				
Airbills (No. of shipments _____)				
Chain of Custody Records				
Sample Tags				
Sample Log-in Sheet (Lab & DC-1)				
Miscellaneous Shipping/Receiving Records (describe or list)				
_____				
_____				

CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____

_____ _____ MOD. REF. NO. _____

	PAGE NOs		CHECK	
	FROM	TO	LAB	USEPA
11. **Internal Lab Sample Transfer Records and Tracking Sheets** (describe or list)				
_____				
_____				
12. **Other Records** (describe or list)				
Telephone Communication Log				
_____				
_____				

13. **Comments**

_____

_____

_____

Completed by: (CLP Lab)	_____ (Signature)	_____ (Printed Name/Title)	_____ (Date)
Verified by: (CLP Lab)	_____ (Signature)	_____ (Printed Name/Title)	_____ (Date)
Audited by: (USEPA)	_____ (Signature)	_____ (Printed Name/Title)	_____ (Date)

EXHIBIT C

TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

NOTE: Specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

The Contract Required Quantitation Limit (CRQL) values listed on the following pages are based on the analysis of samples according to the specifications given in Exhibit D.

For soil samples, the moisture content of the samples must be used to adjust the CRQL values appropriately.

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Exhibit C - Target Compound List and Contract Required Quantitation Limits

Table of Contents

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1.0 VOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

	Volatiles	CAS Number	Quantitation Limits: Trace Water By SIM µg/L	Trace Water µg/L	Low Water µg/L	Low Soil µg/kg	Med. Soil µg/kg
1.	Dichlorodifluoromethane	75-71-8		0.50	5.0	5.0	250
2.	Chloromethane	74-87-3		0.50	5.0	5.0	250
3.	Vinyl chloride	75-01-4		0.50	5.0	5.0	250
4.	Bromomethane	74-83-9		0.50	5.0	5.0	250
5.	Chloroethane	75-00-3		0.50	5.0	5.0	250
6.	Trichlorofluoromethane	75-69-4		0.50	5.0	5.0	250
7.	1,1-Dichloroethene	75-35-4		0.50	5.0	5.0	250
8.	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1		0.50	5.0	5.0	250
9.	Acetone	67-64-1		5.0	10	10	500
10.	Carbon disulfide	75-15-0		0.50	5.0	5.0	250
11.	Methyl acetate	79-20-9		0.50	5.0	5.0	250
12.	Methylene chloride	75-09-2		0.50	5.0	5.0	250
13.	trans-1,2-Dichloroethene	156-60-5		0.50	5.0	5.0	250
14.	Methyl tert-butyl ether	1634-04-4		0.50	5.0	5.0	250
15.	1,1-Dichloroethane	75-34-3		0.50	5.0	5.0	250
16.	cis-1,2-Dichloroethene	156-59-2		0.50	5.0	5.0	250
17.	2-Butanone	78-93-3		5.0	10	10	500
18.	Bromochloromethane	74-97-5		0.50	5.0	5.0	250
19.	Chloroform	67-66-3		0.50	5.0	5.0	250
20.	1,1,1-Trichloroethane	71-55-6		0.50	5.0	5.0	250
21.	Cyclohexane	110-82-7		0.50	5.0	5.0	250
22.	Carbon tetrachloride	56-23-5		0.50	5.0	5.0	250
23.	Benzene	71-43-2		0.50	5.0	5.0	250
24.	1,2-Dichloroethane	107-06-2		0.50	5.0	5.0	250
25.	1,4-Dioxane	123-91-1	2.0	20	100	100	5000
26.	Trichloroethene	79-01-6		0.50	5.0	5.0	250
27.	Methylcyclohexane	108-87-2		0.50	5.0	5.0	250
28.	1,2-Dichloropropane	78-87-5		0.50	5.0	5.0	250
29.	Bromodichloromethane	75-27-4		0.50	5.0	5.0	250
30.	cis-1,3-Dichloropropene	10061-01-5		0.50	5.0	5.0	250
31.	4-Methyl-2-pentanone	108-10-1		5.0	10	10	500
32.	Toluene	108-88-3		0.50	5.0	5.0	250
33.	trans-1,3-Dichloropropene	10061-02-6		0.50	5.0	5.0	250
34.	1,1,2-Trichloroethane	79-00-5		0.50	5.0	5.0	250
35.	Tetrachloroethene	127-18-4		0.50	5.0	5.0	250

1.0 VOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (Con't)

Volatiles		CAS Number	Quantitation Limits: Trace Water By SIM µg/L	Trace Water µg/L	Low Water µg/L	Low Soil µg/kg	Med. Soil µg/kg
36.	2-Hexanone	591-78-6		5.0	10	10	500
37.	Dibromochloromethane	124-48-1		0.50	5.0	5.0	250
38.	1,2-Dibromoethane	106-93-4	0.050	0.50	5.0	5.0	250
39.	Chlorobenzene	108-90-7		0.50	5.0	5.0	250
40.	Ethylbenzene	100-41-4		0.50	5.0	5.0	250
41.	o-Xylene	95-47-6		0.50	5.0	5.0	250
42.	m,p-Xylene	179601-23-1		0.50	5.0	5.0	250
43.	Styrene	100-42-5		0.50	5.0	5.0	250
44.	Bromoform	75-25-2		0.50	5.0	5.0	250
45.	Isopropylbenzene	98-82-8		0.50	5.0	5.0	250
46.	1,1,2,2-Tetrachloroethane	79-34-5		0.50	5.0	5.0	250
47.	1,3-Dichlorobenzene	541-73-1		0.50	5.0	5.0	250
48.	1,4-Dichlorobenzene	106-46-7		0.50	5.0	5.0	250
49.	1,2-Dichlorobenzene	95-50-1		0.50	5.0	5.0	250
50.	1,2-Dibromo-3-chloropropane	96-12-8	0.050	0.50	5.0	5.0	250
51.	1,2,4-Trichlorobenzene	120-82-1		0.50	5.0	5.0	250
52.	1,2,3-Trichlorobenzene	87-61-6		0.50	5.0	5.0	250

2.0 SEMIVOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

			Quantitation Limits				
			Low Water By SIM[1]	Low Water	Low Soil By SIM[1]	Low Soil	Med. Soil
	Semivolatiles	CAS Number	µg/L	µg/L	µg/kg	µg/kg	µg/kg
53.	Benzaldehyde	100-52-7		5.0		170	5000
54.	Phenol	108-95-2		5.0		170	5000
55.	Bis(2-chloroethyl) ether	111-44-4		5.0		170	5000
56.	2-Chlorophenol	95-57-8		5.0		170	5000
57.	2-Methylphenol	95-48-7		5.0		170	5000
58.	2,2'-Oxybis(1-chloropropane)[2]	108-60-1		5.0		170	5000
59.	Acetophenone	98-86-2		5.0		170	5000
60.	4-Methylphenol	106-44-5		5.0		170	5000
61.	N-Nitroso-di-n propylamine	621-64-7		5.0		170	5000
62.	Hexachloroethane	67-72-1		5.0		170	5000
63.	Nitrobenzene	98-95-3		5.0		170	5000
64.	Isophorone	78-59-1		5.0		170	5000
65.	2-Nitrophenol	88-75-5		5.0		170	5000
66.	2,4-Dimethylphenol	105-67-9		5.0		170	5000
67.	Bis(2-chloroethoxy) methane	111-91-1		5.0		170	5000
68.	2,4-Dichlorophenol	120-83-2		5.0		170	5000
69.	Naphthalene	91-20-3	0.10	5.0	3.3	170	5000
70.	4-Chloroaniline	106-47-8		5.0		170	5000
71.	Hexachlorobutadiene	87-68-3		5.0		170	5000
72.	Caprolactam	105-60-2		5.0		170	5000
73.	4-Chloro-3-methylphenol	59-50-7		5.0		170	5000
74.	2-Methylnaphthalene	91-57-6	0.10	5.0	3.3	170	5000
75.	Hexachlorocyclo-pentadiene	77-47-4		5.0		170	5000
76.	2,4,6-Trichlorophenol	88-06-2		5.0		170	5000
77.	2,4,5-Trichlorophenol	95-95-4		5.0		170	5000
78.	1,1'-Biphenyl	92-52-4		5.0		170	5000

[1]CRQLs for optional analysis of water and soil samples using SIM technique for PAHs and phenols.

[2]Previously known as Bis(2-chloroisopropyl)ether.

2.0 SEMIVOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (Con't)

			Quantitation Limits				
			Low Water By SIM[1]	Low Water	Low Soil By SIM[1]	Low Soil	Med. Soil
	Semivolatiles	CAS Number	µg/L	µg/L	µg/kg	µg/kg	µg/kg
79.	2-Chloronaphthalene	91-58-7		5.0		170	5000
80.	2-Nitroaniline	88-74-4		10		330	10000
81.	Dimethylphthalate	131-11-3		5.0		170	5000
82.	2,6-Dinitrotoluene	606-20-2		5.0		170	5000
83.	Acenaphthylene	208-96-8	0.10	5.0	3.3	170	5000
84.	3-Nitroaniline	99-09-2		10		330	10000
85.	Acenaphthene	83-32-9	0.10	5.0	3.3	170	5000
86.	2,4-Dinitrophenol	51-28-5		10		330	10000
87.	4-Nitrophenol	100-02-7		10		330	10000
88.	Dibenzofuran	132-64-9		5.0		170	5000
89.	2,4-Dinitrotoluene	121-14-2		5.0		170	5000
90.	Diethylphthalate	84-66-2		5.0		170	5000
91.	Fluorene	86-73-7	0.10	5.0	3.3	170	5000
92.	4-Chlorophenyl-phenyl ether	7005-72-3		5.0		170	5000
93.	4-Nitroaniline	100-01-6		10		330	10000
94.	4,6-Dinitro-2-methylphenol	534-52-1		10		330	10000
95.	N-Nitrosodiphenylamine	86-30-6		5.0		170	5000
96.	1,2,4,5-Tetra chlorobenzene	95-94-3		5.0		170	5000
97.	4-Bromophenyl-phenylether	101-55-3		5.0		170	5000
98.	Hexachlorobenzene	118-74-1		5.0		170	5000
99.	Atrazine	1912-24-9		5.0		170	5000
100.	Pentachlorophenol	87-86-5	0.20	10	6.7	330	10000
101.	Phenanthrene	85-01-8	0.10	5.0	3.3	170	5000
102.	Anthracene	120-12-7	0.10	5.0	3.3	170	5000
103.	Carbazole	86-74-8		5.0		170	5000
104.	Di-n-butylphthalate	84-74-2		5.0		170	5000
105.	Fluoranthene	206-44-0	0.10	5.0	3.3	170	5000
106.	Pyrene	129-00-0	0.10	5.0	3.3	170	5000
107.	Butylbenzylphthalate	85-68-7		5.0		170	5000

[1]CRQLs for optional analysis of water and soil samples using SIM technique for PAHs and phenols.

2.0 SEMIVOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (Con't)

			Quantitation Limits				
			Low Water By SIM[1]	Low Water	Low Soil By SIM[1]	Low Soil	Med. Soil
Semivolatiles		CAS Number	µg/L	µg/L	µg/kg	µg/kg	µg/kg
108.	3,3'-Dichlorobenzidine	91-94-1		5.0		170	5000
109.	Benzo(a)anthracene	56-55-3	0.10	5.0	3.3	170	5000
110.	Chrysene	218-01-9	0.10	5.0	3.3	170	5000
111.	Bis(2-ethylhexyl) phthalate	117-81-7		5.0		170	5000
112.	Di-n-octylphthalate	117-84-0		5.0		170	5000
113.	Benzo(b)fluoranthene	205-99-2	0.10	5.0	3.3	170	5000
114.	Benzo(k)fluoranthene	207-08-9	0.10	5.0	3.3	170	5000
115.	Benzo(a)pyrene	50-32-8	0.10	5.0	3.3	170	5000
116.	Indeno(1,2,3-cd) pyrene	193-39-5	0.10	5.0	3.3	170	5000
117.	Dibenzo(a,h)anthracene	53-70-3	0.10	5.0	3.3	170	5000
118.	Benzo(g,h,i)perylene	191-24-2	0.10	5.0	3.3	170	5000
119.	2,3,4,6-Tetrachlorophenol	58-90-2		5.0		170	5000

[1]CRQLs for optional analysis of water and soil samples using SIM technique for PAHs and pentachlorophenol.

3.0 PESTICIDES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[1]

Pesticides	CAS Number	Quantitation Limits Water µg/L	Quantitation Limits Soil µg/kg
120. alpha-BHC	319-84-6	0.050	1.7
121. beta-BHC	319-85-7	0.050	1.7
122. delta-BHC	319-86-8	0.050	1.7
123. gamma-BHC (Lindane)	58-89-9	0.050	1.7
124. Heptachlor	76-44-8	0.050	1.7
125. Aldrin	309-00-2	0.050	1.7
126. Heptachlor epoxide[2]	1024-57-3	0.050	1.7
127. Endosu1fan I	959-98-8	0.050	1.7
128. Dieldrin	60-57-1	0.10	3.3
129. 4,4'-DDE	72-55-9	0.10	3.3
130. Endrin	72-20-8	0.10	3.3
131. Endosulfan II	33213-65-9	0.10	3.3
132. 4,4'-DDD	72-54-8	0.10	3.3
133. Endosulfan sulfate	1031-07-8	0.10	3.3
134. 4,4'-DDT	50-29-3	0.10	3.3
135. Methoxychlor	72-43-5	0.50	17
136. Endrin ketone	53494-70-5	0.10	3.3
137. Endrin aldehyde	7421-93-4	0.10	3.3
138. alpha-Chlordane	5103-71-9	0.050	1.7
139. gamma-Chlordane	5103-74-2	0.050	1.7
140. Toxaphene	8001-35-2	5.0	170

[1]There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of pesticides.

[2]Only the exo-epoxy isomer (isomer B) of heptachlor epoxide is reported on the data reporting forms (Exhibit B).

4.0 AROCLORS TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[1]

Aroclors	CAS Number	Quantitation Limits Water µg/L	Quantitation Limits Soil µg/kg
141. Aroclor-1016	12674-11-2	1.0	33
142. Aroclor-1221	11104-28-2	1.0	33
143. Aroclor-1232	11141-16-5	1.0	33
144. Aroclor-1242	53469-21-9	1.0	33
145. Aroclor-1248	12672-29-6	1.0	33
146. Aroclor-1254	11097-69-1	1.0	33
147. Aroclor-1260	11096-82-5	1.0	33
148. Aroclor-1262	37324-23-5	1.0	33
149. Aroclor-1268	11100-14-4	1.0	33

[1]There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of Aroclors.

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APPENDIX C

WESTON Procured Subcontractor Laboratory SOPs, Reporting, Accuracy, and Precision Limits

Analytical Method Details - TriMatrix Laboratories, Inc.

Method	Analyte	MDL	MRL	Units	Surr. %R	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike RPD	CAS #
Extractable Petroleum Hydrocarbons by EPA Method 8015B in Soil/Sediment										
USEPA-8015B	DRO - 8015B (C10-C28)	1.3	6.7	mg/kg dry wt	-	30-141	20	44-135	20	
USEPA-8015B	Oil Range Organics (C28-C36)	4.4	10	mg/kg dry wt	-	50-150	20	50-150	20	
USEPA-8015B	o-Terphenyl			Surrogate	44-137	-	-	-	-	84-15-1

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Diesel Range Organics (DRO) Extraction

SW-846 Method 8015C
SW-846 Method 3550C

APPROVALS:

Area Supervisor: Andrea Colborn (Andrea S. Colborn) Date: 10/2/08

QA Officer: (Tom C. Boocher) Date: 10-2-08

Operations Manager: (Jeff P. Glaser) Date: 10/7/08

Procedure Number: GR-09-123
Revision Number: 0.2

Date Initiated: 4/19/01
Effective Date: 10/15/08

Date Revised: 4/25/08
Pages Revised: All

By: Andrea S. Colborn

Total Number of Pages: 15

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4/16/10	Brian Hall	4/16/11

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the extraction of diesel range organics from soil, sediment, sludge or waste samples as diesel range organics (DRO).

1.2 The DRO corresponds to any component that elutes between decane (C_{10}) and octacosane (C_{28}) with an approximate boiling point range of 170 – 430° C.

1.3 This procedure corresponds to the low-concentration technique in the referenced extraction method.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update IV, Revision 3, February, 2007, Method 8015C, Nonhalogenated Organics by Gas Chromatography

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, Ultrasonic Extraction

3.0 SUMMARY OF PROCEDURE

3.1 A 30 g sample aliquot is mixed with sodium sulfate to form a free-flowing powder then spiked with surrogate. The mixture is extracted with solvent three times, using ultrasonic extraction.

3.2 The combined extract is separated from the sample by filtration and/or centrifugation.

3.3 The extract is dried and concentrated to a final volume of 1.0 mL.

3.4 The concentrate is then ready for DRO analysis by gas chromatography (GC) using a flame ionization detector (FID).

4.0 PARAMETER OR COMPOUND LIST

4.1 Diesel Range Organics (DRO)

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration*, latest revision

5.2 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.3 TriMatrix SOP GR-03-101, *Semi-Volatiles Laboratory Quality Control Corrective Actions*, latest revision

5.4 TriMatrix SOP GR-10-125, *Method Detection Limit*, latest revision

5.5 TriMatrix SOP GR-03-122, *Diesel Range Organics (DRO)*, latest revision

5.6 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

5.7 TriMatrix SOP GR-09-128, *Mixing and Grinding Samples for Organic Extractions*, latest revision

5.8 TriMatrix SOP GR-09-103, *Extraction of BNA Semi-Volatiles from Soil, Sediment and Sludge*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Organic compounds including animal or vegetable oil/grease, chlorinated hydrocarbons, phenols, phthalate esters and motor oils can have components that elute within the DRO window. This is addressed in TriMatrix SOP GR-03-122.

6.2 Interferences can be from contaminants in solvents, reagents, glassware and other sample processing equipment.

6.2.1 Rinse washed glassware with methylene chloride just prior to use.

6.2.2 Use only pesticide grade (or better) solvents.

6.2.3 Rinse sodium sulfate with methylene chloride before use.

6.2.4 Extraction blanks must be extracted with samples. Refer to Section 15.0.

6.3 Matrix and/or laboratory-induced interferences can affect analyte and/or surrogate spike concentrations. Sample re-extraction may be necessary if quality control criteria are exceeded.

6.4 Use only PTFE dispensing bottles for solvent rinsing. Do not use plastic equipment for this procedure.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses when in the extractions laboratory. Wear disposable gloves whenever samples or reagents are handled.

7.2 Follow all safety instructions as outlined in the TriMatrix laboratory safety manual and chemical hygiene plan.

7.3 For proper spill response and waste disposal, refer to TriMatrix GR-15-102.

7.4 All chemicals in the laboratory must be treated as a potential health hazard. Reduce exposure to the lowest possible level.

7.5 Material safety data sheets (MSDS) are available on the laboratory intranet library from the extraction laboratory office computer.

7.6 NO solvents or solvent work is to be done in the extraction laboratory office. Chemicals are not permitted in laboratory offices.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Collect samples in screw-cap glass jars (60 mL, 125 mL or 250 mL) with PTFE-lined lids.

8.2 Handle sample containers with care to avoid breakage.

8.3 Store samples in the walk-in cooler at 4 ±2° C when not in use.

8.4 Extract samples within 14 days of the collection date. Extracts must be analyzed within 40 days of the extraction date.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Heavy duty Pyrex beakers, 400 mL and 600 mL

9.2 500 mL Kuderna-Danish evaporation flasks

9.3 10 mL graduated concentrator tubes

9.4 Snyder columns, 3-ball macro and 2-ball micro

9.5 Variable temperature steam bath in the fume hood

9.6 Analytical balance, capable of accurately weighing to the nearest 0.1 mg

9.7 Filter paper, qualitative, Fisher P8, VWR 415 or Whatman 41

9.8 Tongue depressors, wooden

9.9 Disposable glass Pasteur pipettes, 2 mL

9.10 Methylene chloride-rinsed boiling chips, approximately 10/40 mesh (PTFE) or equivalent

9.11 2, 10, 15, and 40 mL vials with PTFE-lined screw cap lids

9.12 Hamilton microsyringes, gastight, 25, 100, 500, 1000 µL

9.13 Filter funnels for drying extracts before concentration, glass

9.14 Erlenmeyer flasks, 250 and 500 mL

9.15 Top-loading balance, capable of accurately weighing to the nearest 0.1 g

9.16 Ultrasonic dismembrators, 2 units, with 3/4 inch horn

9.17 Sonabox for each dismembrator unit

9.18 Centrifuge, for extracts with fine particles that pass through the filter paper

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Tune the ultrasonic dismembrator at the beginning of each work shift.

10.2 Clean the disrupter horn before each sample.

10.3 Perform a thorough cleaning of all equipment at the end of the work shift.

10.4 Clean glassware in accordance with TriMatrix SOP GR-16-100.

11.0 CHEMICALS AND REAGENTS

11.1 Sodium sulfate (granular, anhydrous), Na_2SO_4, ACS

Note: Extract a blank for each lot of sodium sulfate before use to demonstrate that there is no interference above the method detection limit (MDL) from the sodium sulfate. If interferences are detected, place in a muffle furnace at 400° C for 4 hours and retest successfully before using.

11.2 Methanol, pesticides grade or better

11.3 Methylene chloride, pesticides grade or better

11.4 o-Terphenyl, certified purity greater than 95%

11.5 Laboratory reagent water, Milli-Q system, pre-extracted with extraction solvent before use

12.0 STANDARDS PREPARATION

12.1 Input all spiking standards data into the laboratory information management system (Element™) as individual solutions.

12.2 The expiration of working dilutions is 6 months from the date prepared if stored without headspace at 4 ±2°C.

12.3 The expiration of stock solutions prepared from neat materials or purchased as certified standards is 1 year from the date prepared or opened if stored without headspace at 4 ±2°C.

12.4 A prepared standard expiration date is not to exceed any manufacturer's expiration date.

12.5 Dispose of expired standards appropriately.

12.6 Monitor the inventory and expiration date of all stock standards to avoid expedited shipping fees.

12.7 Store surrogate/spiking standards in 40 mL amber vials labeled with the following information:

12.7.1 Standard name

12.7.2 Identification number

12.7.3 Date prepared

12.7.4 Analyst initials

12.7.5 Solvent

12.7.6 Concentration with units

12.7.7 Expiration date

12.8 Store all surrogate/spiking standards in the extractions laboratory refrigerator at 4 ±2° C. Remake any standard solution containing less than 5 mL, that shows evidence of evaporation or degradation, or that fails quality control limits.

12.9 Dispose of standard solutions appropriately.

12.10 All standard preparation calculations are verified and documented by Element™. Refer to Attachment 20.2.

12.11 All prepared standards must be verified and documented by analysis before using.

12.11.1 Prepare a dilution of the standard at the same concentration to be used.

12.11.2 Give to the analysis laboratory.

12.11.3 Review results after analysis to verify the concentration using default 80-120% acceptance limits. Results should be well within the acceptance limits. If not acceptable or if the analyst suspects a problem exists, remake the standard successfully and repeat the analysis check.

12.11.4 If results indicate the standard was prepared correctly and accurately, it may be used for sample extractions and/or analysis.

12.12 Diesel fuel #2 is purchased as a certified 50,000 ug/mL stock standard in methylene chloride for quality control spiking. This standard is not used for instrument calibration.

12.13 Prepare a 2,000 ug/mL DRO blank spike (BS) and matrix spike (MS) spiking solution in methylene chloride from the 50,000 ug/mL stock standard as follows:

$$\frac{50000\ \mu g\ \#2\ diesel\ fuel}{mL} \Big| \frac{2\ mL}{50\ mL} = 2000\ ug/mL\ \#2\ diesel$$

12.14 Prepare a 200 ug/mL o-terphenyl surrogate spiking standard by serial dilution in methanol as follows:

12.14.1 Use the analytical balance located in the instrumental laboratory. Make sure the balance has been calibrated that day in accordance with TriMatrix GR-10-113.

12.14.2 Tare a 25 mL volumetric flask on the balance. Do not add solvent.

12.14.3 Weigh and record 0.5000 g of o-terphenyl into the flask to the nearest 0.0001 g.

12.14.4 Once the o-terphenyl is weighed and recorded, add approximately 20 mL of methanol then swirl and check for dissolution. If the o-terphenyl does not dissolve, do not dilute to volume. Continue to swirl until dissolved or if dissolution does not appear to be happening, perform the following:

12.14.4.1 Stopper and position in an ultrasonic bath to aid in dissolution.

12.14.4.2 Closely monitor the solution during sonication as the ultrasonic bath will warm the standard. Do not allow the solution to get warm.

12.14.4.3 Remove from the bath as soon as the o-terphenyl begins to dissolve.

12.14.4.4 Swirl until dissolved.

12.14.4.5 After dissolving, dilute to volume with methanol. Invert and shake vigorously several times to mix thoroughly. Concentration is 20,000 ug/mL.

12.14.5 Prepare the 200 ug/mL solution as follows:

12.14.5.1 Fill a 100 mL volumetric flask with about 80 mL of methanol.

12.14.5.2 Measure and add 1.0 mL of the 20,000 ug/mL solution into the flask.

12.14.5.3 After diluting to volume with methanol, invert and shake vigorously several times to mix thoroughly. Concentration is 200 ug/mL.

12.14.5.4 Pour into 5 pre-labeled 16 mL vials and store without headspace at 4 ±2° C until needed. Discard any residual solution. Expiration is 6 months from the date prepared.

12.14.6 Pour the remaining 20,000 ug/mL solution into a pre-labeled 20 mL vial and store without headspace at 4 ±2° C until needed. Expiration is 12 months from the date prepared.

13.0 ANALYTICAL PROCEDURE

13.1 Tune the disruptor horns at the beginning of each work shift. Refer to the instrument manual for tuning instructions.

13.2 Perform the sample extraction as follows:

13.2.1 Remove and discard any extraneous foreign objects such as sticks, leaves or rocks.

13.2.2 After removing, homogenize the sample in accordance with TriMatrix SOP GR-09-128.

13.2.3 Label all glassware with the complete sample number and extraction type. For example, label whether the extraction is to be a matrix spike (MS), matrix spike duplicate (MSD) or blank spike (BS/LFB).

13.2.4 Perform the following steps rapidly to minimize the evaporation of volatile compounds.

13.2.4.1 For sandy to dry matrices, weigh 30 g of sample into a 400 mL beaker and record to the nearest 0.1 g (example: 30.2 g). Then, add 20-25 g of anhydrous sodium sulfate. Mix well. The mixture should have a sandy appearance and be free-flowing after mixing. If not, mix in additional sodium sulfate until it is.

13.2.4.2 For wet to gummy matrices, weigh 30 g of sample into a 400 mL beaker and record to the nearest 0.1 g (example: 30.2 g). Then, add approximately 60 g of anhydrous sodium sulfate. Mix well. The mixture should have a sandy appearance and be free-flowing after mixing.

13.2.4.3 If not free-flowing, mix in additional sodium sulfate until it is. Record the total amount used to match with quality control samples.

13.2.4.4 Add 200 µL of 200 ug/mL surrogate (Section 12.14) to all extractions including extraction blanks (BLK), blank spikes (BS/LFB) and matrix spikes (MS/MSD). Concentration in the final extract volume and sample is as follows:

$$\frac{\frac{200\ \mu g\ \text{o-terphenyl}}{mL}}{\frac{0.2\ mL}{1\ mL\ \text{extract}}} = \begin{array}{c}40\ \text{ug/mL o-terphenyl (in extract)}\\ (1.3_3\ \text{mg/kg in 30 g sample})\end{array}$$

13.2.4.5 Add 500 µL of the 2,000 ug/mL spiking standard (Section 12.13) to blank and matrix spikes.

13.2.4.6 After spiking, immediately add 100 mL of methylene chloride.

13.2.5 Position the bottom surface of the 3/4 inch disrupter horn tip about 1/2 inch below the solvent surface but above the sediment layer.

13.2.6 Extract ultrasonically for 3 minutes at full power (output control knob set at 10 and percent-duty cycle knob set at 50 percent).

13.2.7 Have a K-D concentrator set up to receive the extraction solvent as follows:

13.2.7.1 Insert a 100 mm filter funnel lined with qualitative filter paper.

13.2.7.2 Add approximately 1 inch of anhydrous sodium sulfate to the filter funnel.

13.2.7.3 Rinse the filter paper and sodium sulfate with methylene chloride.

13.2.7.4 Insert into the top of the K-D concentrator flask with attached concentrator tube.

13.2.8 Decant the extraction solvent into the filter funnel and let drain to the K-D flask.

13.2.9 Repeat the extraction two more times with two additional 100 mL volumes of methylene chloride. Decant to the K-D flask after each extraction. On the final extraction, pour all sediment from the extraction beaker into the filter funnel and rinse with methylene chloride. Collect all rinsings in the K-D flask.

13.2.10 Concentrate extracts as follows:

13.2.10.1 Add one or two clean boiling chips to the K-D flask and attach a three-ball Snyder column.

13.2.10.2 Pre-wet the Snyder column by adding approximately 1 mL methylene chloride to the top.

13.2.10.3 Position in the water bath (50-60° C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor.

13.2.10.4 At the proper rate of distillation, the column will actively chatter but the chambers will not flood with condensed solvent. Adjust the depth of the concentrator tube accordingly.

13.2.10.5 When the apparent volume of liquid reaches 1 mL (usually within 10 to 15 minutes), remove the K-D apparatus from the bath. Let drain and cool for at least 10 minutes before removing the Snyder column.

Note: Do not let the concentration go to dryness. If it does, repeat the entire extraction.

13.3 After cooling, remove the Snyder column and rinse the lower joints of the column and the flask into the concentrator tube with 1-2 mL of methylene chloride.

13.4 Add another clean boiling chip to the concentrator tube and attach a 2-ball micro-Snyder column. Pre-wet the column with 0.5 mL methylene chloride then partially immerse in the 60-70° C water bath. Do not immerse too deep or bumping of the extract and/or flooding of the micro-Snyder column will occur. Immerse only to the point that the balls in the column actively chatter but do not flood.

13.5 When the apparent volume reaches 0.5 mL, remove from the water bath and allow to cool for 10 minutes (do not remove the micro-Snyder column before the cool-down).

Note: Do not let the concentration go to dryness. If it does, repeat the entire extraction.

13.6 After cooling, remove the column and transfer the extract to a pre-calibrated 1 mL vial using a clean, disposable, Pasteur pipette.

13.7 Rinse the lower joints of the micro-Snyder column with approximately 0.2 mL of methylene chloride into the concentrator tube then transfer the rinse to the 1 mL vial also.

13.8 Adjust the final volume in the vial to 1 mL with methylene chloride then cap tightly.

13.9 Store extracts at 4 ±2° C in the GC refrigerator until analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Analysts are responsible for data quality and for correctly filling in all paperwork, laboratory notebooks and other documentation. This is required for quality assurance and to provide the client with fully traceable and defensible data.

14.2 Input all required extraction data to the laboratory information management system (LIMS/Element™).

14.3 Batch reports must be filled in completely to insure that results are reported correctly and data is associated with the batch quality control.

14.4 If internal chain-of-custody (CoC) is required, it is important that the CoC form be filled in completely and correctly.

14.5 Logbooks must be filled in completely and correctly. **Corrections are to be made with a lineout, initials, and date, not a write-over**. Blank lines in the logbook must be Z'd out.

15.0 QUALITY ASSURANCE

15.1 Extract an extraction blank (BLK), blank spike (BS/LFB), matrix spike (MS) and matrix spike duplicate (MSD) for each extraction batch of up to 20 samples or each day samples are extracted. Whichever is more frequent.

15.2 Add 200 µL of 200 ug/mL surrogate (Section 12.14) to all extractions including extraction blanks (BLK), blank spikes (BS/LFB) and matrix spikes (MS/MSD).

15.3 Prepare the extraction blank by extracting 30 g of sodium sulfate as a sample. Add extra sodium sulfate to match the maximum amount of sodium sulfate used in samples. Extract and evaluate as a 30 g sample.

15.4 Prepare the blank spike by adding 500 µL of 2000 ug/mL DRO spiking solution (Section 12.13) to 30 g of sodium sulfate. Add extra sodium sulfate to match the maximum amount of sodium sulfate used in samples. Extract and evaluate as a 30 g sample with a 33.3_3 mg/kg spike.

$$\frac{\text{2000 µg \#2 diesel fuel}}{\text{mL}} \Big| \frac{\text{0.5 mL}}{\text{30 g}} = 33.3_3 \text{ mg/kg \#2 diesel}$$

15.5 Prepare matrix spikes by adding 500 µL of 2000 ug/mL DRO spiking solution (Section 12.13) to 30 g of sample before extracting. Add the same amount of sodium sulfate to match the amount of sodium sulfate used in the unspiked sample. Extract and evaluate as a 30 g sample with a 33.3_3 mg/kg spike.

15.6 An out-of-control blank spike requires the generation of a non-conformance report to initiate root-cause analysis and corrective action. These need coordinated with the analysis laboratory and with the quality assurance department.

15.7 Address all quality control issues in accordance with TriMatrix SOP GR-03-101.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a successful Initial Demonstration of Capability (IDC) study.

16.2 Perform the IDC as follows:

16.2.1 Prepare a 2,000 ug/mL DRO blank spiking standard from a source other than that used for calibration of the DRO gas chromatograph (GC) instrument.

16.2.2 Set up four 30 g sodium sulfate blank spikes, spike with 0.5 mL of the spiking standard (and surrogate), and extract as samples.

16.2.3 Analyze in accordance with TriMatrix SOP GR-03-122 then input results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must fall within LIMS blank spike acceptance limits. Relative standard deviation must be ≤20%.

16.3 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

16.4 Repeated IDC failure will indicate a problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques used and repeat the study successfully.

16.5 Samples may not be extracted by the analyst until an IDC study has been successfully completed.

16.6 Copies of successful IDC studies (Spreadsheet and extraction data) must be submitted to the quality assurance department for training documentation.

16.7 A Continuing Demonstration of Capability (CDC) study is required annually. The CDC can be accomplished by any of the following approaches:

16.7.1 By repeating the IDC study.

16.7.2 By using the last four results from the annual method detection limits (MDL) study if extracted exclusively by the analyst.

16.7.3 By inputting four consecutive blank spike results obtained during the course of routine sample processing to the IDC spreadsheet if extracted exclusively by the analyst.

16.7.4 By exclusively and successfully extracting a blind PT study sample during the course of routine sample processing.

16.8 A Method Detection Limit (MDL) study must be extracted annually in accordance with TriMatrix GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update IV, Revision 3, February, 2007, Method 8015C, Nonhalogenated Organics by Gas Chromatography

19.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, Ultrasonic Extraction

20.0 ATTACHMENTS

20.1 Preparation Batch Report Example

20.2 Standards Log Example

Attachment 20.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0804647 Page 1 of 1

Printed: 6/16/2008 1:45:36PM

Semivolatiles GC, Soil, 3550B Sonication Extraction

Batch comments:

no bsd or ms msd per work order comments
DO NOT ADD ANY SAMPLES TO THIS BATCH

Lab Number	Container	Prepared	By	Initial (g)	Final (mL)	Client Source ID	Client Spike ID	Client uL Spike	Comments
0804444-04	A	Apr-25-08 16:15	ASC	30	1	[redacted]			
DRO EPA 8015B									
0804647-BLK1		Apr-25-08 16:47	ASC	30	1				same as batch 0804646
0804647-BS1		Apr-25-08 16:47	ASC	30	1		7120133	500	same as batch 0804646

Comments	Analyst Initials

bch_TM_byAnalysis.rpt

Approved By: QA 10-2-08 **QA Officer**

Approved By: ASC 10/2/08 **Area Supervisor**

Attachment 20.2
Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

7030944

Description:	(AMP) Diesel Fuel #2 2o	Expires:	Apr-23-08
Standard Type:	Other	Prepared:	Aug-31-06
Solvent:	Solvent Lot #A046397	Prepared By:	** Vendor **
Final Volume (mls):	1	Department:	Expired
Vials:	1	Last Edit:	May-13-08 13:05 by JLW

Restek part=31259
Lot: A046397
Received 3/27/07

Analyte	CAS Number	Concentration	Units
DRO - 8015B (C10-C28)		50000	ug/mL

Approved By: [signature] 10-2-08
QA Officer

Approved By: ASC 10/2/08
Area Supervisor

TRIMATRIX LABORATORIES

STANDARD OPERATING PROCEDURE

Diesel or Oil Range Organics (DRO or ORO)

SW-846 Method 8015C

APPROVALS:

Area Supervisor: Jodi L. Blouw Date: 6-30-10

QA Officer: Tom C. Boocher Date: 6-30-10

Operations Manager: Jeff P. Glaser Date: 6/30/10

Procedure Number: GR-03-122
Revision Number: 2.4

Date Initiated: 6/28/95
Effective Date: 7/15/10

Date Revised: 6/30/10
Pages Revised: All

By: Andrea S. Colborn

Total Number of Pages: 22

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 Analytes

1.1.1 This procedure is designed to measure Diesel Range Organics (DRO) or Oil Range Organics (ORO) in water and soil extracts. The DRO analysis corresponds to an alkane range of C_{10} to C_{28} and a boiling point range of approximately 170 to 430° C. The ORO analysis corresponds to an alkane range of C_{28} to C_{36}.

1.1.2 Diesel Range Organics includes any mid-range petroleum product eluting within the specified retention time range.

1.1.3 Other hydrocarbon mixtures such as motor oil will be quantified only by client request.

1.2 Dynamic Range

1.2.1 Dilutions must be performed as necessary to put the extract concentration within the linear range of calibration.

1.2.2 The linear range of calibration is approximately 125 to 4000 ug/mL DRO or ORO.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update IV, Revision 3, February, 2007, Method 8015C, "Nonhalogenated Organics by Gas Chromatography"

3.0 SUMMARY OF PROCEDURE

3.1 Methylene Chloride extracted water and soil samples are injected into a capillary column gas chromatograph equipped with a Flame Ionization Detector (FID).

3.2 Quantitation is performed by comparing the total chromatographic area from alkanes C_{10} to C_{28} including resolved and unresolved peaks to the response of a #2 diesel fuel calibration for DRO.

3.3 Quantitation is performed for ORO by comparing the total chromatographic area from alkanes C_{28} to C_{36} including resolved and unresolved peaks to the response of a SAE 30 W motor oil calibration.

4.0 PARAMETER OR COMPOUND LIST

4.1 Diesel Range Organics (DRO)

4.2 Oil Range Organics (ORO)

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-09-123, *EPA DRO Soil Extraction*, latest revision.

5.2 TriMatrix SOP GR-09-124, *EPA DRO Water Extraction*, latest revision.

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision.

5.4 TriMatrix SOP GR-04-101, *Semi-Volatile Organic Laboratory Corrective Action,* latest revision.

5.5 TriMatrix SOP GR-10-123, *Element™ Data Transfer (Datatool),* latest revision.

5.6 TriMatrix SOP GR-16-100, *Cleaning Organics Glassware,* latest revision.

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL),* latest revision.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Organic compounds including, chlorinated hydrocarbons, phenols and phthalate esters are detectable under conditions of this procedure. As defined by the method, the result includes these compounds if they fall within the DRO or ORO retention time range.

6.2 Interferences are reduced by washing all glassware in accordance with TriMatrix SOP GR-16-100 then rinsing the clean glassware with methylene chloride just prior to using. Extraction blanks are extracted with each batch of up to 20 samples to demonstrate that the extraction is free of contaminants.

6.3 High purity methylene chloride is used to minimize contamination from the solvent.

6.4 Pattern recognition of diesel or other fuel types can sometimes be complicated by weathering, biodegradation or the presence of fuel mixtures.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses while in the laboratory. In addition, disposable gloves must be worn whenever samples or reagents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 For laboratory waste disposal, refer to TriMatrix SOP GR-15-102.

7.4 The total toxicity and/or carcinogenicity of reagents used in this procedure have not been precisely defined.

7.4.1 Treat all chemicals as a potential health hazard.

7.4.2 Reduce exposure to the lowest possible level by adherence to established safety policies.

7.4.3 Material Safety Data Sheets are maintained on the laboratory intranet of all chemicals used in this procedure. Consult the MSDS for detailed chemical information.

7.5 Samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean contaminated personal protective equipment before using again.

7.6 Bring all safety issues to the immediate attention of the Area Supervisor and/or Health and Safety Officer.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Refer to TriMatrix SOP GR-09-123 for soil sample collection and handling.

8.2 Refer to TriMatrix SOP GR-09-124 for water sample collection and handling.

8.3 All analyses must take place within 40 days of sample extraction.

8.4 Store extracts in the GC refrigerator at 4 ±2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Hewlett Packard 5890 and 5890 series II gas chromatograph equipped with FID.

9.2 Column, J&W DB-5, 30 m x 0.53 mm ID x 0.5 um film thickness.

9.3 Leap A200SE autosampler

9.4 PE Nelson Turbochrome Data Acquisition System

9.4.1 Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

9.4.2 Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

9.5 Only FID may be used for DRO and other hydrocarbon mixtures. FID response is essentially the same for all hydrocarbons. Other detectors will not produce equivalent results.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 The GC is equipped with silicone septa (Supelco, Thermogreen, 10 mm) which eventually core and leak. Replace as necessary to avoid contaminating the injection port and to minimize column bleed.

10.2 Inlet sleeves can become contaminated and restrict flow after accumulating sample debris and septum particles. Replace the sleeve periodically to minimize loss in resolution.

10.3 In addition to sleeve replacement and when resolution degrades, clip about 6 inches off the front end of the column. Replace a column that becomes too short for adequate resolution after clipping.

11.0 CHEMICALS AND REAGENTS

11.1 Methylene Chloride, ultra resi-analyzed for organic residue analysis.

11.2 Diesel fuel #2 composite, 50,000 ug/mL in methylene chloride, purchased as a certified solution.

11.3 Diesel range organics mix containing 10 n-alkanes from C_{10} to C_{28} in methylene chloride, each at 2,000 ug/mL, purchased as a certified standard and used to establish the DRO retention time window.

Note: This stock must be sonicated prior to opening.

11.4 SAE-30 W Motor Oil Standard, 20,000 ug/mL in methylene chloride, purchased as a certified solution.

11.5 Florida TRPH Standard containing 17 n-alkanes from C_8 to C_{40} in hexane, each at 500 ug/mL, purchased as a certified standard and used to establish the ORO retention time window.

11.6 Surrogate (5,000 ug/mL triacontane in acetone/tetrahydrofuran (5:1)), purchased as a certified solution.

Note: This surrogate is not spiked into samples unless two surrogates are specifically requested by the client and/or project.

11.7 o-Terphenyl, purchased as a certified material with at least 98.5% purity.

12.0 STANDARDS PREPARATION

12.1 Prepare the following standards in methylene chloride as follows:

12.1.1 Surrogate (20,000 ug/mL o-terphenyl):

12.1.1.1 Weigh 0.5000 g of neat o-terphenyl into a 25 mL volumetric flask half full of methylene chloride.

12.1.1.2 Stopper and sonicate until thoroughly dissolved.

Note: Sonication is required to thoroughly dissolve.

12.1.1.3 Once dissolved, bring to volume with methylene chloride and invert until mixed. Store with minimal headspace at 0 - 6° C.

12.1.1.4 Expiration is 1 year from the date prepared.

12.1.2 Diesel Fuel #2 composite (4,000 ug/mL):

12.1.2.1 Measure 800 uL of 50,000 ug/mL Diesel fuel #2 composite (section 11.2) into a 10 mL volumetric flask half full of methylene chloride.

12.1.2.2 Measure 100 uL of 20,000 ug/mL o-terphenyl stock (section 12.1.1) into the flask.

12.1.2.3 Measure 400 uL of 5,000 ug/mL triacontane stock (section 11.6) into the flask.

12.1.2.4 Dilute to volume and invert until mixed thoroughly.

12.1.2.5 Store with minimal headspace at 0 - 6° C

12.1.2.6 Expiration is 6 months from the date prepared.

Note: Prepare a second-source standard in the same way.

12.1.3 Motor Oil Standard (4,000 ug/mL):

12.1.3.1 Measure 1.0 mL of 20,000 ug/mL SAE 30 W motor oil standard (Section 11.4) into a 5.0 mL volumetric flask half full of methylene chloride.

12.1.3.2 Measure 50 uL of 20,000 ug/mL o-terphenyl stock (Section 12.1.1) into the flask.

12.1.3.3 If a second surrogate is requested by the client, measure 200 uL of 5,000 ug/mL triacontane stock (Section 11.6) into the flask.

12.1.3.4 Dilute to volume with methylene chloride until mixed thoroughly.

12.1.3.5 Store with minimal headspace at 0 - 6° C.

12.1.3.6 Expiration is 6 months from the date prepared

Note: Prepare a second-source standard in the same way.

12.2 Other fuel range organics such as fuel oil, kerosene or mineral spirits may be used to calibrate upon client request and should be prepared in the same way.

12.3 Record all standard preparations in Element™ (Attachment 23.3). Label all standards with the following information:

12.3.1 Standard name

12.3.2 Element™ assigned ID

12.3.3 Date made

12.3.4 Analyst initials

12.3.5 Solvent used

12.3.6 Concentration and units

12.3.7 Expiration date

12.4 Purchased stock standards in ampoules are one-use standards and once opened must be used or disposed of. Do not store or use beyond the assigned expiration date in Element™.

12.5 Working standards expire 6 months from the date made except when the stock used in preparation expires earlier. Then, the working standard must expire on the stock expiration date.

12.6 Remove expired standards from the laboratory to the waste disposal area for proper disposal.

12.7 Promptly replace chemicals before the current supply is entirely consumed or expired to avoid expedited shipping costs.

13.0 SAMPLE PREPARATION

13.1 Refer to TriMatrix SOP GR-09-123 for the extraction of soil samples.

13.2 Refer to TriMatrix SOP GR-09-124 for the extraction of water samples.

14.0 CALIBRATION PROCEDURES

14.1 Prepare six calibration standards from the working fuel standard by dilution using the following chart. Combine the working standard volume with the appropriate methylene chloride volume. Use a glass microsyringe for all measurements.

Calibration Level	Concentration of Working Standard (ug/mL)	Volume of Working Standard (uL)	Volume Methylene Chloride (uL)	Final Concentration (ug/mL)
6	4000	1000	0	4000
5	4000	500	500	2000
4	2000	500	500	1000
3	1000	500	500	500

Calibration Level	Concentration of Working Standard (ug/mL)	Volume of Working Standard (uL)	Volume Methylene Chloride (uL)	Final Concentration (ug/mL)
2	500	500	500	250
1	250	500	500	125

14.2 After injecting a methylene chloride blank, inject each calibration standard beginning with the lowest concentration. Use a 1.0 uL injection volume for all standards, quality control and extracts.

14.3 Construct a linear calibration from the results as follows:

14.3.1 Use the retention time window established in Section 15.2 for all calibration standards. Integrate the entire DRO or ORO area including the "hump" by projecting a horizontal baseline from the C_{10} (C_{28} for ORO) retention time start to the C_{28} (C_{36} for ORO) retention time end.

14.3.2 Subtract the methylene chloride blank run just before the calibration from each calibration point. Measure the area of the blank in the same way as a sample or calibration point.

14.3.3 Since o-terphenyl elutes within the DRO retention time range, subtract the o-terphenyl peak area in each calibration point from the DRO area.

14.3.4 Since o-terphenyl does not elute within the ORO range it does not need to be subtracted. If triacontane is used as a second surrogate due to client request, it does elute within the ORO retention time range and the peak does need to be subtracted from the ORO range only.

Note: Integrating just the surrogate peak takes a separate integration from the DRO or ORO integration and requires a valley-to-valley baseline.

14.3.5 Using the spreadsheet on the laboratory intranet library, enter the methylene chloride area, the o-terphenyl area and the DRO or ORO area for each calibration standard. The spreadsheet will calculate an adjusted DRO or ORO area. Enter the adjusted area back into the GC integration software. Refer to Attachment 23.7 for an example spreadsheet.

Note: Although the GC integration software does not directly calculate sample results, the DRO or ORO calibration is input to the software for documentation and uploading to Element™.

14.3.6 The GC integration software calculates a liner regression plot based on the adjusted areas entered, including a slope, an intercept and the coefficient of determination (r^2). If r^2 is greater than 0.99, input the slope and intercept into the spreadsheet. If not, the calibration is unacceptable and must be re-run.

Note: The spreadsheet also calculates slope and intercept to monitor whether these values are entered to the spreadsheet correctly.

14.4 When requested, samples will be qualitatively screened for fuel types other than DRO or ORO and reported as the fuel type tentatively identified. If no match is found, the sample will be reported against the fuel type that best approximates the observed chromatographic fingerprint and retention time window.

14.5 When requested, calibration will be performed using the specific fuel type contaminating the sample collection site. Otherwise, the DRO result is reported based on the purchased diesel fuel #2 composite standard. The ORO result is reported based on the purchased SAE 30 W motor oil standard unless otherwise specified.

14.6 Verify the linear regression curve each 12-hour work shift by injection of an initial Continuing Calibration Verification (CCV) standard, by injection every ten sample injections and injection at the end of each run sequence. Percent recovery must be 85 – 115% to be acceptable. Samples may not be run or must be re-injected if the initial CCV of the 12-hour work shift fails in any way. Samples run in association with a non-initial failing CCV are addressed as follows:

14.6.1 If the CCV is less than 85% from the expected value, all samples run before and after the failing CCV must be re-injected on a system that is in control, to be bracketed with acceptable QC.

14.6.2 If the CCV is more than 115% from the expected value, samples run before and after the failing CCV with positive results must be re-injected on a system that is in control, to be bracketed with acceptable QC.

14.6.3 If the CCV is more than 115% from the expected value, non-detect samples do not need re-injected but only if the next CCV in the run sequence is greater than 85%.

14.7 The six calibration point concentrations are 125, 250, 500, 1000, 2000, and 4000 ug/mL unless otherwise specified. The low calibration standard determines the minimum reporting limit and the high calibration standard will define the calibration range.

14.8 Prepare a second source calibration verification (SCV) to validate calibration standards accuracy. The SCV concentration must be near the calibration midpoint when prepared. Analysis recovery must be within 75 – 125% of the expected value to validate the calibration standards and begin sample analysis. If recovery is not within 75 – 125%, re-inject the SCV once. If the re-injection fails, prepare new calibration solutions and repeat the SCV test on the new curve.

15.0 ANALYTICAL PROCEDURE

15.1 Instrument Set-up

15.1.1 GC conditions used unless otherwise specified:

15.1.1.1 Set helium column pressure to 4 ±1 mL/min

15.1.1.2 Set air to 400 ±15 mL/min

15.1.1.3 Set hydrogen to 30 ±1 mL/min

15.1.1.4 Set make-up gas to 20 ±1 mL/min

15.1.1.5 Set column temperature program to 70° C for 1.0 minute, increase to 320° C at 10°/min then hold for 10.0 minutes (run time is 36 minutes).

15.1.1.6 Set the FID to 320° C and injector to 280° C.

15.2 Calculate the retention time window as follows:

15.2.1 Before establishing the retention time window, be certain the GC is at optimum operating conditions. Make three injections of the diesel range organics mix (Section 11.3) throughout the course of a 72-hour period.

15.2.1.1 The retention time window for each component analyte is defined as plus or minus three times the standard deviation of the average retention time. Use the beginning of the retention time window for C_{10} and the end of the retention time window for C_{28} for the DRO retention time window. Use the beginning of the

retention time window for C_{28} and the end of the retention time window for C_{36} for the ORO retention time window.

15.2.1.2 In cases where the standard deviation for a particular peak is zero, use the average retention time ±0.05 minutes as a retention time window.

15.2.2 Calculate retention time windows for o-terphenyl and DRO on each column and whenever a new column is installed. Retain all retention time window data in the laboratory area group's folder.

15.2.3 Calculate the Oil Range Organics retention time window in the same way.

15.3 Sample analysis is as follows:

15.3.1 Use flame ionization detection (FID) only. Use an injection volume of 1.0 uL.

15.3.2 Use forward baseline projection to integrate the DRO area. Valley-to-valley integration disregards the un-resolvable hump in the chromatogram which contributes significantly to the DRO area count. However, use a second valley-to-valley integration to obtain the surrogate peak area.

15.3.3 Inject a methylene chloride blank prior to each 12-hour work shift to determine the area generated by baseline bleed. Integrate area by projecting a horizontal baseline from the beginning of the retention time window for C_{10} (C_{28} for ORO) to the end of the retention time window for C_{28} (C_{36} for ORO). Subtract the area integrated from all calibration standards, QC and samples in the run sequence.

15.3.4 Run a CCV to verify the existing calibration (Section 14.0) in accordance with Section 14.6.

15.3.5 If a sample concentration exceeds the calibration range (≥4,000 ug/mL), dilute the extract to approximately the mid-point of the curve and reanalyze.

Note: Run at least one methylene chloride blank after any highly concentrated sample to minimize carryover.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Quantitate samples using the calibration specified in Section 14.0.

16.2 The spreadsheet located on the laboratory intranet library uses the following equations to calibrate DRO or ORO:

16.2.1 An adjusted DRO peak area is determined by subtracting out the surrogate and methylene chloride areas:

$$A_{DRO} = A_{fb} - (S_{vv} + B_{mc})$$

Where:
A_{fb} = DRO area
S_{vv} = Surrogate area
B_{mc} = Methylene chloride blank area
A_{DRO} = DRO adjusted area

16.2.2 The initial calibration is based on a linear regression plot as follows:

$$C_{ext} = (A_{DRO} - b)/m$$

Where:
A_{DRO} = DRO adjusted area
C_{ext} = DRO extract concentration, in ug/mL
m = Slope
b = Intercept

16.3 From the calibration, sample results are obtained as follows:

16.3.1 Water Samples:

$$C_{s\text{-}water} = (C_{ext} \cdot V_e \cdot DF)/V_w$$

Where:
V_e = Total extract volume, in mL
DF = Extract dilution factor, in mL/mL
V_w = Sample volume extracted, in mL
C_{ext} = DRO extract concentration, in ug/mL
$C_{s\text{-}water}$ = DRO sample concentration in mg/L

16.3.2 Soil samples:

$$C_{s\text{-}soil} = (C_{ext} \cdot V_e \cdot DF)/(V_s \cdot PS)$$

Where:
V_e = Total extract volume, in mL
DF = Extract dilution factor, in mL/mL
V_s = Sample mass extracted, in grams
C_{ext} = DRO extract concentration, in ug/mL
$C_{s\text{-}soil}$ = DRO sample concentration, in mg/kg
PS = Sample percent solids in decimal form (0.98 not 98%).

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for data quality and for filling in documentation correctly. It is important to document each analysis by correctly filling in, handing in and filing paperwork. This is required for quality control and to provide clients with defensible data.

17.2 Upload sample data from the instrument to Element™ in accordance with TriMatrix SOP GR-10-123.

17.3 If internal chain-of-custody is required, it is important that the CoC form be filled in correctly and completely.

17.4 All run, maintenance and instrument file archival logbooks must be filled in completely and correctly.

17.5 Make all hardcopy corrections with a single lineout (not a writeover) then initial and date the correction.

17.6 Cross out unused lines in run logbooks to document the lines as purposely blank. Refer to Attachment 23.8 for a run logbook example.

17.7 Archive calibration and other run sequence hardcopies in the correct storage box for ease in retrieval.

18.0 QUALITY ASSURANCE

18.1 An extraction blank (BLK) is extracted with each extraction batch of up to 20 samples. The DRO concentration present in the BLK must be less than the reporting limit. If this limit is exceeded, samples with positive results must be re-extracted. If there is insufficient sample to re-extract or the holding time has expired, positive results must be qualified as estimated based on laboratory contamination.

18.2 Analyze continuing calibration verifications (CCV) in accordance with section 14.6.

18.3 Calculate surrogate recovery for each extract analysis. If recovery is not within established acceptance limits consult TriMatrix SOP GR-04-101 to determine when and how data is qualified.

18.3.1 High recovery might be due to a co-eluting matrix interference from the sample. Examine the chromatogram for co-elution. No corrective action is required in this instance.

18.3.2 Low recovery may be due to poor extraction efficiency. This should be verified by re-extracting the sample if sample volume and hold times permit.

18.3.2.1 If surrogate recovery in the extraction blank (BLK) is below the lower control limit, only samples with failing surrogate recoveries will require re-extraction and/or narration.

18.3.2.2 If surrogate recovery in the extraction blank (BLK) is above the upper control limit, no corrective action is required as long as sample surrogate recoveries are acceptable.

18.3.3 If surrogate recoveries are outside control limits in the MS/MSD, re-analysis is only required if blank spike (BS) recoveries are also outside control limits. If the BS recovery is still out-of-control after re-analysis, all associated samples must be re-extracted and/or narrated.

18.4 Blank Spikes (BS) and Matrix Spikes

18.4.1 Matrix spike and matrix spike duplicates are extracted for each batch of up to 20 samples, for each matrix. The blank spike is extracted for each batch of up to 20 samples or each 12-hour work shift (whichever is more frequent), for each matrix. Analyze all extraction QC with the samples extracted

18.4.2 Until 15 – 20 Matrix Spikes (MS), Matrix Spike Duplicates (MSD) and blank spikes have been analyzed for a matrix, recoveries will be validated against default limits of 50 – 150% and a relative percent difference (RPD) of 20%.

18.4.3 Element™ calculates and stores recovery limits and data.

18.4.4 If recovery or precision is not within acceptable limits, take corrective action in accordance with TriMatrix SOP GR-04-101 to determine when and how data is qualified.

18.5 If any blank spike is out-of-control, the problem must be immediately identified and corrected before further samples are run. Failure of the blank spike requires immediate corrective action. Every effort must be made to determine the root cause of failure (for example: mis-spiking or mis-extracting). Once the cause is found, take the appropriate corrective measures:

18.5.1 Failure of a BS requires re-extraction and/or narration of the sample batch. If re-extraction is not possible, qualify as follows:

18.5.1.1 If blank spike recovery exceeds the upper control limit, report all positive results for associated samples in the batch as "estimated".

18.5.1.2 All results less than the reporting limit are acceptable and need no qualification.

18.5.1.3 If blank spike recovery is less than the lower control limit, qualify positive results for every sample in the batch and the reporting limit as "estimated".

18.5.1.4 A batch narrative must be written each time the blank spike fails.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a successful Initial Demonstration of Capability (IDC) study.

19.2 Perform a DRO IDC as follows:

19.2.1 Prepare a 1,000 ug/mL DRO standard from a source other than that used for calibration of the DRO gas chromatograph (GC).

19.2.2 Analyze four aliquots in accordance with every step in the procedure beginning with an initial calibration.

19.2.3 Input results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must 85 – 115%. Relative standard deviation must be ≤20%.

19.3 Perform an ORO IDC by following the steps in Section 19.2 but use an ORO standard instead.

Note: An IDC is required for all fuel types before samples can be extracted or analyzed.

19.4 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

19.5 Repeated IDC failure will indicate a problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques used and repeat the study successfully.

19.6 Samples may not be analyzed by the analyst until an IDC study has been successfully completed.

19.7 Submit copies of all IDC study attempts (spreadsheet and extraction data) to the quality assurance department for training documentation.

19.8 A Continuing Demonstration of Capability (CDC) study is required annually. The CDC can be accomplished by any of the following approaches:

19.8.1 By repeating the IDC study.

19.8.2 By using the last four results from the annual method detection limit (MDL) study if analyzed exclusively by the analyst.

19.8.3 By inputting four consecutive blank spike results obtained during the course of routine sample analysis to the IDC spreadsheet if analyzed exclusively by the analyst.

19.8.4 By exclusively and successfully analyzing a blind PT study sample during the course of routine sample analysis.

19.9 A Method Detection Limit (MDL) study is required annual in accordance with TriMatrix SOP GR-10-125. A separate MDL study is required for all fuel types that are analyzed. Refer to Attachment 23.6 for a Method Detection Limit Study example.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix Laboratory SOP GR-15-102 for laboratory waste disposal.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update IV, Revision 3, February, 2007, Method 8015C, "Nonhalogenated Organics by Gas Chromatography"

23.0 ATTACHMENTS

23.1 DRO Chromatogram on DB-5 Column Example

23.2 Diesel Range Organics Mix Chromatogram on DB-5 Column Example

23.3 Standards Log Example

23.4 Analysis Sequence Report Example

23.5 Preparation Batch Report Example

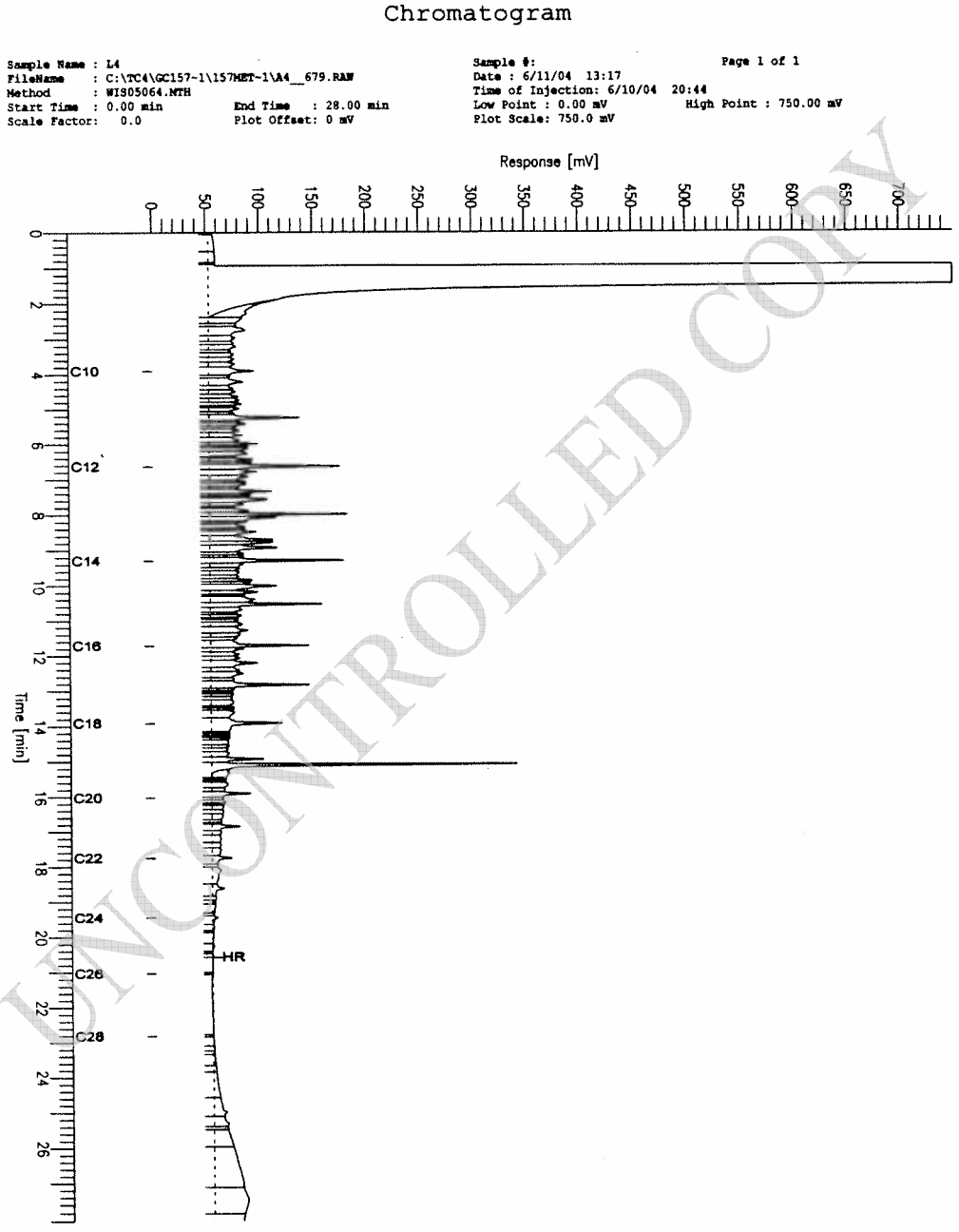
23.6 Method Detection Limit (MDL) Study Example

23.7 DRO Calculation Spreadsheet Example

23.8 Instrument Run Logbook Example

23.9 ORO Chromatogram on DB-5 Column Example

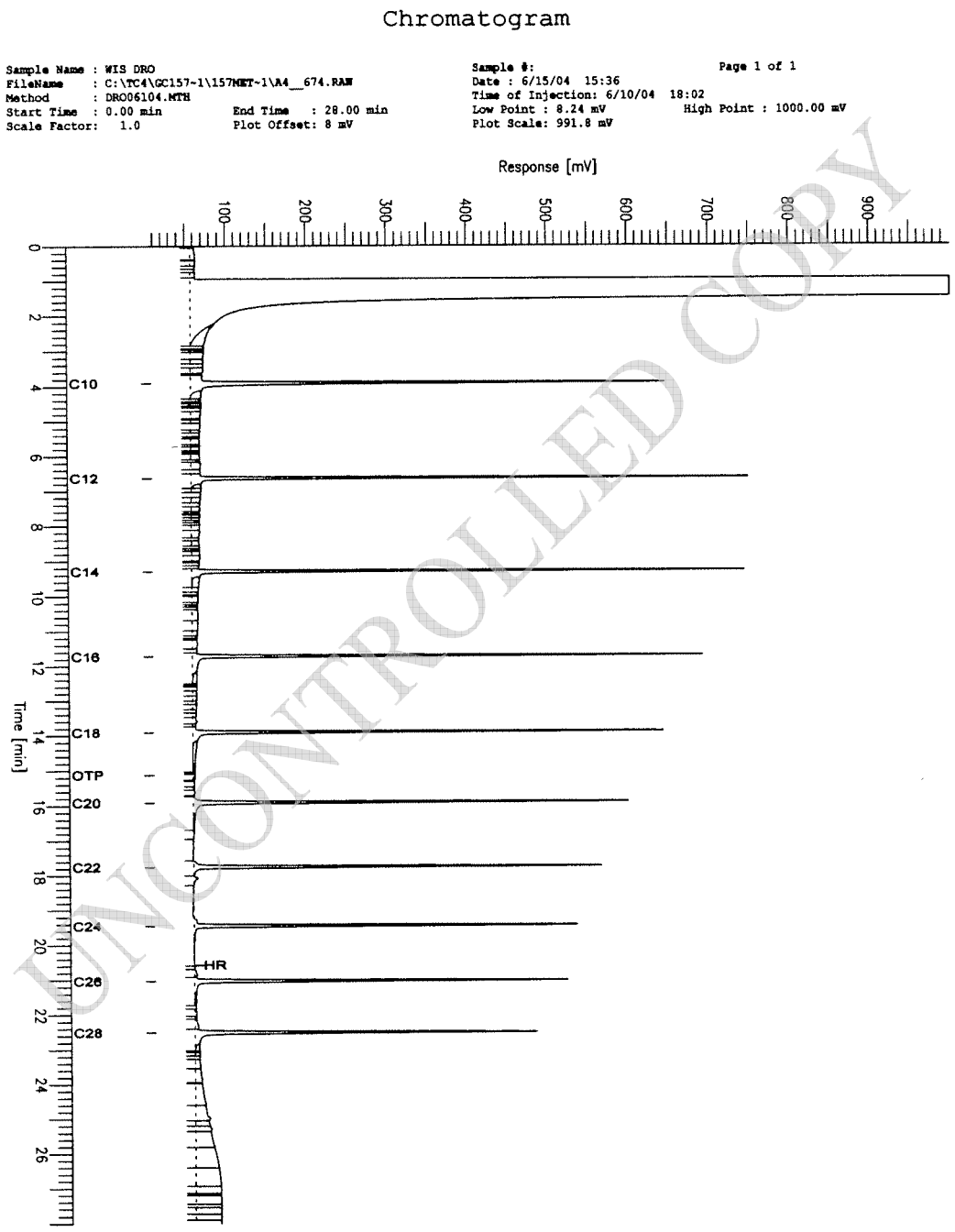
Attachment 23.1
DRO Chromatogram on DB-5 Column Example



Approved By: 6-30-10 Approved By: 6/30/10

QA Officer Area Supervisor

Attachment 23.2
Diesel Range Organics Mix Chromatogram on DB-5 Column Example



Approved By: 6-30-10 QA Officer

Approved By: 6/30/10 Area Supervisor

Attachment 23.3
Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8080409

Description:	(AMP) Diesel Fuel #2 2o	Expires:	Aug-19-09
Standard Type:	Other	Prepared:	Aug-13-08
Solvent:	Solvent Lot #A059536	Prepared By:	** Vendor **
Final Volume (mls):	1	Department:	Semivolatiles GC
Vials:	1	Last Edit:	Sep-10-08 16:24 by JMK

Restek part#31259
Lot: A059536
Received 8/13/08

Analyte	CAS Number	Concentration	Units
DRO - 8015B (C10-C35)		50000	ug/mL
DRO - 8015B (C10-C28)		50000	ug/mL

Approved By: 6-30-10 QA Officer

Approved By: 6/30/10 Area Supervisor

Attachment 23.4
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. **ANALYSIS SEQUENCE** **8122329** Page 1 of 1 Printed: 1/12/2009 5:02:55PM

Semivolatiles GC, Waste, Dec-22-08

Instrument = 157, Calibration = UNASSIGNED

Sequence Analyses:
DRO EPA 8015B

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client / QC Type*	*Extraction Comments*
0812259-01	DRO EPA 8015B	A 02			[redacted]	
0814664-BLK1	QC				BLANK	
0814664-BS1	QC				LCS	
0814664-MS1	QC				MATRIX SPIKE	
0814664-MSD1	QC				MATRIX SPIKE DUP	

Approved By: [signature] 6-30-10 QA Officer

Approved By: [signature] 6/30/10 Area Supervisor

Attachment 23.5
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0814664 Page 1 of 1

Printed: 1/12/2009 4:59:25PM

Semivolatiles GC, Waste, 3580A Waste Dilution

Surrogate #1 = 8070950 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0812259	DRO EPA 8015B				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0814664-BLK1		Dec-17-08 08:12	BJH	1	10	20				BLANK	
0814664-BS1		Dec-17-08 08:12	BJH	1	10	20		8060406	200	LCS	
0814664-MS1		Dec-17-08 08:12	BJH	1	10	20	0812259-01	8060406	200	MATRIX SPIKE	
0814664-MSD1		Dec-17-08 08:12	BJH	1	10	20	0812259-01	8060406	200	MATRIX SPIKE DUP	
0812259-01	A	Dec-17-08 08:12	BJH	1	10	20				[redacted]	

Approved By: _____ 6-30-10
QA Officer

Approved By: _____ 6/30/10
Area Supervisor

Attachment 23.6
Method Detection Limit Study Example

SEMI-VOLATILE LABORATORY
INSTRUMENT NUMBER 157 2003 SOIL
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average % Recovery	Standard Deviation	MDL
EPA DRO	8015B	1/29/2004	6.67	mg/kg	9.03	9.78	11.9	10.6	10.0	9.79	11.5	10.4	156%	1.03	3.23

Attachment 23.7
DRO Calculation Spreadsheet Example

TRIMATRIX LABORATORIES

Sample type	Known QC Concentration (ug/L or mg/kg)	DRO Area	o-terphenyl Area	Methylene Chloride Blank Area	Adjusted Area	DRO (mg/L)	Extracted volume (L) or mass (g)	Final Extract Volume (mL)	Final Extract Dilution (uL/uL)	Fractional Solids (%/100)	TurboChrom y-intercept	TurboChrom slope	DRO result to report	units (ug/L or mg/kg)	QC % recovery	Sample in calibration range?
cert1	33.333		716680	648933			30	1	1	1	-695656	11341		mg/kg	#VALUE!	
cert2	33.333	13624642	716846	648933	12258723	1142	30	1	1	1	-695656	11341	38.076	mg/kg	114	YES
cert1	33.333	14049096	716680	648933	12680473	1180	30	1	1	1	-695656	11341	39.341	mg/kg	118	YES
cert2	33.333	13624642	716846	648933	12258723	1142	30	1	1	1	-695656	11341	38.076	mg/kg	114	YES
cert3	33.333	14100431	737671	648933	12713827	1182	30	1	1	1	-695656	11341	38.413	mg/kg	118	YES
cert4	33.333	13853876	729603	648933	12475140	1161	30	1	1	1	-695656	11341	38.711	mg/kg	116	YES
CCV1	1000	12713268	1049784	648933	11014641	1033	1	1	1	1	-695656	11341	1032.5	ug/L	103	YES
							1	1	1	1						
							1	1	1	1						
							1	1	1	1						
							1	1	1	1						
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							1	1	1	1						
ICV1	1000	11976043	819740	406796	10349508	974	1	1	1	1	-695656	11341	973.9	mg/L	-	-
L1	125	1343349	102632	406796	834522	135	1	1	1	1	-695656	11341	135.0	mg/L	-	-
L2	250	2826421	211986	406796	2209840	256	1	1	1	1	-695656	11341	256.8	mg/L	-	-
L3	500	5766676	427119	406796	4963461	498	1	1	1	1	-695656	11341	498.1	mg/L	-	-
L4	1000	11925846	846291	406798	10674856	1003	1	1	1	1	-695656	11341	1002.6	mg/L	-	-
L5	2000	23727167	1683126	406796	21638247	1999	1	1	1	1	-695656	11341	1969.3	mg/L	-	-
L6	4000	49663792	3328284	406796	44831613	4014	1	1	1	1	-695656	11341	4014.4	mg/L	-	-

Attachment 23.8
Instrument Run Logbook Example

TRIMATRIX LABORATORIES

Instrument 157 HP5890	Date: 3/18/10	Sequence#: 19	Date Archived:	Analysts: ASC
Instrument Settings / Injection Volume	GC Program		CCV Standards	Routine Maintenance Items
Column Type: DB-5ms	Initials:		9120894	Column Clipped: Yes / No
Column ID:	Holds:			New Injection Port Liner: Yes / No
Injection Volume: 1 µL	Rates:			New [illegible]: Yes / No
Injector / Detector: 250°C / 320°C	Finals:			New Syringe: Yes / No
Default Temperature Program: YES/No	Hold:			

Run ID	File ID	Analysis Method	Dilution	ICV/CCV Check (Pass or High/Low)	Sample Notes, Calibration Standards, Analytical Batch Information, etc.
MeCl2	374	SW3630C SW8021B/8015B			
↓	375	↓			
↓	376				
WIS DRO	377				
DRO 125	378				9120897
250	379				9120896
500	380				9120895
1000	381				9120894
2000	382				9120893
↓ 4000	383				9120887
DRO SCV 1000	384			Pass	0030737
1003240-01	385				
-02	386				
↓ -03	387				
1003261-01	388				
↓ -02	389	↓			

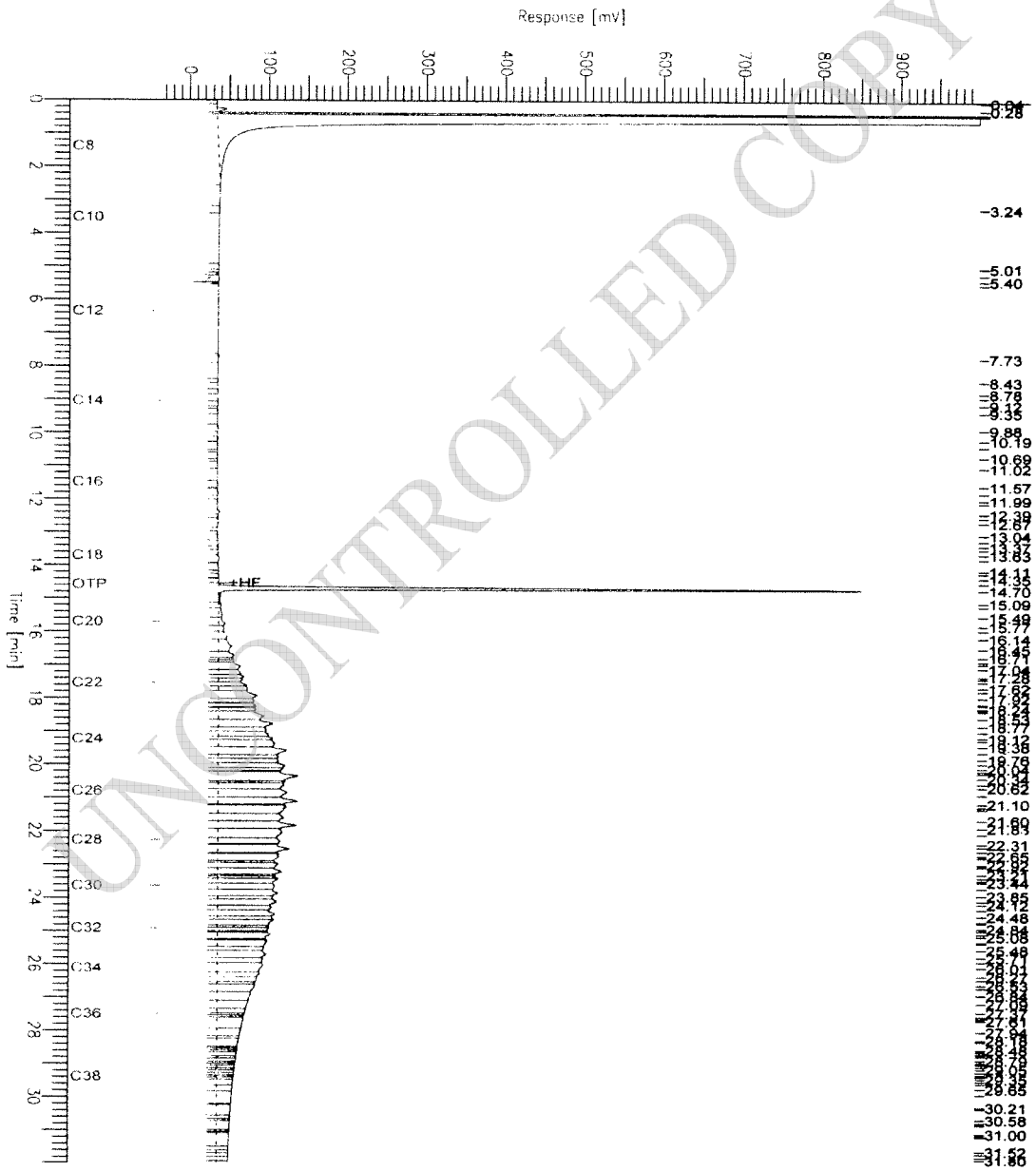
file: 157_RUN.XLS page: 1 of 40 revision: 2.0

Attachment 23.9
ORO Chromatogram on DB-5 Column Example

Chromatogram

Sample Name : ORO 4000
FileName : C:\TC4\GC157~1\A19\A19_092.RAW
Method : ORO2170.MTH
Start Time : 0.00 min End Time : 32.00 min
Scale Factor: 0.0 Plot Offset: -35 mV

Sample #: Page 1 of 1
Date : 6/29/10 10:17
Time of Injection: 2/17/10 12:38
Low Point : -35.27 mV High Point : 1000.00 mV
Plot Scale: 1035.3 mV



Approved By: 6-30-10
QA Officer

Approved By: 6/30/10
Area Supervisor

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Diesel Range Organics (DRO)

SW-846 Method 8015B

APPROVALS:

Area Supervisor: Janet M. Kudirka Date: 1/13/09
Janet M. Kudirka

QA Officer: Date: 1-12-09
Tom C. Boocher

Operations Manager: Date: 1/15/09
Jeff P. Glaser

Procedure Number: GR-03-122
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By: Jeff P. Glaser
Total Number of Pages: 23

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-16-10		4-16-11

1.0 SCOPE AND APPLICATION

1.1 Analytes

1.1.1 This procedure is designed to measure Diesel Range Organics (DRO) in water and soil extracts. The analysis corresponds to an alkane range of C_{10} - C_{28} and a boiling point range of approximately 170° C and 430° C.

1.1.2 Diesel Range Organics measures mid-range petroleum products such as diesel or fuel oil. Components greater than C28 present in products such as motor oils or lubrication oils are detectable under conditions of the method. If, based on review of chromatogram, the presence of these product types is suspected, additional efforts may be performed including, but not limited to, analysis of additional reference materials. These additional efforts are not contained within this procedure.

1.2 Quantitation Limits

1.2.1 Quantitation limits are 0.2 mg/L for water and 6.7 mg/kg for soil.

1.3 Dynamic Range

1.3.1 Dilutions must be performed as necessary to put extract concentrations within the linear range of calibration. In general, the individual compound range is 12.5 ug/mL to 400 ug/mL in the final extract. This approximates 125 ug/mL to 4000 ug/mL of DRO.

1.4 Experience

1.4.1 This procedure is based on solvent extraction and gas chromatography (GC), and must be used by or under the supervision of experienced analysts.

1.4.2 Analysts must be skilled in chromatographic interpretation as a quantitative tool.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 2, December, 1996, Method 8015B, "Nonhalogenated Organics Using GC/FID"

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 2, December, 1996, Method 8000, "Determinative Chromatographic Separations"

3.0 SUMMARY OF PROCEDURE

3.1 One liter of water or thirty grams of soil are spiked with o-terphenyl surrogate and extracted with methylene chloride. Extracts are dried and concentrated to 1.0 mL, then injected into a capillary column gas chromatograph, equipped with a Flame Ionization Detector (FID). Quantitation is performed by comparing

total chromatographic area from alkanes C_{10} to C_{28}, including resolved and unresolved components, to the response of a diesel calibration.

4.0 PARAMETER OR COMPOUND LIST

Retention Time Standards	**Surrogate Standard**
Decane	Ortho-Terphenyl
Dodecane	
Tetradecane	
Hexadecane	
Octadecane	
Eicosane	
Decosane	
Tetracosane	
Hexacosane	
Octacosane	

Diesel Range Organics (DRO): All chromatographic peaks eluting between decane (n-C10) and octacosane (n-C28). Quantitation is based on direct comparison of the area within this range to the total area of a fuel standard calibration curve. Refer to Attachment 23.1 for an example diesel range organic chromatogram on a DB-5 column. Refer to Attachment 23.2 for an example retention time standard chromatogram on a DB-5 column.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-09-123, *EPA DRO Soil Extraction*, latest revision

5.2 TriMatrix SOP GR-09-124, *EPA DRO Water Extraction*, latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.4 TriMatrix SOP GR-04-101, *Semi-Volatile Organic Laboratory Corrective Action,* latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Organic compounds including, chlorinated hydrocarbons, phenols, and phthalate esters are detectable under conditions of this procedure. As defined by the method, the result includes these compounds if they fall within the DRO retention time range.

6.2 Interferences are reduced by washing all glassware with hot soapy water and rinsing with tap water, acetone, and then hexane. Immediately before use, the clean glassware is prerinsed with methylene chloride. Method performance blanks must be analyzed with each batch extracted or for every 20 samples, to demonstrate that reagents are free of contaminants.

6.3 High purity, pesticide-grade methylene chloride is used to minimize contamination from the solvent.

6.4 Contamination by carryover can occur whenever high and low level samples are sequentially analyzed. If an unusually concentrated sample is analyzed, a solvent blank analysis must be performed after the analysis to check for residual contamination.

6.5 Qualitative identification of diesel or other fuel types may be complicated by environmental processes such as weathering, biodegradation, or the presence of fuel mixtures.

7.0 SAFETY PRECAUTIONS

7.1 The toxicity or carcinogenicity of reagents used in this procedure have not been precisely defined. All chemicals must be treated as a potential health hazard. From this viewpoint, chemical exposure must be reduced to the lowest possible level by the proper use of hoods and personal protective equipment. A current file of Material Safety Data Sheets (MSDS) is available to all personnel on the laboratory intranet.

7.2 Solvents used in extraction and concentration of samples are highly toxic. Avoid all skin contact, vapor inhalation and ingestion. These solvents must only be used in a fume hood. Use protective clothing, disposable gloves and safety glasses when handling.

7.3 All analyzed sample extracts and expired standards must be properly disposed of, following the TriMatrix standard operating procedure GR-15-102 for laboratory waste disposal.

7.4 Analysts must comply with all procedures for health and safety, including those outlined in the TriMatrix Laboratory Safety Manual.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Water samples are collected in a 1 L glass container and acidified to pH 2 with HCl. Solids are collected in a core tube or glass jar. Samples are stored at 4° C ± 2° C from the time of collection until extraction. Extraction must be performed on water samples within seven days and soil samples within 14 days. All analyses must take place within 40 days, after extraction. Extracts are stored in the GC refrigerator at 4° C ± 2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Gas Chromatograph: Hewlett Packard 5890 and 5890 series II gas chromatograph equipped with FID. Recommended conditions:

- Injector: 280° C
- Detector: 320° C
- Hydrogen Flow: 30 mL/minute
- Air Flow: 400 mL/minute
- Make-up: 20 mL/minute

9.2 Column

9.2.1 J&W DB-5 – 30 m X 0.53 mm ID, 0.5-micron film thickness or equivalent.

9.2.2 Temperature Program:

9.2.2.1 70° C hold for 1.0 minute, ramp 10° C/minute to 320° C, hold for 2.0 minutes.

9.3 Data System: PE Nelson Turbochrome Data Acquisition System

9.4 Autosamplers:

9.4.1 Leap A200SE

9.5 Note: FID must be used for DRO and other hydrocarbon measurements as described in this procedure. FID response is essentially the same for all hydrocarbons. Other detectors will not produce equivalent results.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Septa Replacement

10.1.1 Direct injection gas chromatographs are equipped with silicone septa (Supelco Thermogreen 10 mm, or equivalent), which eventually core and leak. These are replaced as necessary to avoid contaminating the inlet sleeve with rubber particles, and to eliminate bleed.

10.2 Inlet Sleeve Cleaning and/or Replacement

10.2.1 Inlet sleeves can become contaminated and restrict flow after accumulating sample debris and septum particles. Sleeves must be replaced periodically to minimize loss in resolution.

10.3 Column Clipping/Replacement

10.3.1 In addition to sleeve replacement and when resolution degrades, about 6" should be clipped off the front end of the column. When a column becomes too short for adequate resolution after clipping, it must be replaced.

11.0 CHEMICALS AND REAGENTS

11.1 Methylene Chloride, Hexane, Acetone: Pesticide grade or equivalent

11.2 Petroleum Fuel Product Standards – Refer to section 12.0 for preparation

12.0 STANDARDS PREPARATION

12.1 Stock Standard Solution: Prepare the following stock standards. Unless noted, all are prepared in the methylene chloride listed above. Standard preparation must follow guidelines in Method 8000.

12.1.1 Recommended Surrogate Standard: 2000 ug/mL ortho-terphenyl (OTP) stock. A working solution is made at 200 ug/mL by adding 1.0 mL of the stock standard to a 10 mL volumetric flask half full of methylene chloride. Dilute to volume, stopper, and invert three times to mix.

12.1.2 Diesel Component Standard: 20,000 ug/mL total concentration C_{10}-C_{28} even normal alkane compounds for establishing a DRO retention time window. A 1000 ug/mL standard is prepared by adding 500 uL of the stock standard to 10 mL in a volumetric, with methylene chloride.

12.1.3 Stock Laboratory Fortified Blank (LFB) – 50,000 ug/mL diesel #2. A working solution is made at 2,000 ug/mL in acetone (a water soluble solvent) by adding 1.0 mL of the stock standard to a 25 mL volumetric flask half full of acetone. Dilute to volume, stopper, and invert three times to mix.

12.1.4 Diesel Fuel #2, Fuel Oil #1, Kerosene-commercially manufactured stock standards at 50,000 ug/mL. Working standards are made at 4000 ug/mL by adding 2 mL of stock standard to a 25 mL volumetric flask half full of methylene chloride. Dilute to volume, stopper, and invert three times to mix.

12.2 All standards must be recorded in the semi-volatile Standards Logbook. Refer to Attachment 23.3 for an example of the standards log record. All standard vials must be labeled with the following information:

12.2.1 Standard name

12.2.2 Laboratory-assigned standard ID

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent used

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 Standard preparation information is recorded in the semi-volatile Standards Logbook. Solutions are labeled with a two-letter prefix, GC, followed by a number depending upon logbook, page and line number. The nomenclature for standards naming will be as follows: lab area, book number, page number, line number. For example, GC2-4.8 would be from GC Standards Logbook 2, page 4, and line 8. Working standard solutions are stored in the Semi-volatile GC lab refrigerator at 4° C ± 2° C.

12.4 Shelf-life of stock standards is either one year from the date of preparation or the manufacturer's expiration date, whichever comes first. Working standard solutions expire six months from the date of preparation, or by the expiration of the stock standard, whichever comes first. Once a chemical or solution has expired, it

must be removed from the laboratory and properly disposed of. Monitor expiration dates of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical before the current supply is entirely consumed.

13.0 SAMPLE PREPARATION

13.1 Refer to TriMatrix SOP GR-09-123 (*EPA DRO Soil Extraction*) for extraction of soil samples.

13.2 Refer to TriMatrix SOP GR-09-124 (*EPA DRO Water Extraction)*, for extraction of water samples.

14.0 CALIBRATION PROCEDURES

14.1 Calibrate the GC with a minimum of five points using diesel fuel #2. Response of the standard is the sum of the peak area counts within the retention time window, for each concentration. The ratio of the area sum to concentration injected is the calibration factor (CF) and is calculated for each concentration, then averaged. If the percent relative standard deviation (% RSD) is less than 20% for the average CF, linearity through the origin can be assumed, and the average calibration factor can be used in place of a linear regression curve.

14.1.1 Calibration Factors (CF) for each calibration point are calculated using the following formula, then used to determine an average calibration factor:

14.1.1.1 $$CF_{stdx} = A_{stdx}/C_{stdx}$$

Where:
A_{stdx} = Peak area summation of a standard, adjusted to compensate for methylene chloride
C_{stdx} = Standard concentration number "x" in ug/mL, where x = 1 through 5

14.1.1.2 $$CF_{avg} = [CF_{std1} + CF_{std2} + CF_{std3} + CF_{std4} + CF_{std5}]/5$$

Where:
C_{stdx} = Standard concentration in ug/mL

14.1.2 Percent Relative Standard Deviation (%RSD) is calculated using the following formula:

$$\%RSD = [(SD_{avg})*100]/CF_{avg}$$

Where:
SD_{avg} = standard deviation of the average CF
CF_{avg} = average calibration factor for an analyte

14.1.3 Percent RSD must be ≤20% using at least 5 calibration points.

14.1.4 If %RSD for any analyte is greater than 20%, a regression curve must be generated by plotting peak area against concentration. A linear regression fit is used, without forcing through the

origin. To be acceptable, the correlation coefficient "r" must be 0.995 or higher, or the coefficient of determination (COD) "r^2" must be 0.990 or higher.

14.1.4.1 Linear regression requires five calibration standards and is calculated based on the following:

$$C_{ext} = (A_{DRO}-b)/a$$

Where:

A_{DRO} = Peak area summation in sample "s"
C_{ext} = Concentration of DRO in extract "ext"
a = Slope of the line (also called the coefficient of C) "s"
b = The intercept

14.1.5 When requested, samples will be qualitatively screened to identify a fuel type other than already listed, and a matching calibration curve run for quantitation. Whenever possible, calibration will be performed using the specific fuel contaminating a site. If it is not possible to identify a fuel, samples will be quantitated against fuel that best approximates the chromatographic fingerprint and retention time window.

14.1.6 **Note: A Diesel fuel #2 calibration will be used for all samples, unless otherwise requested.**

14.2 The average calibration factor or linear regression curve must be verified each working day by injection of an Initial Calibration Verification (ICV) standard and by analysis of subsequent Continuing Calibration Verification (CCV) standards, every ten sample injections. If calibration factors for these injections vary from the predicted response by more than ±15%, a new calibration must be prepared.

$$\text{Percent Difference} = \frac{CF2 - CF1}{CF1} \times 100\%$$

where:
CF1 = Average CF from the calibration curve
CF2 = Calibration Factor from ICV or CCV

14.3 The six points used to calibrate are 200, 250, 500, 1000, 2000, 4000 ug/mL. The lab reserves the right to change calibration concentrations, depending upon project requirements. The low calibration standard will determine the minimum reporting limit, and the high calibration standard will define the upper linear range.

14.4 Prepare a separate-source Laboratory Control Sample (LCS) to validate calibration standards accuracy. The LCS concentration should be equal to the midpoint of the calibration curve when prepared. Analysis recovery must be within 75-125% of the LCS expected value, to validate calibration and begin sample analysis. If recovery is not within 75-125%, the LCS must be reinjected. If the second injection fails, a new calibration curve must be prepared and validation repeated.

15.0 ANALYTICAL PROCEDURE

15.1 Instrument Set-up

15.1.1 Conditions (Recommended): Set helium column pressure to 4 mL/minute (±1), Air to 400 mL/minute (±15), Hydrogen to 30 mL/minute (±1), and make-up to 20 mL/minute (±1). Set column temperature to 70° C for 1.0 minute, increasing to 320° C at 10°/minute, then hold for 2.0 minutes. (Run time = 28.00 minutes). Set the FID to 320° C and injector to 280° C.

15.2 Retention Time Window Definition

15.2.1 Before establishing retention time windows, be certain the GC system is at optimum operating conditions. Make three injections of the diesel component standard throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

15.2.1.1 The retention time window for o-terphenyl is defined as plus or minus three times the standard deviation of the average retention time. The diesel fuel retention time window is based on the retention times of C10 and C28.

15.2.1.2 In those cases where the standard deviation for a particular peak is zero, the laboratory will use the average retention time ± 0.05 minutes as a retention time window.

15.2.2 The laboratory must calculate retention time windows for o-terphenyl and diesel fuel on each GC column, and whenever a new column is installed. Retention time studies must be retained in the laboratory.

15.3 Gas Chromatograph Analysis

15.3.1 Samples are analyzed by GC/FID only. The suggested injection volume is 1.0 uL, using the conditions established in 15.1.1.

15.3.2 A methylene chloride solvent blank must be run in each analysis sequence to determine the area generated by baseline bleed, prevailing over the 24-hour period of the sequence. This area is generated by projecting a horizontal baseline between the retention times of n-C_{10} and n-C_{28}. The area must be subtracted from the DRO area of all standards and samples run within the analysis period.

15.3.3 If initial calibration has been performed, verify by analysis of a mid-point ICV initially, followed by continuing calibration verifications (CCV) every ten sample injections, and at the end of the analytical sequence.

15.3.4 Calculate the ICV/CCV percent differences from the mean calibration factor, as in 14.2. If any calibration verification has a percent difference greater than ±15%, the instrument must be recalibrated.

15.3.5 Forward baseline projection must be used to integrate the DRO area count. (Valley-to-valley integration disregards the un-resolvable area of the chromatogram, which contributes

significantly to the DRO area count). However, valley-to-valley integration must be used for the surrogate.

15.3.6 Methylene chloride blanks must be run after samples suspected of being highly concentrated, to minimize carryover.

15.3.7 If product concentration exceeds the linear range of the calibration in the final extract, the extract must be diluted and reanalyzed. The individual compound range is 12.5 ug/mL to 400 ug/mL in the final extract. This is approximately equivalent to 125 ug/mL to 4000 ug/mL of diesel.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Quantitate using the average CF of the calibration, or a linear regression curve as specified in Section 14.1.

16.2 Use the equations below to calculate ug/mL of DRO in an extract:

16.2.1 Calculate an adjusted DRO peak area by subtracting out surrogate and methylene chloride areas:

$$A_{DRO} = A_{fb} - S_{vv} - B_{mc}$$

Where:
A_{fb} = DRO area using forward baseline integration
S_{vv} = Surrogate area using valley to valley integration
B_{mc} = Methylene chloride blank using forward baseline integration
A_{DRO} = The adjusted DRO area used for quantitation

16.2.2 For an average calibration factor, use the following equation:

$$C_{ext} = A_{DRO}/CF_{avg}$$

Where:
CF_{avg} = The average calibration factor from the initial calibration (refer to section 14.1)
C_{ext} = DRO extract concentration in ug/mL

16.2.3 For a linear regression curve, use the following equation:

$$C_{ext} = (A_{DRO} - b)/a$$

Where:
A_{DRO} = The adjusted DRO area used for quantitation
C_{ext} = DRO extract concentration in ug/mL
a = Slope of the line (also called the coefficient of C) "s"

b = The intercept

16.3 Next, calculate sample concentration by one of the following equations:

16.3.1 Water Samples:

$$C_{s\text{-}water} = (C_{ext} * V_e * DF)/V_w$$

Where:

V_e = Total extract volume in mL
DF = Extract dilution factor
V_w = Sample volume extracted in mL
C_{ext} = DRO extract concentration in ug/mL
$C_{s\text{-}water}$ = DRO sample concentration in mg/L

16.3.2 Soil samples:

$$C_{s\text{-}soil} = (C_{ext} * V_e * DF)/(V_s * PS)$$

Where:

V_e = Total extract volume in mL
DF = Extract dilution factor
V_s = Sample mass extracted in grams
C_{ext} = DRO extract concentration in ug/mL
$C_{s\text{-}soil}$ = DRO sample concentration in mg/kg
PS = Sample percent solids in decimal form (0.98 not 98%).

16.3.3 Use the method of external standards to calculate concentrations in all samples and spikes.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for data quality and also for filling in documentation correctly. It is important to document each analysis by correctly filling in, handing in, and filing paperwork. This is required for quality control purposes, and to provide clients with defensible data.

17.2 LIMS Reporting

17.2.1 When an analyst finishes running a sample batch, all data must be given to LIMS. Benchsheets must be filled in completely to ensure that results are reported correctly, and samples are associated with the right quality control. It is important that the quality control batch number from the extraction summary and analytical batch information from the 24-hour shift is filled in correctly. Dilution factors need added so reporting limits can be raised accordingly. All

positive hits must be recorded by crossing out the default reporting limit and writing down the final client result to the right of the reporting limit. This result must already have been through all necessary calculations, including the dilution factor. If a dilution is made to an extract and there is a positive hit, the elevated reporting limit must be written to the left of the default reporting limit. Surrogate results are reported by extract concentrations found, amount spiked, and percent recovery obtained.

17.2.2 If there are matrix spikes, the quality control benchsheet must also be handed in. In addition to section 17.2.1 requirements, quality control benchsheets report matrix spiking by the concentration spiked and percent recovery/percent difference for spike/spike duplicates. If matrix spike results are out-of-control due to extraction or matrix problems, the exclude (EXC) box must be checked to prevent biasing acceptance limit statistics.

17.2.3 An extracted Method Preparation Blank (MPB) must be handed in for every extraction batch. A daily blank (BLK) must be handed in with every 24-hour shift. Both sets of benchsheets need handed in. It is important to remember that no LFB can be handed in without first having handed in the associated MPB.

17.2.4 Internal chain-of-custody (COC) forms must be filled in correctly, when required. These are a very important part of the data package.

17.2.5 All LIMS benchsheets (including COC forms) must be placed in the correct colored folder and given to data entry. Blue folders containing BLK benchsheets must also include raw data quantitation reports and chromatograms.

17.3 Laboratory Required Paperwork

17.3.1 All run, maintenance, tape, and standard logbooks must be filled in completely and correctly. Corrections must only be made using line outs, not writeovers or scribbles. Blank lines in run logbooks must be Z'd out.

17.3.2 All ICV and CCV standard runs must be archived.

17.3.3 All LIMS documentation except for sample and quality control benchsheets, and raw data except for blanks and ICV/CCV standard runs, must be placed in the correct folder then given to the Semi-Volatile Laboratory technician. The technician must record the date, time and contents handed in.

17.4 Rounding and Significant Figures

17.4.1 Rounding is performed on final quantitated results only. All non-quality control results are rounded to 2 significant figures.

17.4.2 Quality control results are reported as whole numbers up to 3 significant figures.

18.0 QUALITY ASSURANCE

18.1 A method preparation blank (MPB) must be extracted with each extraction batch. The DRO concentration present in an MPB must be less than the reporting limit. If this limit is exceeded, samples with positive results must be re-extracted. If there is insufficient sample to re-extract or the holding time has expired, positive results must be qualified as estimated based on laboratory contamination.

18.2 Continuing calibration verifications (CCV) must be run before sample analysis begins, and after every 10 samples (refer to section 15.3.4). The analytical sequence must also end with a passing calibration verification. Acceptance limits for CCVs are ±15% difference from the calibration. If the CCV fails high, only samples with positive results require re-analysis. If the CCV fails low, all samples run before and after the CCV must be re-analyzed. If corrective action does not resolve the problem, a new initial calibration must be performed.

18.3 Calculate surrogate recovery for each extract analysis. If recovery or precision is not within acceptable limits, consult TriMatrix SOP GR-04-101 to determine when and how data is qualified.

18.3.1 High recovery may be due to a co-eluting matrix interference from the sample. Examine the chromatogram for evidence of co-elution. No corrective action is required in this instance.

18.3.2 Low recovery may be due to poor extraction efficiency. This should be verified by re-extracting the sample, if sample volume and hold times permit.

18.3.3 If surrogate recovery in a method performance blank (MPB) is below the lower control limit, only samples with failing surrogate recoveries will require re-extraction. If MPB surrogate recovery is above the upper control limit, no corrective action is required as long as sample surrogate recoveries are acceptable.

18.3.4 If surrogate recoveries are outside control limits in the MS/MSD, re-analysis is only required if LFB spike recoveries are also outside control limits. If the LFB is still out-of-control after re-analysis, all associated samples must be re-extracted.

18.4 Laboratory Fortified Blanks (LFB) and Matrix Spikes

18.4.1 Matrix spike and matrix spike duplicates are extracted at least every 20 samples, for each matrix. LFBs are extracted every 20 samples or each shift, for each matrix. Whichever is more frequent. This QC must be analyzed with the samples extracted

18.4.2 Until 15-20 Matrix Spikes (MS), Matrix Spike Duplicates (MSD), and LFBs have been analyzed, recoveries of each will be validated against default limits of 50-150%, and a duplication limit of 20% RSD.

18.4.3 To calculate recovery limits, calculate average percent recovery (R) and standard deviation (SD) of the average, by matrix. Express acceptable recovery as the interval from R – 3*SD to R + 3*SD. For example, if R = 90% and SD = 10%, recovery limits would be 60-120%. Precision limits for spike duplicates are calculated the same way except using average and standard deviation of relative percent difference. Limits must be updated at least annually.

18.4.4 Calculate percent recovery as follows:

$$\text{Percent Recovery} = \frac{|(\text{Aspk} - \text{Asmp})|}{\text{SPK}} \times 100$$

where:

Aspk = actual concentration found in the spiked sample, in mg/L or mg/kg
Asmp = amount found in non-spiked sample, in mg/L or mg/kg
SPK = amount spiked, in mg/L or mg/kg [(concentration of spike standard in ug/mL x mL spiked) / initial sample volume (mL) or mass (g)]

18.4.5 Calculate the relative percent difference as follows:

$$\text{Percent RPD (\%RPD)} = \frac{|(\text{SPK1} - \text{SPK2})|}{\left[\frac{(\text{SPK1} + \text{SPK2})}{2}\right]} \times 100$$

where:

SPK1 = Matrix Spike
SPK2 = Matrix Spike Duplicate

18.4.6 If recovery or precision is not within acceptable limits, the Corrective Action SOP GR-04-101 will determine when and how data is qualified.

18.4.7 If any LFB is out-of-control, the problem must be immediately identified and corrected before further samples are run. Failure of an LFB requires corrective action. Every effort must be made to determine the root cause of failure (for example: mis-spiking or mis-extracting). Once the cause is found, corrective measures must be taken

18.4.7.1 Failure of an LFB requires re-extraction of the sample batch. If re-extraction is not possible, qualify as follows:

18.4.7.1.1 If DRO in the LFB exceeds the upper recovery limit, positive results for associated samples in the batch are to be reported as "estimated".

18.4.7.1.2 All results less than the reporting limit are acceptable and need no qualification (LIMS qualifier 66).

18.4.7.1.3 If DRO recovery in the LFB is less than the lower control limit, positive results for every sample in the batch must be qualified as "estimated" and the reporting limit considered an approximation. A batch narrative must be written each time the LFB fails (LIMS qualifier 5).

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Preparation and analysis of an initial demonstration of capability.

19.1.1.1 Spike 0.500 mL of 2,000 ug/mL diesel fuel #2 standard and 1.0 mL of o-Terphenyl surrogate spiking solution into each of four, 1000 mL aliquots of water or 30 g aliquots of Ottawa Sand. Process following every step in the extraction procedure. The IDC spiking solution must be prepared independently from standards used for quantitation.

19.1.1.2 Analyze the four aliquots following every step in the procedure.

19.1.1.3 Calculate average DRO recovery "x" in mg/L(or mg/kg), and standard deviation of the average "s" in mg/L for each analyte, using all four results.

19.1.1.3.1 Average recovery "x" must be within laboratory established limits, and relative standard deviation "s" must be $\leq$20%. If "x" and "s" meet these criteria, the demonstration of capability study is complete. The analyst and the instrument are authorized to run samples.

19.1.1.3.2 When an analyst fails to achieve acceptable results, Section 19.1.1.4 must be followed.

19.1.1.4 Locate and correct the source of the problem and repeat the study. Repeated failure however, may indicate a general problem with technique. If this occurs, locate the problem and correct the technique, then repeat the study. Samples may not be analyzed by any analyst or on any instrument until an IDC is successfully completed.

19.1.1.5 Demonstration of capability studies must be given to the Quality Assurance department for analyst training purposes.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A demonstration of capability study must be performed annually. A CDC may be accomplished by repeating the IDC study, by processing four consecutive LCSs run during the course of routine sample analysis on the IDC spreadsheet, or by submitting an acceptable PT result on the appropriate paperwork. LCSs and PT samples must have been run exclusively by the analyst to be used for a CDC study.

19.2 Method Detection Limit Studies

19.2.1 MDL studies must be performed annually on every instrument using this procedure. The MDL is defined as the minimum concentration of a substance that can be measured and reported with

99 percent confidence that the value is above zero. Actual reporting limits are derived from the MDL study. The minimum possible non-estimated reporting limit is equal to the concentration spiked in the MDL study, provided the MDL passes. Reporting limits achieved for any given analysis will vary depending on instrument sensitivity, matrix effects and dilutions. MDL studies must be completed for both water and soil matrices.

19.2.2 The procedure followed for a MDL study is based on the method given in 40 Code of Federal regulations, Part 136, Appendix B, latest revision.

19.2.3 Seven replicate analyses are performed using reagent water or clean sand, spiked with #2 Diesel at the estimated minimum reportable concentration (0.1 mg/L or 6.7 mg/kg). The last four of the seven water results can be used for an IDC or CDC study, if analyzed exclusively by one analyst.

19.2.4 Since a blank is required to calculate the measured level of DRO, seven separate blanks must be analyzed, one after each of the seven MDL samples. The average blank measurement is subtracted from each of the seven MDL results.

19.2.5 The standard deviation of the average found for the analyte(s) using all seven results is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

19.2.6 If the concentration spiked is between the calculated MDL and ≤ 5 times the calculated MDL, and there are no zero percent recoveries in the set of seven, the MDL is acceptable. If not, the MDL must be re-analyzed. If a study needs re-analyzed at a different concentration, the entire set of seven needs re-analyzed. If a study does not pass due to poor reproducibility on one of the results, only that result needs re-analyzed. Not more than one result may be rejected and re-analyzed however. All seven results do not need obtained in the same analytical batch.

19.2.7 If at any time the nominal reporting limit is above a client's or state's desired reporting limit, the calculated MDL may be used as the reporting limit, provided results are narrated. The narration must state that the reporting limit is based on the calculated MDL, and DRO when spiked at that level was not observed.

19.2.8 Refer to Attachment 23.6 for a Method Detection Limit Study Example.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix Laboratory SOP GR-15-102 (*Laboratory Waste Disposal*), for laboratory waste disposal.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 2, December, 1996, Method 8015B, "Nonhalogenated Organics Using GC/FID"

22.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 2, December, 1996, Method 8000, "Determinative Chromatographic Separations"

23.0 ATTACHMENTS

23.1 Example Diesel Range Organic Chromatogram on DB-5 Column

23.2 Example Retention Time Standard Chromatogram on DB-5 Column

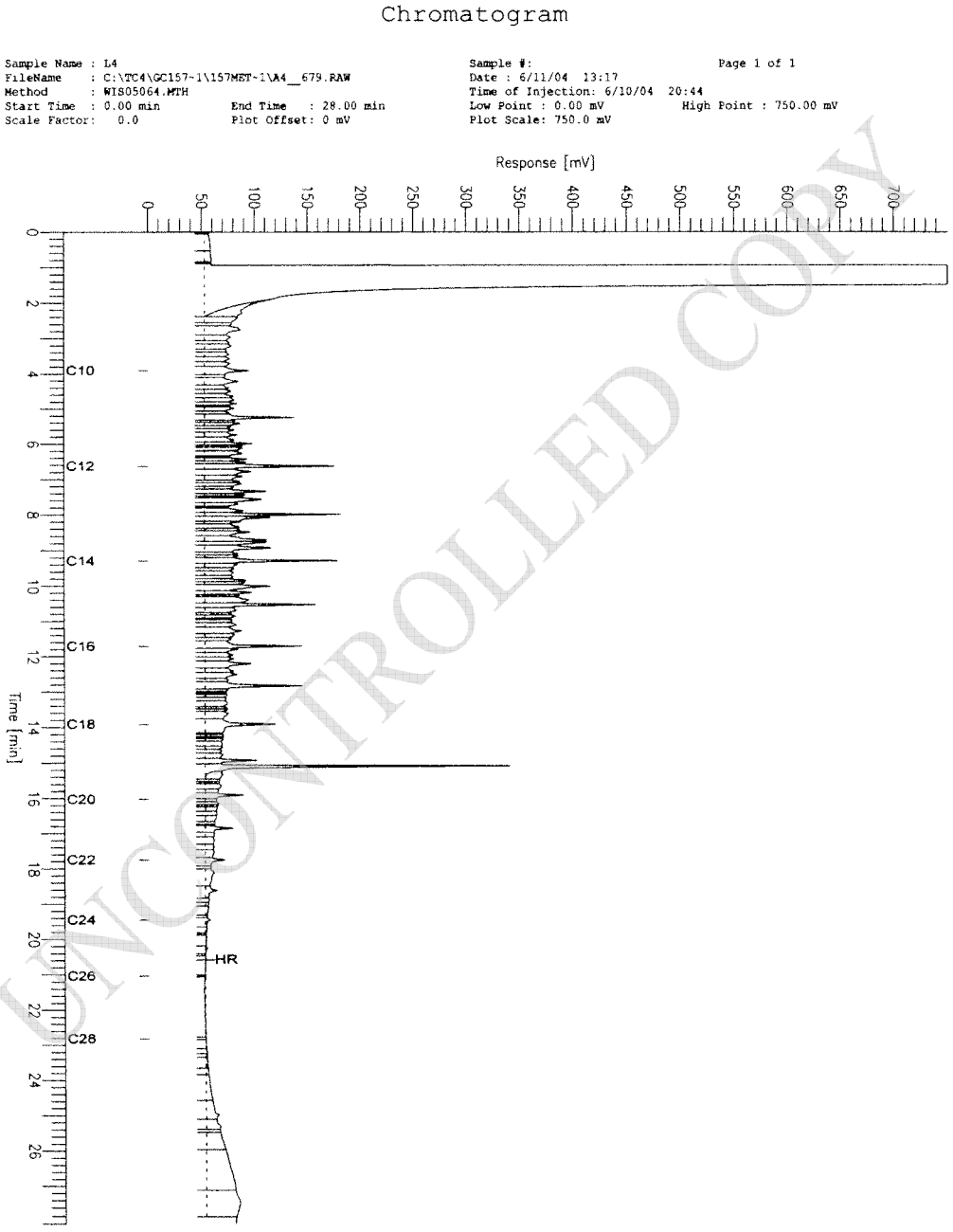
23.3 Standards Log Example

23.4 Analysis Sequence Report Example

23.5 Preparation Batch Report Example

23.6 Method Detection Limit Study Example*
*Only part of the Table or Benchsheet example has been included.

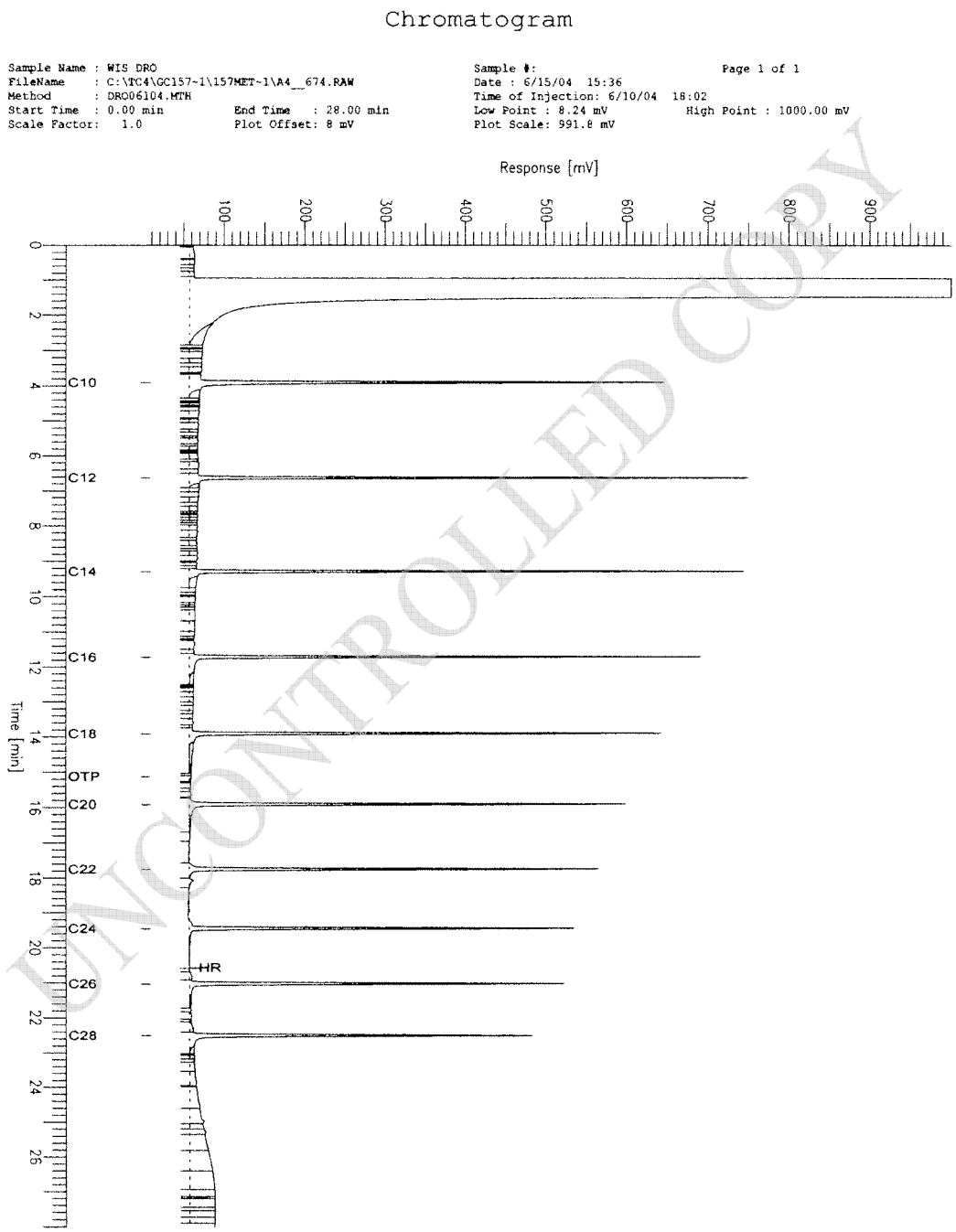
Attachment 23.1
Example Diesel Range Organic Chromatogram on DB-5 Column



Approved By: _____ 1/12/09 **QA Officer**

Approved By: _____ 1/13/9 **Area Supervisor**

Attachment 23.2
Example Retention Time Standard Chromatogram on DB-5 Column



Approved By: _____ 1-12-09 **QA Officer**

Approved By: _____ 1/13/09 **Area Supervisor**

Attachment 23.3
Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8080409

Description:	(AMP) Diesel Fuel #2 2o	Expires:	Aug-19-09
Standard Type:	Other	Prepared:	Aug-13-08
Solvent:	Solvent Lot #A059536	Prepared By:	** Vendor **
Final Volume (mls):	1	Department:	Semivolatiles GC
Vials:	1	Last Edit:	Sep-10-08 16:24 by JMK

Restek part=31259
Lot: A059536
Received 8 13 08

Analyte	CAS Number	Concentration	Units
DRO - 8015B (C10-C35)		50000	ug/mL
DRO - 8015B (C10-C28)		50000	ug/mL

Approved By: [signature] 1-12-09 **QA Officer**

Approved By: [signature] 1/13/09 **Area Supervisor**

Attachment 23.4
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. **ANALYSIS SEQUENCE** **8122329** Page 1 of 1 Printed: 1/12/2009 5:02:55PM

Semivolatiles GC, Waste, Dec-22-08

Instrument = 157, Calibration = UNASSIGNED

Sequence Analyses:
DRO EPA 8015B

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client QC Type*	*Extraction Comments*
0812259-01	DRO EPA 8015B	A 02			[redacted]	
0814664-BLK1	QC				BLANK	
0814664-BS1	QC				LCS	
0814664-MS1	QC				MATRIX SPIKE	
0814664-MSD1	QC				MATRIX SPIKE DUP	

Approved By: _____ 1-12-09 **QA Officer**

Approved By: _____ 1/13/09 **Area Supervisor**

Attachment 23.5
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0814664** Page 1 of 1

Printed: 1/12/2009 4:59:25PM

Semivolatiles GC, Waste, 3580A Waste Dilution

Surrogate #1 = 8070950 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0812259	DRO EPA 8015B				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client QC Type*	*Extraction Comments*
0814664-BLK1		Dec-17-08 08:12	BJH	1	10	20				BLANK	
0814664-BS1		Dec-17-08 08:12	BJH	1	10	20		8060406	200	LCS	
0814664-MS1		Dec-17-08 08:12	BJH	1	10	20	0812259-01	8060406	200	MATRIX SPIKE	
0814664-MSD1		Dec-17-08 08:12	BJH	1	10	20	0812259-01	8060406	200	MATRIX SPIKE DUP	
0812259-01	A	Dec-17-08 08:12	BJH	1	10	20				[redacted]	

Approved By: _____ 1-12-09
QA Officer

Approved By: _____ 1/13/09
Area Supervisor

Attachment 23.6
Method Detection Limit Study Example

SEMI-VOLATILE LABORATORY
INSTRUMENT NUMBER 157 2003 SOIL
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average % Recovery	Standard Deviation	MDL
EPA DRO	8015B	1/29/2004	6.67	mg/kg	9.03	9.78	11.9	10.6	10.0	9.79	11.5	10.4	156%	1.03	3.23

Analytes	MDL	RL	Matrix Spike Recovery	LCS Recovery	RPD	Surrogate Recovery
Tributyltin	0.37 ug/Kg	1.5 ug/Kg	30-160 %	30-160 %	30%	NA
Tripentyltin	NA	NA	NA	NA	NA	30-120 %

SOP Change in Progress Attachment (CIPA)

SOP Number	SOP Title	SOP Revision	SOP Effective Date	CIPA Effective Date
BR-WC-008	TOC Lloyd Kahn Method	12	03/17/10	03/30/10

The following revisions were made to this standard operating procedure (SOP). These changes are effective as of the CIPA Effective Date. Changes to this document will be incorporated into the document with the next revision. This document change is authorized and issued by the laboratory's QA Department.

Page 5 of 18: Change the acceptance criteria for the LCS and MS to 75-125.

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	< RL
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	%R ~~(85-115)~~ 75-125
Sample Duplicate (DP)	Client Request	RPD (≤ 20)
Matrix Spikes (MS)	Client Request	%R ~~(85-115)~~ 75-125

Title: TOC Lloyd Kahn Method

Approval Signatures:

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Bryce E. Stearns
Technical Director

Dan Helfrich
Health & Safety Coordinator

Approval Date: March 17, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of total organic carbon (TOC) and black carbon in soils, sediments and other solids.

The procedure for TOC in soils and sediments is provided in the main body of this SOP. The procedure for the determination of TOC in marine sediment high in inorganic carbon is provided in Appendix B and the procedure for black carbon is provided in Appendix D.

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used to determine percent dry weight in soil and solid materials.

The routine reporting limit is 1000 mg/kg based on an initial sample weight of 10 mg. Additional weight of sample may be used (up to 25 mg) to achieve as low a reporting limit as 500 mg/kg.

2.0 Summary of Method

A 10 mg aliquot of sample is transferred to a tin capsule, treated with phosphoric acid and dried in an oven at a temperature 105°C for 30 minutes to one hour in order to separate the organic carbon from inorganic carbonates and bicarbonates. The sample is analyzed on an instrument where it is pyrolyzed in an inductive type furnace. The carbon is converted to carbon dioxide and measured by a differential thermal conductivity detector.

This procedure is based on the following reference documents:

- EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.

- Dixon, Wilfrid J., and Massey, Frank J. Jr.: Introduction to Statistical Analysis (fourth edition). Edited by Wilfrid J. Dixon. McGraw-Hill Book Company, New York, 1983. P377 and P548.

If the laboratory's SOP has been modified from the above referenced document, a list of modifications is provided in Section 16.0 of this SOP.

3.0 Definitions

A list of general laboratory terms and definitions are provided in Appendix A.

4.0 Interferences

Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias.

Maintaining the sample at 4°C, analyzing the sample within the specified holding time, and analyzing the wet sample, may minimize bacterial decomposition and volatilization of the organic compounds.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 Equipment and Supplies

- Drying Oven: Capable of maintaining a temperature of 105 ±2°C.
- Carlo Erba Elemental Analyzer Model EA1108 and Model NA 1500 or equivalent.
- Costech Elemental Analyzer: Model 4010 or equivalent.
- Analytical Balance: Capable of weighing to the nearest 0.001mg.
- Aluminum Weigh Boats.
- Tweezers
- 5mm X 9mm tin capsules
- Quartz Columns: Costech Analytical or equivalent.
- Quartz wool: for segregating and containing column materials
- Copper Wire, Reduced: Costech Analytical or equivalent.
- Tungsten on Alumina: Costech Analytical or equivalent.
- High Temperature Gloves
- Clear Plastic Sample Trays: Costech Analytical or equivalent.

7.0 Reagents and Standards

7.1 Reagents

- Reagent water
- Phosphoric Acid, Concentrated: Reagent Grade, J.T. Baker recommended.

Phosphoric Acid Solution (1:19): Add approximately 100 mL of reagent water to a 200 mL volumetric flask. Add 18.34 g of concentrated phosphoric acid to the volumetric flask then adjust to volume with reagent water. Mix the solution well then transfer the solution to a 250 mL polyethylene bottle. Assign an expiration date of six months from date made and store the solution at room temperature.

7.2 Standards

- Acetanilide Crystals of known Carbon percentage: Purchased from Costech Analytical. Used to check instrument calibration.
- Sulfanilamide Crystals (41.84% Carbon): Purchased from Costech Analytical. This material is used to calibrate the instruments.
- Laboratory Control Samples (LCS) Material, Organic Material of known Carbon percentage: Purchased from LECO Corporation.
- Matrix Spike Material, 1632B trace elements in coal (76.86% Carbon)

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so sampling procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are the recommended minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Solids	Amber glass	10 g	Chilled to ≤ 4°C	14 Days	TOC by Lloyd Kahn

[1] Holding time is determined from date of collection.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance

		Criteria
Method Blank (MB)	1 in 20 or fewer samples	< RL
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	%R (85-115)
Sample Duplicate (DP)	Client Request	RPD (≤ 20)
Matrix Spikes (MS)	Client Request	%R (85-115)

9.2 Instrument QC

The laboratory analyzes the following instrument check standards:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initial Method Set-Up, after combustion chamber is changed (approx. every 200 drops)	Correlation coefficient must be >0.995
Calibration Verification (Acetanilide)	Every 20 drops and at the end of the analytical sequence	%R (85-115)
Calibration Blank (CCB)	After every acetanilide	<RL

10.0 Procedure

10.1 Calibration

Analyze a calibration curve each time the combustion column is changed. Change the column after 200 drops or when you experience result issues or odd peak shapes or baseline issues. The column change procedure is provided in Appendix C.

The recommended formulations for each calibration level are provided in the following table:

Calibration Standard Sulfanimide	Weight[1] (mg)	% Carbon	Carbon (mg)
Calibration Level 1	0.100	41.84	0.0418
Calibration Level 2	0.500	41.84	0.2092
Calibration Level 3	1.00	41.84	0.4184
Calibration Level 4	1.50	41.84	0.6276
Calibration Level 5	1.75	41.84	0.7322

[1]These weights are approximate. Enter the actual weight used into the software program.

Measure a single drop for each calibration point. The instrument software system plots peak area against mg of Carbon and calculates a correlation coefficient using standard linear regression. The correlation coefficient (r) must be $\geq$ 0.995 for the calibration to be considered acceptable. If it is not, repeat the calibration prior to further analysis.

1.0 Troubleshooting

- Calibration passes at > 0.995 correlation, but LCS fails abnormally low: Re-calibrate. Calibration usually needs to be > 0.999 correlation.

- Carbon peak “maxes out” at instrument 1200mv (peak has flat top): Reanalyze sample at lower weight.

- No peaks on any chromatograms, no results: Gases to instrument may be off. Turn on all gasses at valve manifold.

- Autosampler will not work at all: Gasses to instrument may be off. Turn on all gasses at valve manifold.

- Single chromatogram shows results at bottom of page, but no peak or baseline in chromatogram window: Re-print single chromatogram.

- Some or all chromatograms show carbon peak at same retention time as Acetanilide, but peak is not identified as carbon, or is identified as another element: Retention time shifted. Adjust retention time in calibration window, and reprint chromatograms.
- Upon recalibration, peaks are not being identified as carbon: In calibration window, general tab, adjust retention time to match peaks. Starting at level 1, “Open Standard”, open level1 curve pt. in calibration directory, click “Add Peak” button, click on peak itself. Increase level #, opening standard for each curve pt and add each peak. Carbon Tab should have all five calibration points on curve, if done correctly.
- Peaks in chromatograms identified as carbon, but all results in summary table below chromatogram are zero: Current calibration not associated with run when started. Open current calibration, copy first two columns for all points (5 rows) in small table in general tab. Then, open calibration that was associated with run (should be empty) and paste into table in calibration tab. Reprint all chromatograms on run.
- Software crashes during analysis: Boot up software normally. Chromatograms already printed/analyzed are ok, but, sample that was analyzing during shutdown is lost. Restart table at next sample by un-checking “run” box for samples already run and sample that was lost.

- Autosampler error causes few samples to remain in autosampler tray after run has finished: Identify samples that got stuck. Create a new run and analyze stuck samples (with initial weights) with bracketing QC. No PBS/LCS needed.
- Autosampler error causes many sequential samples to remain in autosampler tray after run has finished (usually end of run): Add rows onto existing table. Identify samples that did not get analyzed and repeat Ids and weights into added rows. Restart table. All analyzed samples’ status should be blue (analyzed), added rows should be green (not analyzed yet).
- Various result issues or odd peak shapes or baseline issues: Column may be leaking or cracked. Change column, recalibrate.

10.3 Sample Preparation

Using tweezers, and working directly from the box, place a tin capsule on the analytical balance and tare the balance. Using the small sample scoop, add approximately 10 mg (or the project specified sample weight) of sample to the capsule. Record the actual sample weight used on sample preparation log. Remove the capsule from the balance and place into one of the aluminum holding trays. Weigh two additional portions of sample into two separate tin capsules for each field sample.

To prepare the method blank, set two empty tin capsules into an aluminum holding tray.

To prepare the LCC, weigh ~9 mg of the LECO LCS material into two separate tin capsules and set them in sequence in an aluminum holding tray.

For the matrix spike, weigh out an additional sample aliquot and record its weight. Add 0.3 – 0.7 mg of matrix spike material and record this weight.

For the sample duplicate, weigh out an additional sample aliquot. Prepare two aliquots for both the matrix spike and the sample duplicate.

Add two drops of 1:19 phosphoric acid to each tin capsule. Place the aluminum trays into a drying oven set to a temperature of 105ºC for 30-60 minutes or until all samples appear dry.

Using tweezers pinch the top of each tin capsule closed and compress the capsule around the material inside. Work carefully so as not to tear the capsule, but crush it down to the smallest size. Set the prepared samples in line in a clear plastic sample tray for storage, or place directly into an autosampler tray for analysis. For the latter, leave positions open for the acetanilide check standards and associated calibration blanks.

Prepare the acetanilide standard and blanks as follows:

For each acetanilide spike, weigh ~0.5 mg of acetanilide material into a tin capsule. Fold the capsule up and compress down to the smallest size possible. Prepare enough acetanilide to ensure a frequency of every 20 drops and the end of the analytical sequence. For each associated calibration blank, leave an empty position in the autosampler tray.

Software Set-up and Analysis

If the column has been changed generate a new calibration curve. If not, use the existing calibration curve for analysis. Each column will analyze approximately 200 individual sample drops. When the counter on the instrument approaches 200, watch the instrument data for signs that the column is deteriorating; poor peak resolution, trailing baselines, extraneous peaks. If a column change is necessary, refer to Appendix C for the procedure. After changing the column, generate a new calibration curve.

Select the appropriate channel: Channel 1 is the NA 1500, Channel 2 is the EA 1108, and Channel 3 is the Costech instrument, which has its own PC. At the main screen select the sample table icon. The last sample table that was run will be shown on the screen.

Open a new sample table, and select the appropriate number of sample positions for the analysis, then name the table with the date and a unique alpha designator (i.e. 061505a). In front of the %3r in the file name column of the sample table, add the sample table name to ensure that each individual chromatogram generated from this sample table has a unique filename associated with it.

If the combustion column has been changed and instrument needs to be calibrated, follow the procedure below:

Prepare a “bypass” drop to determine the retention time for carbon with the new column. The bypass is an aliquot of acetanilide. The weight is not needed. Drop the bypass into the instrument and initiate a singular analysis. Set the retention time for carbon in the software to match that of the bypass drop.

Identify the first five sample lines with the names Std1 through Std 5. Enter their respective weights in the weight column, assign them a level # in the level column (Std1 is level 1, Std2 is level 2, etc.) to alert the software the order in which to place the calibration standards. In the sample type column, use the drop down and select “standard” for each. Finally, use the drop down in the Standard name column and select “sulfanilamide” for each. Add the standards to the autosampler tray and hit “start” to run the calibration.

Sample Analysis:

Open a new sample tray and create a unique file name. When the instrument was last calibrated, the software creates a calibration file with the same name as the sample table in which it was run. Open this file and save it with the same name as the sample table about to be run to ensure that the analysis is calculated from the most recent calibration. To do this, click on the calibration icon (looks like a little calibration curve) and use the file option to open the calibration file last performed. Save this file with the same name as your sample table. Click on the sample table icon (looks like a little sample table) to get back to your sample table.

Enter each sample ID and their respective weights and save the sample table. Enter a weight of 10 mg for the Method Blank (PBS) and instrument blanks.

An example analytical sequence follows:

Initial Calibration (calibration blank and 5 calibration standards)

Acetanilide	(1 drop)
Blank	(1 drop)
PBS	(2 individual drops)
LCS	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Acetanilide	(1 drop)
Blank	(1 drop)

Add the samples and acetanilides to the autosampler tray and set the tray into the autosampler carriage. Turn the autosampler tray until the number 1 position is behind the post, in front of the autosampler. The tray is now set to run.

Click the “start” icon to begin the analysis.

After analysis review the analytical results against the acceptance criteria given in Table 2, Section 18.0, and perform corrective action as necessary. Report results in mg/kg Carbon and corrected for % solids.

11.0 Calculations / Data Reduction

11.1 Calculations

11.2 Percent Carbon to mg/kg Carbon Conversion

% Carbon × 10,000 = mg/kg Carbon

11.3 LCS Percent Recovery (%R)

$$\%R = \frac{\text{LCS Result}}{\text{LCS True Value}} \times 100$$

11.4 MS Percent Recovery (%R)

$$\text{mg/Kg wet SA} = \frac{\text{Spike TV} \times \text{weight of MS added}}{\text{sample weight}} \times 1\text{million}$$

$$\text{mg/Kg dry SA} = \frac{\text{mg/Kg wet SA}}{\%\,\text{solid}} \times 100$$

$$\text{mg/Kg dry Carbon} = \frac{\text{mg/Kg wet Carbon (from instrument)}}{\%\,\text{solid}} \times 100$$

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

Where:
A= Average of two drops of MS sample result: mg/Kg dry carbon
B= Average of two drops of parent sample: mg/Kg dry carbon
C= Average of two drops of mg/Kg dry SA
SA= spike added (mg/Kg)
Spike TV= 0.7686 (mg/Kg)

11.5 Relative Percent Difference (RPD)

$$RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} \times 100$$

Where:

D_1 = First Sample Value

D_2 = Second Sample Value (duplicate)

11.6 Dixon Test (Use 3-7 results)

1. Sort all the results in ascending order (low values to high).
2. Calculate the tau statistic for the low and high values.
3. Compare the calculated tau statistics (low and high) to critical values listed below.
4. If either calculated tau is higher than the critical value, reject that value and repeat the test.

Tau statistic for lowest value = $\tau_L = (X_2 - X_1) / (X_k - X_1)$
Tau statistic for highest value = $\tau_H = (X_k - X_{k-1}) / (X_k - X_1)$

Where:
X_2 = Second lowest value in sorted list.
X_1 = Lowest value in sorted list.
X_k = Highest value in sorted list.
X_{k-1} = Second highest value in sorted list.

Number of observations, k	Critical Values
3	0.941
4	0.765
5	0.642
6	0.560
7	0.507

11.2 Data Review

11.2.1 Primary Data Review

Evaluate and QC samples against the acceptance criteria given in Table 2. Perform the recommended corrective action as necessary. If corrective action is not taken or is not successful, initiate a nonconformance report (NCR) to document the situation.

11.2.2 Secondary Data Review

Spot-check the calculations using the equations given in Section 11.1.

Verify that the performance criteria for the QC items listed in Table 2 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If corrective action was not taken or is unsuccessful, ensure the situation is documented with a nonconformance report (NCR) and ensure data is qualified accordingly. Report the nonconformance in the narrative note program.

11.3 Data Reporting

Report analytical results above the adjusted reporting limit (RL) as the value found. Report analytical results less than the adjusted RL with a "U" data qualifier. Adjust the RL for sample dilution/concentration and dry weight. The laboratory's routine reporting limit (RL) for TOC is 1000 mg/Kg based on a 10.0 mg sample mass, assuming 100% solids. Unless otherwise specified for the project, report all soils in dry weight.

Review project documents such as the environmental test request (ETR) analytical worksheets, Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Generate the data report in the deliverable format specified by the laboratory PM and release the report to report management.

Retain, manage and archive electronic and hardcopy data as specified in laboratory SOP BR-QA-014 Laboratory Records.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when time there is a significant change in instrumentation or procedure.

Each analyst that performs this procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training, BR-QA-011.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

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

14.0 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 BR-EH-001 *Hazardous Waste*.

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

- Caustic waste – 2.5 L glass satellite container.
- Acidic Waste - 2.5L glass satellite container

The satellite containers are labeled “Hazardous Waste” along with the type of waste category generated. Authorized personnel routinely transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations.

15.0 References / Cross-References

- EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.
- Dixon, Wilfrid J., and Massey, Frank J. Jr.: Introduction to Statistical Analysis (fourth edition). Edited by Wilfrid J. Dixon. McGraw-Hill Book Company, New York, 1983. P377 and P548.
- Corporate SOP CW-E-M-001 Corporate Environmental Health and Safety Manual
- Laboratory SOP BR-QA-005, Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).
- Laboratory SOP BR-QA-011 Employee Training
- Laboratory SOP BR-EH-011 Hazardous Waste
- Laboratory SOP BR-QA-014 Laboratory Records
- Laboratory Quality Assurance Manual (QAM)

16.0 Method Modifications

The laboratory procedure is modified from the reference method as follows:

Modification Number	Method Reference	Modification
1	TOC by Lloyd Kahn	The laboratory analyzes two drops per sample and if the RPD is greater than 40% the Dixon test is utilized.

17.0 Attachments

- Table 1: Primary Materials Used
- Table 2: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: TOC Procedure for High Concentration Marine Sediments (CITHON)
- Appendix C: Column change procedure
- Appendix D: Determination of Black Carbon in Sediment Procedure

18.0 Revision History

BR-WC-008, Rev. 12:

- Section 11.2: The procedure for evaluating data using the low-level spreadsheet was removed.

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Following each column change	correlation coefficient $\geq$ 0.995	Standards check, re-calibration
Acetanilide	Every 20 drops and at the end of the analytical run	%R (85-115)	Re-prepare and reanalyze samples not bracketed by passing standard.
Blank (paired with Acetanilide)	Following each Acetanilide	< RL	Re-prepare and reanalyze batch.
Method Blank (MB)	Once per batch of 20 samples	< RL DoD: ½ RL	Re-prepare and reanalyze batch.
LCS	Once per batch of 20 samples	%R (75-125)	Re-prepare and reanalyze batch.
Sample Duplicate (DP)	One per batch of 20 or less samples	RPD (≤ 20)	Discuss outlier in project narrative
MS/MSD	One per batch of 20 or less samples	%R (75-125)	Discuss outlier in project narrative
Sample precsion	Each sample is run in duplicate	%RPD<40%	Analyze 2 more replicates and perform Dixon test for high and low outliers. Include Dixon spreadsheet in the data package and narrative note results.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Terms and Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration: the establishment of an analytical curve based on the absorbance, emission intensity or other measured characteristic of known standard.

Calibration Standards: a series of known standard solutions used to calibrate the instrument response with respect to analyte concentration. A standard containing the analyte in question (sulphanilimide) is prepared at varying weights and analyzed. This standard is a separate source from the LCS. The sulphanilimide is used to calibrate the instrument response with respect to analyte concentration.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Appendix B: Marine Sediments High in Inorganic Carbon

Sample Preparation

Transfer approximately 10 g of a thoroughly mixed sample to an aluminum weigh dish, and dry in the 105°C oven. Grind the sample with the pink mortar and pestle to a fine powder. Record the weight of a 250 mL Teflon beaker then transfer ~ 5 g of the ground sample to this beaker.

If the sample is to be spiked, weigh the beaker to the nearest 0.1mg and record the weight. Likewise determine and record the weight of the added sample. Add 0.1g of NIST 1632b Trace Elements in Coal (80.11% Carbon) to the sample. Record the weight added. Evenly distribute the spike over the sample and use a glass stir rod to mix the spike with the sample. Do not use that stir rod with any other sample.

Use Talc-free latex gloves from this point on to minimize the risk of acid burns. Add several drops of 1:1 HCL to each sample and stir each sample with its own glass stir rod. Carefully rinse the stir rod and beaker walls with DI water using a fine-tipped squirt bottle. Use only what is needed to bring the entire sample to the bottom of the beaker. ***When adding water to acid use necessary precautions to avoid splashing!*** Samples with high concentrations of inorganic carbon may effervesce to the point of overflowing the beaker, so take care to add the acid in small aliquots and stir vigorously. If the sample "boils over" it must be re-prepared. Continue to add 1:1 HCL in small aliquots until there is no further reaction, taking sample to dryness after each addition of acid in a 105-degree oven.

Dry the treated samples in the oven after each acid/water addition. Do not add more than a total of 200 mL of 1:1 HCL to any sample.

NOTE*: Samples are hydroscopic and will absorb water if they are exposed to air for too long.*

Weigh beaker with residue and record the residue weight measurement. After the sample is thoroughly dry, scrape the sample residue from the beaker and grind to a powder using the pink mortar and pestle. Transfer the ground sample to a clean, dry 40-mL vial reserved for this analysis.

NOTE: *Depending on the nature of the sample, it may be difficult to completely remove the dried residue from the beaker or to grind it to a homogenous powder. Where difficulties are encountered, make a note on the preparation worksheet.*

Analysis

Perform TOC analysis on processed sample material as outlined in section 10.0 of this SOP.

Appendix C: Column Change Procedure

Turn off the helium and oxygen supplies to the instrument.

Dial the left furnace temperature to a reading of 052 (this equates to 520˚C). Wait until the temperature drops below 600˚C to remove the column.

Remove the panel covering the furnace and unscrew the autosampler connection from the top of the column.

Unscrew the fitting at the bottom of the column and remove.

Lift the column up and out of the furnace using high temperature gloves.

CAUTION: The column will still be 500-600˚C. Do not touch the center portion of the column. Place the spent column in the metal can designated for this purpose.

Lay a new quartz column on the bench top, measure and mark off for the following:

- One inch up from the bottom and add a ½ inch plug of quartz wool. Note: pack the quartz wool tightly enough for it to stay in place.
- Pour in 2 ½ inches of copper wire
- Pack another ½ inch quartz wool plug on top of the copper
- Pour in 3 inches of tungsten
- Pack a final ½ inch quartz wool plug on top of the tungsten

Place the new column into the furnace and reconnect the top and bottom fittings. Snug these up, but don't over tighten.

Replace the panel covering the furnace, dial the furnace temperature back to 102 (this equates to

1020˚C), and turn the helium and oxygen supplies back on.

When the instrument comes up to operating temperature, it is ready to calibrate.

Appendix D: Determination of Black Carbon in Sediment Procedure

1. Obtain a representative subsample of the sediment. Weight 10 grams of sample into a clean pre-tared aluminum drying pan or equivalent.

2. Dry the sample at 105^{o}C for at least 12 hours.

3. Grind the sample using a mortar and pestle.

4. Sieve the sample using a number 35 sieve (500 um).

5. Treat the sample with phosphoric acid. Add acid drop wise until effervescence is no longer observed.

6. Dry the sample at 105^{o}C for 1 hour.

7. Set aside an aliquot of the sample at this stage for direct TOC analysis, reported without correction for the IN623 percent solids. Continue with the sample for Black Carbon.

8. Place the dried sample into a clean crucible and cover the sample.

9. Bake the samples at 375^{o}C in a muffle for 24 hours or until the LCS is +/- 50% of the true value.

10. Allow the samples to cool and transfer approximately 5.0 mg into each of two tin capsules.

11. Transfer the sample (in the tin capsules) to the TOC analyzer for analysis by the Lloyd Kahn Method.

12. The sample is pyrolyzed in an inductive type furnace, where the carbon is converted to carbon dioxide, which is measured using a differential thermal conductivity detector.

13. The results will be reported as mg/Kg Black Carbon.

Note: Black carbon LCS material: NIST Standard Reference Material 1944 New York-New Jersey Waterways Sediment.

References:

Orjan Gustafsson, Thomas D. Bucherli, Zofia Kukulska, Mette Andersson, Claude Largeau, Jean-Noel Rouzaud, Christopher M. Reddy and Timothy I. Eglinton (December 2001) Evaluation of a Protocol for the Quantification of Black Carbon in Sediments, Global Biogeochemical Cycles, Volume 15, pages 881-890.

Orjan Gustafsson, Farnaz Haghseta, Charmaine Chan, John MacFarlane & Philip M. Gschwend (1997) Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability, Environmental Science & Technology, Volume 31, pages 203-209.

Title: Particle Size Analysis (ASTM D 2217 and D422-63)

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Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of particle size distribution in soils.

2.0 Summary of Method

A portion of sample is soaked in a dispersing agent then partitioned into separate portions, material retained on a #10 sieve and material passing the #10 sieve. The material retained on the #10 sieve is dried to constant weight then passed through a large size sieve stack; the material retained on each sieve is measured and recorded. Material passing the #10 sieve is subject to hydrometer analysis then passed through a small size sieve stack, the material retained on each sieve is measured and recorded. All measurements, large and small sieves and hydrometer readings and the hygroscopic moisture are used to establish the particle size distribution of the sample.

This SOP is based on the following reference methods:

- ASTM Standard D 2217 – 85 (Rapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

NOTE: ASTM D2217 was method was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard.

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

Not Applicable

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Not Applicable

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Top-Loading Balance, capable of weight measurement to 0.01 g
- Mechanical Stirring Device and Dispersion Cup
- Thermometer: Accurate to 0.5°C
- Mortar and Rubber Tipped Pestle
- Sedimentation Cylinder(s) 1000 mL
- Hydrometer: ASTM 151H in specification E 100.
- Sieves, of the following size(s): Gilson Company, Inc. or equivalent
  3.0” (75.00 mm)
  2.0” (50.00 mm)
  1.5” (37.50 mm)
  1.0” (25.00 mm)
  3/4" (19.00 mm)
  3/8” (9.50 mm)
  # 4 (4.75 mm)
  #10 (2.00 mm)
  #20 (850.0 um)
  #40 (425 um)
  #60 (250.0 um)
  #80 (180.0 um)
  #100 (150.0 um)
  #200 (75.0 um)
- Drying Oven with temperature range of 60-110°C
- Stainless Steel Spatulas & Spoons
- Metal & Bristle Brushes
- Ro-Tap Sieve Shaker, W. S. Tyler or equivalent.
- Timing Device with second hand and capable of counting up to 25 hours

7.0 Reagents and Standards

- Reverse Osmosis (RO) water: In-House System
- Sodium Hexametaphosphate: ELE International or equivalent.

Sodium Hexametaphosphate Solution: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon bottle. Add a stir rod to the container and place on a stir plate. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made

unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Solid	Glass Jar w/ Teflon Lid	500 g	None	None	ASTM D422-63

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Equipment Calibration

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

NOTE: The QA Manager or her designee checks the calibration of liquid in glass thermometers annually against a NIST-traceable thermometer following the procedures given in laboratory SOP BR-QA-004. Electronic / digital thermometers that are battery-operated are checked quarterly using the same procedure.

Calibrate the hydrometers every two years following the procedure given in BR-GT-008.

Calibrate the sieves 6 months following the procedure given in BR-GT-008.

Calibrate the Ro-Tap sieve shaker every 12 months following the procedure given in BR-GT-008.

10.2 Hygroscopic Moisture Determination

Label an aluminum pan with the Lab ID for each sample. Tare the balance, weigh each pan and record the weight measurement in the spreadsheet.

Mix the sample with a stainless steel spatula. Measure at least 10-15 g of each sample into the labeled aluminum pan and record the weight of sample in the spreadsheet.

Place the pan + sample in an oven maintained at a temperature of 110ºC and dry the sample for at least 16 hours. Reweigh each pan and record the weight measurement in the spreadsheet.

Percent solids are calculated using the equation given in Section 11.0.

10.3 Sample Preparation

Use the calculated percent solids and the sample characteristic for each sample to determine the amount needed for analysis using Table 2. For example, if the calculated percent solids for a sample are 50% and the sample characteristic is sand, use 200 g for analysis. If there is an insufficient amount of sample available, initiate a nonconformance memo (NCM) and contact the PM for further instruction.

Place a 1000 mL plastic beaker on the balance and tare the balance. Weight the amount of sample for analysis and record the weight in the bench sheet.

Add 125 mL of sodium hexametaphosphate solution to each beaker. Stir to mix and soak the sample in this solution for 16 hours

10.4 Sample Partition

Rinse the sample slurry into a dispersion cup using reagent water. Fill the dispersion cup ½ full with reagent water and place the cup on the blender to mix for one minute.

NOTE: Some samples may not be amendable to using the blender examples include but not limited to large gravel, sands, or organic material. If the sample is not amenable, initiate a NCM to notify the PM of the anomaly and proceed to the next step without blending the sample.

Place a #10 sieve on a 1000 mL graduated cylinder. Pour the sample through the sieve. Rinse the dispersion cup with reagent water and pour the rinse through the sieve. Repeat until transfer is complete. Bring the volume in the graduated cylinder to 1000 mL with reagent water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature in preparation for hydrometer analysis.

Label a medium size aluminum dish with the sample's LAB ID then transfer the sample material that was retained on the #10 sieve to the dish. Place the aluminum dish in the drying oven set at 110 ± 5° C and dry the sample material for at least 16 hours or until constant weight. Set aside for sieve analysis.

10.5 Hydrometer Analysis

Prepare a hydrometer rinse bath by adding 1000 mL of reagent water to a 1000 mL graduated cylinder

Record the hydrometer ID and start time on the worksheet. Set the timer for the elapsed time and perform each task as listed in Table 1: Hydrometer Reading Table.

To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait ~ 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the worksheet. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the worksheet. Rinse the temperature probe in the hydrometer rinse bath.

Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes as per Table 1 then proceed to small sieve analysis.

10.6 Sieve Analysis

Inspect the sample material in the aluminum pan and record a description of the non-soil material (e.g.- sticks, grass, wood, plastic), hardness of material and shape of material in the worksheet.

Hardness qualifiers include hard, soft or brittle. Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

Large Sieves

Weigh the 3/4", 3/8", #4 and #10 sieves and enter the weight measurements in the worksheet as the tare weight.

Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 10 minutes.

Weigh each sieve and record these measurements in the worksheet.

Small Sieves

Transfer the sample from the graduated cylinder to a #200 wet wash sieve. Wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve to a 250 mL glass beaker labeled with the sample's LAB ID.

Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh the sieve stack sized between #20 and #200 and record the tare weights.

Transfer the sample to the sieve stack and ensure complete transfer. Use hair or wire brushes to clean the beaker. Place the sieve stack on the Rotap machine and shake for ten minutes.

Weigh each sieve and record these measurements in the worksheet.

11.0 Calculations / Data Reduction

11.1 Calculations

Sample Used (SU): Dry Preparation

$$SU = (pan + dry\ sample\ -\ pan) - (pan + non\ -\ soil\ material\ -\ pan) \otimes HMCF$$

Where:
HMCF = Hygroscopic moisture correction factor.

Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow
Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample

passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

11.2 Data Reduction

11.2.1 Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

11.2.2 Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

11.2.3 Lab Complete

Review the batch, run QC checker as appropriate and set the status to lab complete.

11.2.4 Data Reporting

Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

11.2.5 Data Archival

Data are stored in the laboratory's LIMS system.

12.0 Method Performance

Not Applicable

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide

by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 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 BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)
- Liquid Waste- 55 gallon poly drum

15.0 References / Cross-References

- ASTM Standard D 2217 – 85 (Reapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

16.0 Method Modifications

D2217: The laboratory performs sample portioning after soaking the solution in the dispersing agent because the dispersion agent helps break up aggregates associated with clay and sediments.

D422: The laboratory does not always use the recommended amount of sample for analysis because sufficient sample volume is not always received.

17.0 Attachments

- Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)
- Table 2: Percent Solids Table for Weight Determination for D422.

18.0 Revision History

BR-GT-006, Revision 6:

- Title Page: Updated approval signatures
- All Sections: Removed references to dry preparation by ASTM D421; Added procedure for wet preparation.
- Attachments: Inserted Percent Solids Table

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406

0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document

Table 2: Percent Solids Table for Weight Determination for D422.

Percent Solid Table

Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

Title: Organotins by Gas Chromatography (GC)

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Facility Distribution No. Electronic **Distributed To: Facility Intranet**

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1.0 Scope and Application

This SOP describes the laboratory procedure used to determine the concentration of organotins in environmental samples using dual column gas chromatography with flame photometric detectors (GC/FPD).

1.1 Analytes, Matrices, and Reporting Limits

This procedure may be used for a variety of matrices including: water, soil, sediment, waste and tissue.

The list of target compounds that can be determined from this method along with the associated reporting limits (RL) is provided in Table 1.

2.0 Summary of Method

3 uL of extract is injected into a dual capillary column gas chromatograph equipped with flame photometric detectors (GC/FPD). Organotins are quantified using external standard technique.

This procedure is a laboratory developed method derived from the NOAA Status and Trends Program Document: Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Vol. IV, NOAA Technical Memorandum, NOS ORCA 71.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

- Method interference may be caused by contaminants in the extraction solvent. Solvents should be stored away from possible sources of contamination.

- Matrix interferences may be caused by contaminants co-extracted from the sample. The extent of the interferences will vary depending on the nature and diversity of the samples.

- Each lot of hexyl-magnesium bromide used during the extraction procedure for derivitization should be tested by the GC department prior to its use to ensure that it is free of contamination.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst must be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

Table 2 lists 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.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

- Autosampler Vials, National Scientific or equivalent.
- Hydrogen Generator: Parker Balston.
- Volumetric Syringes, Class “A” (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

6.2 Analytical System

- Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5 or higher.
- GC/FPD: with dual columns, dual FPDs, and auto-sampler capable of a 3-µl injection split onto two columns: HP 7673A, HP 5890 with Leap Technology CTC A200SE and A200S Fisons autosamplers, Agilent Technologies 6890N with 7683 Series injector, or equivalent.
- GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses:
    - RTX-35, (30m x 0.32 mmID x 0.25um)
    - RTX-5, (30m x 0.32 mmID x 0.25um)

Equivalent columns may be used, provided the elution orders are documented and compound separations are maintained.

7.0 Reagents and Standards

7.1 Reagents

- Hexane, Ultra-Resi Analyzed, JT Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The extractions laboratory prepares all calibration standards, CCVs and ICVs. The recommended formulation for the standards used in this procedure is provided in the extraction SOP. The final concentration of each calibration level is provided in Appendix B of this SOP.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are the recommended sample amounts needed for analysis and size, preservation and holding time requirements:

Matrix	Sample Container	Recommended Sample Size	Preservation	Extract Holding Time[1]	Reference
Water	Glass	1 L	Chilled to 4°C (±5°C)	40 Days	Lab
Solid	Glass	50 g	Chilled to 4°C (±5°C)	40 Days	Lab
Tissue	Glass	50 g	-15°C (±5°C)	40 Days	Lab

[1]Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3
Matrix Spike(s) MS/MSD	1 pair per extraction batch when sufficient sample volume is provided or per client request	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Table 3
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 3
Continuing Calibration Verification (CCV)	Daily, every 10 samples, end of sequence	See Table 3
Retention Time Windows	As Needed	See Table 3

10.0 Procedure

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column into the injection port and connect the guard column to the separate analytical columns using a glass "Y". The analytical columns are installed into independent FPD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature:	100°C for 3 minutes
Temperature Program:	20°C per minute to 290°C Hold for 1 minute.
Detector Temperature	290°C
Injector Temperature:	225°C
Injection volume:	3µL
Carrier Gas:	Helium flow set at 6.0-10.0 mL/min
Makeup Gas:	Hydrogen and zero air (each supplied by gas generators) should be optimized for sensitivity, but is generally set at approximately 175 mL/min total flow

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

10.2 Retention Time Window Establishment

Whenever a new GC column is installed, establish RT windows for each analyte by analyzing three standards over a 72-hour period and calculating the mean RT and Standard Deviation (SD). Calculate the RT window as the mean RT $\pm$ 3SD. If the SD is <0.01 minutes, a default SD of 0.01 minutes may be used.

If this procedure results in a RT window that is too tight, favoring false negatives, the laboratory may opt to use an alternate method to determine the RT windows. An alternate method consists of using a RT window of $\pm$ 0.05 minutes. The center of the RT window is set at the midpoint calibration level in the initial calibration sequence. RT windows are then updated daily (minimum frequency), re-centering the windows on the retention times established in a CCV.

10.3 Instrument Calibration

10.3.1 Initial Calibration (ICAL)

Before initial or daily calibration, inject an instrument blank (IBLK) consisting of hexane to bring the GC/FPD system online.

The instrument is calibrated using a minimum of five different concentration levels for each target analyte. The calibration standards are prepared and derivatized following the procedures given in the extraction SOP. Prepare calibration standards for analysis by adding 10 uL of internal standard (tetra-n-propyltin) to 100 uL of standard in an autosampler vial insert. Cap the vials and place on the autosampler tray. Enter the standard names into the acquisition software. Set the autosampler to inject 3-µl of each calibration standard. Start the acquisition process and autosampler.

The data processing system calculates the Response Factor (RF), mean RF and Percent Relative Standard Deviation (%RSD) for each analyte on both columns. The %RSD for each target analyte must be less than or equal to 20% in order to use the mean RF for quantification. If this criterion is not met, use another suitable quantification method for that analyte or correct the problem and repeat the calibration. Once a method of quantification is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is performed.

Alternate Quantification Option:

Linear Regression & Weighted Linear Regression: Generate a curve of concentration vs. response for each analyte and calculate the correlation coefficient. The calibration must have a correlation coefficient (r) $\geq$ 0.995 (or $r^2 \geq$ 0.990). If this criterion is not met, correct the problem and repeat the calibration. The use of linear regression requires a minimum of 5 calibration points.

10.3.2 Second Source Calibration Verification (ICV)

Immediately after each calibration and prior to the analysis of QC or field samples, verify the accuracy of the initial calibration by analyzing a second source ICV.

The ICV is prepared and derivatized following the procedures given in the extraction SOP. Prepare the ICV for analysis by adding 10 uL of internal standard (tetra-n-propyltin) to 100 uL of ICV in an autosampler vial insert. Inject 3 µl of the ICV standard onto the instrument in the same manner as performed for the initial calibration standards.

The percent recovery of each analyte must be within $\pm$ 25% of the expected value (%R: 75-125). If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards or ICV standard and/or perform instrument maintenance and recalibrate. The QC acceptance criteria must be met on both columns.

10.3.3 Continuing Calibration Verification (CCV)

CCVs are prepared and derivatized following the procedures given in the extraction SOP. Prepare CCVs for analysis by adding 10 uL of internal standard (tetra-n-propyltin) to 100 uL of CCV in an autosampler vial insert. Inject 3 µl of the CCV standard onto the instrument in the same manner as performed for the initial calibration standards.

Analyze a CCV at or below mid-calibration level each day before sample analysis, after every ten injections and at the end of each analytical batch to monitor instrument drift. Calculate the RF and percent difference or drift (Appendix C) for each analyte on both columns. The percent difference or drift must be within ±25% for each analyte. Compare the RT of each analyte in the CCV with the established RT windows; the RT must be within the established window (refer to section 10.2). The acceptance criteria must be met on both columns.

If the CCV fails, it may be repeated once. If repeat analysis fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples must be bracketed by passing CCVs. Samples analyzed before and after CCV failure must be reanalyzed, unless the CCV is high and there are no detects in the associated samples.

10.4 Troubleshooting

Check the following items in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if the chromatography has degraded as evidenced by peak shapes.

- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column and remove one or more loops from each analytical column.

- Needle crushed during injection - Replace the needle and check the injection port for obstructions and check the autosampler for misalignment.

- Auto-sampler failure - Reset the auto-sampler.

- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

10.5 Sample Preparation

Remove the sample extract from refrigerated storage and warm to room temperature.

Prepare samples for analysis by adding 10 uL of internal standard (tetra-n-propyltin) to 100 uL of sample in an autosampler vial insert. Cap the vials and place on the autosampler tray. Enter the sample ID's into the data acquisition program.

10.6 Sample Analysis

Arrange the samples in a sequence that begins with the calibration standards and ICV followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs).

Enter the standard and sample names into the data acquisition program in the order the samples were placed in the autosampler tray and initiate the analytical sequence. Set the autosampler to inject 3-µl of each standard and sample onto the instrument.

An example analytical sequence that includes initial calibration (ICAL) and subsequent sample analysis is provided below.

Injection Number	Lab Description
1	Instrument Blank
2	50ppb Tin Standard
3	100ppb Tin Standard
4	250ppb Tin Standard
5	500ppb Tin Standard
6	1000ppb Tin Standard
7	Instrument Blank
8	ICV
9 - 18	10 injections
19	CCV (250ppb Tin Standard)
	Repeat steps 9 -19

Cleaning blanks (IBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention times of the peaks to the established retention time windows (refer to section 10.2).

Review and accept or reject the qualitative identifications made by the data processing software using the following guidelines:

Compare the retention times of the peaks to the established RT windows (refer to section 10.2), taking into account the shift of the surrogate peak. If the surrogate peak has shifted, open the retention time window in the direction of the shift. The processing software identifies the peak in the retention time window that is closest to the expected retention time set in the Target method, so the peak may need to be re-identified if a shift has occurred.

Look for shoulders on large peaks that may be the peaks of interest. The processing software does not always automatically integrate the shoulder off of the larger peak, so manual integration (split) of the shoulder may be necessary.

Each target analyte must be detected above the reporting limit on each column for qualitative identification to be made.

11.2 Quantitative Identification

The data system calculates the corrected concentration for each target analyte from the calibration curve using the equations given in Appendix C. If sample interference is suspected, the laboratory may choose to report the value from the result that is not affected by interference. The lower value between the two columns is reported unless otherwise specified for the project.

11.3 Calculations

See Appendix C.

11.4 Data Review

11.4.1 Primary Review

Review project documents such as the environmental test request (ETR) analytical worksheets, Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Confirm qualitative and quantitative identification criteria using the criteria provided in Section 11.1 and Section 11.2. If the data system does not properly integrate a peak, perform manual integration in accordance with laboratory SOP BR-QA-006.

Review the instrument QC against the acceptance criteria given in Section 10.0 and summarized in Table 3. If the results do not fall within acceptance criteria, perform the recommended corrective action. If corrective action is not taken or is not successful, document the situation with a nonconformance memo (NCM).

Dilute and reanalyze samples whose results exceed the calibration range. The dilution analysis should result in a determination within the calibration range, preferably in the upper half of the calibration range. A more concentrated analysis is not necessary unless the project requires it. Dilution analyses may also be performed to minimize matrix interference.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carry over. If carry over is suspected, reanalyze the sample.

Upload the data into the LIMS. Enter worksheet information and verify batch information is complete. Set results to primary, secondary, acceptable or rejected as necessary. Verify QC and calibration associations then set the batch to first level review.

11.4.2 Secondary Data Review

Spot-check quantitative and qualitative identifications using the criteria provided in Section 11.1 and Section 11.2.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-005. If a problem is found, immediately consult with the primary analyst or notify the Technical Director or QA Manager.

  Reintegration (by secondary data reviewers) should not be performed except in limited circumstances such as when the primary analyst who performed the initial integration is not available to correct any errors found during secondary review. If reintegration is performed, each integration performed by the secondary reviewer must be reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-005.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

- Verify a "before" and "after" chromatogram for every manual integration performed on an instrument performance check standard (Tune, ICAL, ICV, CCV), QC sample (MB, LCS) and for any manual integration performed on any surrogate or internal standard in any field sample was created.

- Document your review of manual integrations on the manual integration summary report and obtain any review signatures of integrations performed during secondary review as required.

Verify that the performance criteria for the QC items listed in Table 1 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. Verify an NCM was initiated for any QC that does not meet established criteria and verify data is qualified accordingly. Set samples to 2nd level review.

Run the QC Checker, investigate and correct any problems found. Run and review the deliverable. Fix any problems found then set the method chain to lab complete.

11.5 Data Reporting

Data reporting and creation of the data deliverable is performed by the LIMS using the formatters set by the project manager during project initiation.

The following sections describe the default reporting scheme for this method:

Analytical results above the reporting limit (RL) are reported as the value found. Analytical results less than the RL are reported as non-detect to the adjusted RL. The RL is adjusted for sample dilution/concentration. The unadjusted RL for each target analyte is provided in Section 1. If estimated values are requested, results between the LOD and the RL are reported and flagged as estimated.

Further guidance on the application and use of method detection limits (MDLs), reporting limits (RLs) and quantitation limits (QL) for the reporting analytical data is provided in laboratory SOP LP-QA-005.

Electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014 Laboratory Records.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

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

14.0 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 BR-EH-001. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.

- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

15.0 References / Cross-References

- NOAA Status and Trends Program Document: Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Vol. IV, NOAA Technical Memorandum, NOS ORCA 71.
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-QA-011
- Laboratory SOP BR-EH-001
- Laboratory SOP BR-QA-014
- Laboratory SOP BR-QA-006
- Laboratory SOP BR-QA-005

16.0 Method Modifications

Not applicable

17.0 Attachments

- Table 1: Target Compound List and Reporting Limit
- Table 1A: Accuracy and Precision Limits
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

18.0 Revision History

BR-GC-008, Rev. 9

- SOP updated to the new TestAmerica format.
- Standard concentration tables were added to Appendix B.
- Formulas in Appendix C were revised to use Response Factors instead of Calibration Factors.
- Language was added to section 10.2 to allow for updating RT windows on CCVs.
- Language was added to section 11.4.1 to allow for dilution to minimize matrix interference.
- Added statement after table 1A indicating Monobutyltin as a poor performer.

Table 1: Routine Target Analyte List & Reporting Limit (RL)

ANALYTE	Routine Reporting Limit (RL)[1,2]	
	Water (ug/L)	Solid (ug/Kg)
Tetrabutyltin	0.050	1.7
Tributyltin	0.045	1.5
Dibutyltin	0.039	1.3
Monobutyltin[1]	0.50	5.0
Tripentyltin (Surrogate)	N/A	N/A

[1] *The routine RL is the unadjusted value that can be achieved in a blank matrix.*
[2] *The RL for tissue matrix is project defined.*

Table 1A: Routine Accuracy and Precision Limits[1]

Analyte	In-House Limits[2] (%R)		Precision (RPD) (≤)
	Water	Solid	
Tetrabutyltin	30-150	30-160	30
Tributyltin	30-150	30-160	30
Dibutyltin	30-150	30-160	30
Monobutyltin[1]	10-48	10-48	30
Tripentyltin (Surrogate)	15-150	30-120	N/A

[1] *The control limits for Monobutyltin are advisory because this analyte is a poor performer.*

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	Option 1: RSD for each analyte ≤ 20% Option 2: Linear Regression: r ≥ 0.995	Correct problem, reanalyze, and repeat calibration.
ICV	After each initial calibration	(% R) ± 25% from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±25%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate	All field and QC samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Internal Standard: a known amount of standard added to a test portion of a sample as a

reference for evaluating and controlling the precision and bias of the applied analytical method.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to them.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in hexane using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance.

Internal standard (tetra-n-propyltin) is added to each calibration, ICV, CCV, and sample aliquot before analysis. 10 uL of internal standard is added to 100 uL of standard or sample aliquot for analysis.

Internal Standard Solution (5 mg/L)

Parent Standard	Vendor	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Tetra-n-propyltin	Retek #31474	2000	0.250	100	5.0

All working calibration standards, ICVs and CCVs for this method are prepared and derivitized by the organic prep department following the procedures given in the extraction SOP.

Final Concentration of Prepared Calibration Standards (as alkyltin chloride compounds)

Component	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	ICV (ug/L)
Tripentyltin Chloride	50	100	250	500	1000	250
Tetrabutyltin	50	100	250	500	1000	250
Tributyltin chloride	50	100	250	500	1000	250
Dibutyltin dichloride	50	100	250	500	1000	250
Monobutyltin trichloride	50	100	250	500	1000	250

Final Concentration of Prepared Calibration Standards (as un-substituted alkyltin compounds)

Component	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	ICV (ug/L)
Tripentyltin	45.0	90.0	225	450	900	225
Tetrabutyltin	50.0	100	250	500	1000	250
Tributyltin	44.5	89.0	222.5	445	895	222.5
Dibutyltin	38.5	77.0	192.5	385	770	19.25
Monobutyltin	31.0	62.0	155	310	620	155

The alkyltin chloride compounds are reported as un-substituted alkyltin compounds. The factors used to convert from the alkyl tin chloride to the alkyl tin are listed below.

Analyte	Conversion Factor	Report as
Tetrabutyltin	---	Tetrabutyltin

Analyte	Conversion Factor	Report as
Tributyltin chloride	0.89	Tributyltin
Dibutyltin dichloride	0.77	Dibutyltin
Monobutyltin trichloride	0.62	Monobutyltin
Tripentyltin chloride (SS)	0.90	Tripentyltin
Tetrapropyltin (ISTD)	--	Tetrapropyltin

Conversion Factors are determined from the following formula:

$$Conversion\ Factor = \frac{MWT - MWC}{MWT}$$

MWT= Total molecular weight of the analyte

MWC= Number of chlorides * molecular weight of chloride (molecular weight of chloride = 35.5)

Appendix C: Equations

Response Factor (RFx) = $\frac{\text{Peak area or height (x) x Concentration (is)}}{\text{Peak area or height (is) x Concentration (x)}}$

Where: x=compound, is = Internal Standard

Mean Response Factor ($\overline{RF}$) = $\frac{\sum_{i=1}^{n} RF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Response Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Response Factor =

$7\frac{SD}{\overline{RF}} \times 100\%$

Percent Difference (%D) = $\frac{RF_v - \overline{RF}}{\overline{RF}} \times 100\%$

where: RF_v = Response Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ x 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Measured concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Measured concentration of the Spiked Sample

C_u = Measured concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (RPD) = $\dfrac{|C_1 - C_2|}{\left(\dfrac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$$C_{extract}\,(ug/L) = \frac{\text{Peak response (x)}}{\text{Peak response (is)}} \times \frac{\text{Concentration (is)}}{\text{Average RF (x)}}$$

Where: x=compound, is = Internal Standard

Water

$$C_{sample}(ug/L) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solid

$$C_{sample}(ug/Kg) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\,\text{solids}} \times DF$$

Title: Extraction Procedure for Organotins

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Facility Distribution No. Electronic **Distributed To: Facility Intranet**

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1.0 Scope and Application

This SOP describes the extraction procedure for the determination of organotins in water, soil, sediment, waste and tissue samples.

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used for the following matrices: non-potable water, soil/sediment waste and tissue.

The analyte list and reporting limits are provided in laboratory SOPs for instrument analysis.

2.0 Summary of Method

Soil, sediment and waste samples are extracted in hexane/tropolone using ultrasonic or soxhlet extraction. Tissue samples are homogenized and extracted in hexane/tropolone using a Tissuemizer. Water samples are extracted in hexane/tropolone by separatory funnel. The extracts are concentrated, exchanged into hexane and reacted with hexyl magnesium bromide to form the hexyl derivatives. The concentrated extracts are fractionated using Silica Gel/Florisil.

This procedure was developed in-house and is based on procedures described in NOAA Status and Trends Program Document: Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Vol. IV, NOAA Technical Memorandum, NOS ORCA 71.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference and glassware must be cleaned prior to use following laboratory SOP BR-EX-017 Glassware Cleaning Procedure.

Each batch of hexyl-magnesium bromide used for derivitization should be tested for contamination and effectiveness before use on field sample extracts.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Soil Extraction: Sonicators can generate high levels of noise and must be used in an appropriate noise reduction area.

Water Extraction: Hexane may create excessive pressure inside the separatory funnel. Initial venting of separatory funnels must be done immediately after the sample has been sealed and inverted. Vent into a fume hood away from your person or other analysts.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

6.1 Extraction Equipment

- High Intensity Ultrasonic Processor - Tekmar 600-watt Model Dual Output with pulsing capability, 3/4" standard disrupter horn.
- Tissumizer Fisher Scientific, Power-gen 700 with 20mm rotor shaft.
- Separatory Funnel shaker – Glas-Col.
- Soxhlet Water Bath
- Soxhlet Glassware: Top, bottom, and soxhlet thimble.
- Glass Funnel, 100 mm diameter. Fisher Scientific or equivalent
- Fiberglass Wool, 8 um. Fisher Scientific or equivalent
- Filter paper, Whatman No. 541 or equivalent.
- Beakers - 400 mL
- Spatula, Teflon or stainless steel Fisher Scientific or equivalent
- Teflon or Glass Separatory Funnel, 2 L with stopcock and stopper

6.2 Extract Concentration (KD Apparatus)

- Concentrator tube, 10 mL graduated.

- Snyder Column, Three ball macro
- Snyder Column, Two ball micro
- Evaporation Flask, 500 mL attached to concentrator tube with clip.
- Boiling Chips, silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride.
- Teflon Ultra Pure PTFE Boiling Stones. Chemware Catalog Number 0919120
- Heating mantle rheostat controlled for water bath capable of temperature control (± 5°C).
- Water Bath, capable of temperature control to ±5°C.
- Solvent Vapor Recovery System, Kontes K-54000-1006, K-547300-000, Ace Glass 6614-30 or equivalent.

6.3 Miscellaneous

- 75ul, 1mL & 10mL adjustable pipettes, Fisher Scientific or equivalent
- Balance, top loading, capable of accurate weight measurements to the nearest 0.01 g.
- pH Paper/Stripes: Range 1-14.
- pH Meter.
- Pasteur glass pipettes, 1 mL, disposable. Fisher Scientific or equivalent
- 0.5 mL – 2.0 mL Hamilton Gastight® syringes or equivalent.
- Vials and caps: 1.8, 4, 8, 16, and 40 mL with Teflon lined septa and screw caps. Fisher Scientific or equivalent
- Vacuum Box with Teflon inserts.
- Wrist Shaker.

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate, granular, anhydrous, (Na_2SO_4): J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours.
- Methylene Chloride (CH_2C_{12}): Pesticide quality, J.T. Baker or equivalent.
- Hexane, (C_6H_{14}): Pesticide quality, J.T. Baker or equivalent.

- Acetone, ($(CH_3)_2CO$): Pesticide quality, J.T. Baker or equivalent.
- Silica Gel/ Florisil Cartridges: 21 gram, Restek or equivalent.
- Tropolone: 98% neat Material, Aldrich Chemical or equivalent.
- Triethylamine, ($(C_2H_5)_3N$): Pesticide quality, Aldrich Chemical or equivalent.
- Hydrochloric Acid (HCl): J.T. Baker or equivalent.
- Magnesium Metal: Mallinkrodt or equivalent.
- 1-Bromohexane: 98% neat Material, Aldrich Chemical or equivalent.
- Diethyl Ether: Anhydrous, Pesticide quality, J.T. Baker or equivalent.

7.1.1 Prepared Reagents

- HCl Solution (1:1 v/v): Add 500 mL of reagent water to a 1 L volumetric flask. Slowly add 500 mL of HCl to the flask to dilute to volume. Store the solution in reagent bottle at room temperature. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.
- Tropolone/Hexane Solution (0.05%): Add 2.00 g of tropolone to a 4 L bottle that contains 4 L hexane. Tumble for one hour. Prepare fresh each day of use.
- Grignard Reagent (Hexylmagnesium Bromide) Preparation (2M):

  Dry all glassware, stir bars and other equipment used to prepare grignard reagent in an oven maintained at a minimum temperature of 105°C for at least 2 hours.

  While the equipment is still warm, fit a 1.0 L two or three-neck round bottom flask with a Liebig condenser and addition funnel sidearm to the glassware. Do not begin water flow at this time.

  Add gas inlet tubes to the top of the condenser and the addition funnel.

  Connect the gas lines from a regulator attached to the dry nitrogen cylinder to the gas inlet adapter on the sidearm addition funnel and from the gas inlet adapter on the condenser to the inlet side of the nitrogen bubbler.

  Remove the condenser and add 30 g of magnesium turnings to the flask. Add a stir bar and replace the condenser. Begin the stirring motor to agitate the turnings.

  Start the flow of nitrogen through the apparatus so that the flow approximates 20-30 mL/min. The nitrogen should sweep the entire apparatus in order to eliminate all trace of air and water vapor through the system. Allow the flow to continue for at least 30 minutes prior to the addition of any other reagent.

Remove the gas inlet adapter from the top of the addition funnel, open the stopcock on the addition funnel and add 500 mL of anhydrous diethyl ether to the round bottom flask through the addition funnel. Close the stopcock and add 140.37 mL or 165 grams of 1-bromohexane to the addition funnel. Replace the gas inlet adapter onto the addition funnel and continue the nitrogen flow.

Maintain the flow of nitrogen throughout the reaction. Start the water flow through the condenser at this point and continue until the reaction is complete. Reaction is complete when all of the 1-bromohexane is added.

Add ~10 mL of 1-bromohexane to the flask and continue stirring. Formation of bubbles around the magnesium turnings indicates that the reaction has started. After 5-10 minutes of continuous stirring, resume drop wise addition of reagent at a rate that prevents the diethyl ether from refluxing higher than 1/3 of the way up the condenser. These additions should take 30 minutes to 1 hour and the magnesium turnings will be mostly consumed in the reaction.

When the addition of 1-bromohexane is complete, stir the reaction for another 10 minutes.

Stop the stir motor, remove the condenser and the sidearm flask and quickly pour the reagent into dry 40 mL vials. Immediately cap the vials, and then label each vial with the reagent name, molarity (2 M), date made, expiration date and your initials. An expiration date of 1 year may be assigned to the prepared reagent unless the parent components expire sooner, in which case, use the earliest expiration date.

7.2 Standards

Purchase stock standard solutions from commercial vendors. From these, prepare surrogate and spiking solutions by diluting known volumes of the stock standard solutions in an appropriate solvent using the formulations provided in Appendix B. Record the preparation of the standard in the LIMS. Unless otherwise specified in Appendix B, store the prepared solutions in glass containers at a temperature of 4°C (±2) and assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Listed below are the recommended minimum sample size needed for analysis and the requirements for preservation and holding times.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]
Water	Glass	1L	4°C (±2°C)	Extraction: 7 days
Solid	Glass	50g	4°C (±2°C)	Extraction: 14 days
Tissue	Glass	50g	-15°C (±5°C)	Extraction: 14 days

[1]Extraction holding time is determined from sampling date; analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	With every batch if sufficient volume is available	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

10.0 Procedure

10.1 Instrument Calibration

Calibrate the pH meter on each day of use, prior to use using pH 4, 7 and 10 buffer solutions. Check the calibration of the balance each day of use prior to use with at least 3 Class S weights that bracket the range of use. Check the calibration of the pipettes per the frequency specified in laboratory SOP BR-QA-008.

10.2 Calibration Standard Preparation

Prepare calibration standards on request from the GC department.

To prepare each calibration standard and the calibration verification standard (ICV) prepare the parent standards using the formulation(s) given in Appendix B. Add the volume of parent standard(s) as specified in the following table to a 16 mL clear labeled vial that contains 5 mL of hexane.

Formulations for the Preparation of the Calibration Standards

Parent Standard	Level 1 (uL)	Level 2 (uL)	Level 3 (uL)	Level 4 (uL)	Level 5 (uL)	ICV (uL)
Tin Surrogate	50	100	1250	500	1000	1250
Tin Spike	50	100	1250	500	1000	1250

Derivatize each standard using the procedure given in Section 10.5. Concentrate calibration levels 1, 2, 4, and 5 to 1.0 mL in hexane. Concentrate level 3 and the ICV to 5.0 mL in hexane. Give he standards to the GC department.

The final concentration of each calibration level is provided in the following table.

Final Concentration of Prepared Calibration Standards

Component	Level 1	Level 2	Level 3	Level 4	Level 5	ICV

	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)
Tripentyltin Chloride	50	100	250	500	1000	250
Tetra-n-butyltin	50	100	250	500	1000	250
Tri-n-butyltin chloride	50	100	250	500	1000	250
Di-n-butyltin dichloride	50	100	250	500	1000	250
n-Butyltin trichloride	50	100	250	500	1000	250

10.3 Sample Preparation

10.3.1 Solid Extraction

Mix sediment samples thoroughly and discard any foreign objects such as sticks, leaves and rocks. Homogenize the sample following the procedures given in laboratory SOP BR-QA-020.

Label and place a 400 mL beaker on the balance and tare the balance. Weigh approximately 30 g ± 1 gram of sample into the beaker and upload the weight measurement into the LIMS worksheet. Use 30 g of purified sodium sulfate for the MB and LCS

Add 10 mL of 1:1 HCl solution to each beaker then add a sufficient amount of anhydrous granular sodium sulfate to each sample beaker and stir to create a free-flowing sample.

Add the 0.5 mL of surrogate solution to each field and QC sample. Add 0.5 mL of spike solution to the LCS and each MS/MSD.

Add 100 mL of 0.05% tropolone/hexane solution to each beaker. Extract the samples by ultrasonic extraction using the procedure specified in laboratory SOP BR-EX-008.

NOTE: For USACE work, extract the samples following the procedure given in laboratory SOP BR-EX-007 *Soxhlet Extraction, SW-846 3540C* using 0.05% tropolone/hexane as the extraction solvent.

Concentrate the sample using the techniques described in Section 10.4 to a volume of 5 mL in hexane.

Derivatize the sample following the steps in Section 10.5.

10.3.2 Tissue Extraction

Homogenize the tissue following the procedure given in laboratory SOP BR-EX-009.

Label and place a 400 mL beaker on the balance and tare the balance. Weigh approximately 30 g ± 1 gram of sample into the beaker and upload the weight measurement into the LIMS worksheet. Use 30 g of purified sodium sulfate for the MB and LCS

Add 10 mL of 1:1 HCl solution to each beaker then add a sufficient amount of anhydrous granular sodium sulfate to each sample beaker and stir to create a free-flowing sample.

Add the 0.5 mL of surrogate solution to each field and QC sample. Add 0.5 mL of spike solution to the LCS and each MS/MSD.

Add 100 mL of 0.05% tropolone/hexane solution to each beaker. Extract the samples by ultrasonic extraction using the procedure specified in laboratory SOP BR-EX-008.

Concentrate the sample using the techniques described in Section 10.4 to a volume of 5 mL in hexane.

Derivatize the sample following the steps in Section 10.5.

10.3.3 Water Extraction

Measure 1 L of sample into a graduated cylinder then quantitatively transfer the sample to the separatory funnel. Alternatively, if samples were received in 1 L containers, mark the meniscus of the aqueous volume on the sample container with a permanent marker. Pour the entire sample into the 2 L separatory funnel. Rinse the sample container with ~60 mL of troplone/hexane mixture and pour the rinsate into the separatory funnel extractor. To measure the actual sample volume, fill the sample container with tap water to the mark of the meniscus and pour the water into a graduated cylinder for volume measurement. Use 1000 mL of reagent water for the method blank and LCS.

Acidify each sample to pH < 2 with 10 mL of 1:1 HCl solution.

Add the 0.5 mL of surrogate solution to each field and QC sample. Add 0.5 mL of spike solution to the LCS and each MS/MSD.

Extract the samples using the procedure given in laboratory SOP BR-EX-005.

Concentrate the sample using the techniques described in Section 10.4 to a volume of 5 mL in hexane.

Derivatize the sample following the steps in Section 10.5.

1.2 Extract Concentration Techniques

Macro Snyder Column (K-D)

Add one or two clean boiling chips to the K-D evaporation flask and attach a three-ball Snyder column to the flask. Add ~1 mL of methylene chloride to the top of the column then place the K-D apparatus in a hot water bath (60-70°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 water vapor.

Attach the solvent vapor recovery glassware to the Snyder column. Adjust the vertical position of the apparatus and check the water bath temperature. The water bath temperature should be between 54.8 – 74.8°C when methylene chloride is the extraction solvent and 84-89°C when hexane is the extraction solvent. Higher water bath temperatures may be used so long as the recovery of target analytes is not impacted. The boiling point of each solvent is provided in the following table:

Solvent	Boiling Point	Water Bath Temperature
Hexane	69°C	84 – 89°C
Methylene Chloride	39.8°C	54.8 – 74.8°C

Monitor the concentration and do not let the extract evaporate to dryness. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with solvent.

When the apparent volume of the extract reaches desired amount remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

Micro Synder Column (K-D)

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column to the tube. Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and check the temperature of the water bath to ensure the proper temperature for the extract solvent.

Continuously monitor the distillation process to ensure sample extracts do not evaporate to dryness. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. Remove setup when desired sample volume is reached.

Nitrogen Blowdown

Nitrogen blow down may be used to concentrate extracts as needed.

Place the concentrator tube in a warm water bath maintained at a temperature of 35°C. Apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

10.4 Derivatization and Extract Cleanup

Perform sulfur cleanup on all soil and sediment extracts prior to derivitization. Refer to laboratory SOP BR-EX-002 for the sulfur cleanup procedure

Add 150 uL of triethylamine and 0.80 mL hexyl magnesium bromide to each extract then shake the extracts for 60 minutes using the wrist shaker.

Working in a fume hood, slowly add 1:1 HCl to each extract to dissolve the precipitate, and vortex the extract. Centrifuge the acid/extract mixture to separate the extract from the acid. Transfer the solvent phase to a concentrator tube. Rinse the acid layer with 1-2 mLs of hexane and transfer the acid rinse to the same concentrator tube.

Attach a 21g silica/florisil cartridge to the vacuum box using a Teflon insert. Elute 100 mL of methylene chloride through the cartridge followed by 100 mL of hexane at gravity flow rate. Remove the cartridge and Teflon insert and place it on top of a K-D flask.

Elute the extract through the cartridge using 100 mL of hexane. Collect extract in a K-D flask. Concentrate the extract to a final volume of 1.0 mL.

Transfer the extract to a labeled Teflon lined screw cap vial. Complete the extraction batch information in the LIMS batch.

11.0 Calculations / Data Reduction

11.1 Calculations

Not applicable.

11.2 Data Review

1.3 Data Review

Primary Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Enter the batch information into LIMS and complete the batch editor and worksheet for each extraction and cleanup performed. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

12.0 Method Performance

12.1 Limit of Detection (LOD) & Limit of Quantitation (LOQ)

A limit of detection (LOD) must be determined for the method if the laboratory reports results below the limit of quantitation (LOQ). If results are not reported below the LOQ, a LOD is not required but may be performed at the laboratory's discretion. The laboratory's procedures for LOD and LOQ are further described in laboratory SOP BR-QA-005.

12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

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

14.0 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 BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 gallon covered and vented drum.
- Extracted water samples - Satellite container: 55 gallon covered and vented drum.
- Vials containing extracts - Satellite container: 5 gallon covered bucket in fume hood.
- Hydrochloric Acid Waste-Satellite Container: 2.5L Waste Bottle Labeled with appropriate acid type (Hydrochloric).
- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)

15.0 References / Cross-References

- *Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992,* Vol. IV, NOAA Technical Memorandum, NOS ORCA 71.

16.0 Method Modifications

Not applicable.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Formulas

18.0 Revision History

Revision 7:
- Title Page: Updated approval signatures.
- All sections: Updated practice for LIMS implementation.
- Section 6.0: Added equipment

- Section 10.0: Added spike information
- Section 10.5: Added sulfur cleanup for soils and sediments

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methylene chloride	Cancer causing	25 ppm (TWA)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of with six months.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Magnesium Metal	Flammable Reactive w/ water Irritant Eye/vision damage		Flammable solid. Dangerous when wet. Highly reactive. May ignite spontaneously on contact with water or damp materials. May cause irritation to skin, eyes, and respiratory tract. Keep away from heat, sparks, and flame. Avoid breathing dust. Keep container closed. Use with adequate ventilation. Wash thoroughly after handling.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above-mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect, or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

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.

Appendix B: Standard Preparation Formulations

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock or parent standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in methylene chloride using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance.

Appendix B: Standard Preparation Formulas

Primary Source Tin Surrogate Solution

Stock Standard	Vendor	Component	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Tripentyltin Chloride Mixture	Restek #31477	Tripentyltin Chloride	2000	250	500	1.0

Solvent: Methylene Chloride

Primary Source Tin Spike Solution

Stock Standard	Vendor	Component	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Butyltin Chlorides Calibration Mixture	Restek #31472	Tetra-n-butyltin	2000	250	500	1.0
		Tri-n-butyltin chloride				
		Di-n-butyltin dichloride				
		n-Butyltin trichloride				

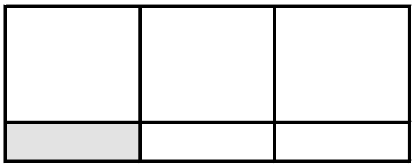
Solvent: Methylene Chloride

Using a different lot of the Butyltin Chlorides Calibration Mixture (Restek #31472) prepare a Second Source Tin Spike Solution following instruction for the Primary Source Tin Spike Solution.

Method	Prep Method	Matrix	Analyte	Units	Method Reporting Limit	Method Detection Limit[a]	
9060M/ASTM D4129-82M	NA	Soil	Organic C	%	0.05	0.02	

a Method Detection Limits are subject to change as new MDL studies are completed.
a MDL is the smallest analyte concentration that can be demonstrated to be different from zero with 99% con

Method	Prep Method	Matrix	Analyte	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
ASTM D4129-82M/PSEP	NA	Soil	Total Organic Carbon	82-119	77-155	20



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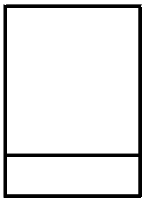

STANDARD OPERATING PROCEDURE

CARBON, TOTAL ORGANIC IN SOIL

GEN-ASTM
Revision 6
October 18, 2007

Approved By:

Supervisor	10/18/07 Date
QA Manager	10-18-07 Date
Laboratory Manager	10/18/07 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: ___ Date: 10/11/08
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

CARBON, TOTAL ORGANIC IN SOIL

1. SCOPE AND APPLICATION

1.1. This procedure is applicable to the determination of Total Organic Carbon (TOC) using ASTM method D4129-82, modified for soil and sediment matrices (Puget Sound Estuary Program and Lloyd Kahn). Total organic carbon is a measure of the total amount nonvolatile, partially volatile and particulate organic compounds in a sample. Sample should be treated to remove inorganic carbon (carbonates, bicarbonates, free CO_2 etc.), prior to analysis, as these compounds will interfere with true readings.

1.2. This method is applicable to all soils and sediments and most matrices that can be dried and shatter-boxed to a fine powder.

1.3. Results are reported as percent (%) carbon, and the applicable range is the MDL - 100%. The Method Reporting Limit (MRL) for TOC on soils is 0.05%, dry weight basis. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The Method Detection Limit (MDL) has been determined at 0.02%.

2. METHOD SUMMARY

2.1. Samples are combusted in an oxygen atmosphere to convert organic and inorganic forms of carbon to CO_2. The combustion temperature is selected to completely oxidize all carbon forms. The combustion product gases are swept through a barium chromate catalyst/scrubber to ensure that all of the carbon is oxidized to CO_2. Other potentially interfering product gases such as SO_2, SO_3, HX, and NO_x are removed from the gas stream in a series of chemical scrubbers. The CO_2 is then swept to the coulometer where it is detected by automatic, coulometric titration, with coulometric end point indication.

2.2. The coulometer cell is filled with a partially aqueous medium containing ethanolamine and a colorimetric indicator. When a gas stream passes through the solution, CO_2 is quantitatively absorbed. CO_2 reacts with the ethanolamine to form a strong titratable acid which caused the indicator to fade. The titration current automatically turns on and electrically generates base to return the solution to its original color.

3. DEFINITIONS

3.1. Analysis Batch - Samples are analyzed in a set referred to as an analysis batch. The batch begins with calibration/standardization followed by QC analyses and samples. The batch ends when the QC analyses and set of samples has been completed.

3.2. Method Blank - The method blank is an artificial sample (empty boat) designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.3. Laboratory Control Sample (LCS) - A standard of known TOC concentration which is used to ensure that the analysis produces an accurate measurement of TOC in samples analyzed in the batch.

4. INTERFERENCES

4.1. Acidic and other gases, including SO_2, SO_3, H_2S, HCl, HBr, HI, Cl_2, and NO_x can be effectively removed using scrubbers such as KI, Ag_2SO_4, $AgNo_3$, and MnO_2.

4.2. Volatile organics may be lost in the decarbonization process.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4. Disconnect teflon tubing from furnace at check valve whenever system is not in use or when O_2 flow is turned off or furnace temperature is reduced. If the carbon cathode solution should be siphoned through a failed check valve into the magnesium perchlorate scrubber potentially explosive DMSO-perchlorate could be formed.

5.5. Do not attempt to combust large samples of organic or other materials that will react with pure oxygen. Such samples can cause the pyrolysis tube to explode.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

Samples can be collected in glass or plastic containers. Samples are preserved by storage at 4±2°C. Samples are analyzed within 28 days of collection.

7. APPARATUS AND EQUIPMENT

7.1. Induction furnace, Coulometrics Incorporated.

7.2. Analytical balance, 0.1mg accuracy.

7.3. Desiccator.

7.4. Quartz combustion boats.

7.5. Sample scoop.

7.6. Porcelain dishes.

7.7. Glass ladles and miscellaneous laboratory glassware,

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Standards

8.1.1. Urea - 20% carbon. Use 10 μg.

8.1.2. Nutrients in Soil, purchased standard with a known TOC value (typically ERA #542). Use 50 mg for LCS.

8.2. Reagents

8.2.1. Hydrochloric acid, 50% and 10%.

8.2.2. Carbon Cathode Solution. Dimethyl Sulfoxide; DMSO. Purchased from Coulometrics Inc. as a prepared solution. Used for coulometer solution.

8.2.3. Anode Solution. Dimethyl Sulfoxide and potassium iodide. Purchased from Coulometrics Inc. as prepared solution.

8.2.4. Manganese dioxide. Gas scrubber solution.

8.2.5. Potassium Hydroxide. Gas scrubber solution.

8.2.6. Potassium Iodide. Anode chemical.

8.2.7. Magnesium Perchlorate desiccant

9. RESPONSIBILITIES

9.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

9.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the *SOP for Documentation of Training*, is also the responsibility of the department supervisor/manager.

10. PREVENTIVE MAINTENANCE

Maintenance is performed as follows:

Maintenance Item	Frequency
Cell	Clean daily with methanol and water to clean frit
Mg Perchlorate Scrubber	change daily
KOH Scrubber	change monthly
NOX scrubber	change as needed
Repack Precombustion Column	as needed
Repack Combustion Column	as needed

11. PROCEDURE

11.1. Sample Preparation.

11.1.1. Turn furnace on to #5 (≈1000°C). Allow furnace to warm-up for about 1/2 hours. Turn on oxygen to ≈ 5 psi and 75 to 125 ml/min at flowmeter.

11.1.2. Clean quartz boats. Scrape out old sample and rinse boats with DI water. Place boats in crucible and muffle for at least 10-15 minutes. Remove boats and place in desiccator until ready for use.

11.1.3. Samples should be dried at 70°C and homogenized prior to analysis.

11.1.4. As a rule, the darker (or closer to black) a sample is, the more carbon it contains. Place a small portion of sample on a watch glass. Add 1 drop of 10% HCl. Watch for effervescence or bubbling. If bubbles are present, the sample contains inorganic carbon (CO_3). If sample bubbles, reduce sample size to prevent sample from bubbling out of boat. If sample is dark, wood product or sludge reduce sample volume to 5 → 10mg. Normal sample volume = 50mg. After boats are loaded with sample add 1 to 2 drops 10% HCl. Place boats in 70°C oven to dry. If samples bubbled when acid was added, add 1 to 2 drops more acid and dry at 70°C. Continue acidifying and drying until samples no longer bubble. Place samples in desiccator until ready for analysis.

11.2. Apparatus Preparation.

11.2.1. Fill cell with carbon cathode solution to 100 → 125 ml, drop in stir bar. Place cell top on snug.

11.2.2. Cover bottom of anode cell with KI. About 2 small scoops.

11.2.3. Add carbon anode solution to cell such that when anode is inserted in the anode cell, the anode solution level is the same as the cathode solution level.

11.2.4. Place cell in coulometer cell holder.

11.2.5. Turn on detector lamp and stir plate. (Power on)

11.2.6. Turn adjust knob to 122 (all the way to the right) then turn back down to 100. Rotate cell until maximum transmittance is obtained.

11.2.7. With oxygen bubbling to cell and maximum transmittance obtained, turn on the current to the anode and cathode. The carbon cathode solution will begin to titrate to a blue color.

11.2.8. Change Magnesium Perchlorate desiccant daily.

11.2.9. The instrument is now ready to run.

11.3. Calibration and Standardization.

11.3.1. Burn both ladles for five minutes each to remove any residual TOC.

11.3.2. Establish baseline.

11.3.2.1. After placing ladles in sample inlet, allow system to purge for 1 minute.

11.3.2.2. Burn three boats empty five minutes each. The average of the three runs is the baseline.

11.4. Analysis.

11.4.1. Place one platinum or quartz boat in a ladle. Place the ladle in the sample inlet and purge for 1 minute. Simultaneously insert the sample into the furnace, press the reset button on the coulometer and start the timer for five minutes.

11.4.2. After five minutes, obtain a reading from the instrument. Remove the ladle from the furnace. (Occasionally, a high sample may require longer than 5 minutes to complete the titration).

11.4.3. Load the other ladle with the next platinum (or quartz) boat. Remove the ladle in use from the inlet port and insert the next ladle.

11.4.4. Repeat steps 10.4.1 through 10.4.3 until all samples are analyzed.

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

The precision and accuracy of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS's are prepared and analyzed. The RSD should be $<20\%$ and average recovery must be 85-115%.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Analyze a minimum of seven spiked blank replicates at a level near the MRL. Follow the procedures starting in Section 11 to analyze the samples. Refer to the CAS *SOP for The Determination of Method Detection Limits and Limits of Detection.*

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described below and in the *SOP for Sample Batches*. Project-specific batching protocols may also be required.

12.3.1. LCS - An LCS must be analyzed with each batch of 20 or fewer samples. Analyze 50mg of the purchased standard (see 8.1.2) is used. The acceptance criteria for this LCS is ± 15% of the true value.

12.3.2. Method Blank - Burn one empty boat per batch of 20 or fewer samples. Method Blank must be <0.05% carbon.

12.3.3. CCV (Continuing Calibration Verification) - A CCV must be analyzed every tenth analysis. Analyze ~10mg urea. The CCV must be 18.0% - 22.0% carbon.

12.3.4. CCB (Continuing Calibration Blank) - A CCB must be analyzed following every CCV.

12.3.5. Sample duplicate - One sample per batch of 20 or fewer samples must be analyzed in duplicate. Duplicates should be 20% RPD, if > five times the MRL.

12.3.6. Matrix Spike - One spike must be analyzed with each batch of 20 or fewer samples. The acidified sample will be spiked with a known amount of urea.

12.3.7. See Table 1 for a summary of acceptance criteria and corrective actions.

13. DATA REDUCTION AND REPORTING

13.1. Calculate % carbon as follows:

$$\%Carbon = \frac{(Gross\ reading\ -\ baseline\ \mu g)(0.1)}{mg\ sample\ analyzed}$$

13.2. For duplicate analyses, calculate relative percent difference as follows:

$$RPD = \frac{S_1 - S_2}{Avg} * 100$$

where S1 = Sample with higher value
S2 = Sample with lower value
Avg = Average of the two sample values

13.3. Calculate percent recovery as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

13.4. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified above. Average, RPD, spike level and spike recovery are entered on spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.5. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

13.6. Refer to the *SOP for Laboratory Data Review Process* for general guidelines for data review.

13.7. Reporting

13.7.1. Total organic carbon is reported as % carbon, normally on a dry weight basis. Results may be reported on an as received basis.

13.7.2. The Method Reporting Limit is 0.05% carbon, on a dry weight basis.

13.7.3. Report all results to three significant figures.

13.7.4. Bench sheets are labeled "Total Organic Carbon, TOC". These benchsheets, located in Appendix I, should be in use at all times during TOC analysis.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for Nonconformity and Corrective Action for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method is validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

17.3. This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. REFERENCES

18.1. Coulometrics Inc. Instruction Manual, Model 5020.

18.2. EPA Method Modified 415.1.

18.3. Total Organic Carbon, Method 9060, EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, September 1986, Revision 0.

18.4. Total Organic Carbon(TOC), Conventional Sediment Variables, Puget Sound Estuary Program, March 1986.

18.5. Determination of Total Organic Carbon in Sediment, Lloyd and Kahn, U.S.E.P.A Region II, July 1998.

18.6. ASTM Method D4129-88

TABLE 1

Summary of Corrective Actions

Method Reference	Analysis	Control Item	Acceptance Criteria	Corrective Action
ASTM Method D4129-82	TOC (Soil)	Urea	±10%	Re-analyze all samples affected.
		Benzoic Acid	± 15%	Re-Analyze.
		Method Blank	<0.05%	Re-analyze. If still high, clean boats and start over.
		Sample Duplicate	20% RPD	Analyze a triplicate. Homogenize again and reanalyze.
		Sample Spike	75-125%	Re-analyze.

APPENDIX I

BENCHSHEETS

Columbia Analytical Services, Inc.

Service Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

Acid Purge Time: 1 minute **Reading Time:** 5 minutes **TOC % =** $\frac{\text{(Net Reading)(µg 0.1)}}{\text{mg Sample Injected}}$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

CCV1 =97 CCV2 =

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C REC%=

Comments :

X= RSD= SPK%REC=

Analyzed By:	Date:
Reveiwed By:	Date:

Columbia Analytical Services, Inc.

Service Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

Acid Purge Time: 1 minute **Reading Time:** 5 minutes TOC % = $\frac{\text{(Net Reading)(µg 0.1)}}{\text{mg Sample Injected}}$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C % Rec =

Comments : _____

Analyzed By:	Date:
Reveiwed By:	Date:

TOC.XLT

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked

Baseline
AVG.

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
CCV-1				
CCB-1				
LCS				Avg
MB				
CCV-2				
CCB-2				
CCV-3				
CCB-3				
CCV-4				
CCB-4				
CCV-5				
CCB-5				

Columbia Analytical Services, Inc.

Service Request #: K7112 / K7040 / K7669
Method: 9060s / PSEP (Combustion/Coulometric)
Analysis For: Total Organic Carbon (TOC)
Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon
CCV-1	6.8	1328.3	7.3	1321.0	19.4
CCB-1	50.0	6.8	7.3	-0.5	< 0.05
LCS-1	47.4	198.4	7.3	191.1	0.40
MB-1	50.0	7.2	7.3	-0.1	< 0.05
LCS-2	47.8	201.1	7.3	193.8	0.41
LCS-3	56.1	247.8	7.3	240.5	0.43
LCS-4	46.1	238.5	7.3	231.2	0.50
LCS-5	47.5	196.5	7.3	189.2	0.40
LCS-6	46.2	194.2	7.3	186.9	0.40
LCS-7	47.0	200.7	7.3	193.4	0.41
LCS-8	46.0	214.9	7.3	207.6	0.45
LCS-9	47.1	194.8	7.3	187.5	0.40
LCS-10	45.6	203.5	7.3	196.2	0.43
CCV2	10.7	2074.1	7.3	2066.8	19.3
CCB-2	50.0	9.3	7.3	2.0	< 0.05

Acid Purge Time: 1 minute **Reading Time:** 5 minutes

TOC % = $\frac{\text{(Net Reading)(µg 0.1)}}{\text{mg Sample Injected}}$

CCV : Urea Baker (lot #A17584) ID#: TOCS/1-10-J TV = 20.0%C

CCV1 = 97 CCV2 = 97

LCS: ERA Cat#: 542 Lot#: D060-542 ID#:PO3/2-26E TV = 0.34%C

Comments :

LCS 1 to 10 $\bar{x}$ = 0.42 RSD = 5%

ERA QC Performance Acceptance Limits for TOC are 436 to 6370 mg/kg

MATERIAL STANDARDIZED TO 0.42%. USE AS TV FOR THIS LOT CWF 8/29/08

Analyzed By:		Date: 8/26/08	Time: 0900
Reveiwed By:		Date: 8/27/08	

TOC.XLT

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

1317 South 13th Avenue
Kelso, Washington 98626
(360) 577-7222

Effective Date: October 30, 2009

Laboratory Director/Technical Director: _____
Jeff Christian

Quality Assurance Manager: _____
Julie Gish

Technical Director – Metals: _____
Jeff Coronado

Technical Director – Metals R & D: _____
Nicholas Bloom

Technical Director – Organics: _____
Jeff Grindstaff

Technical Director – Organic Extractions: _____
Dr. Gregory Salata

Technical Director – Inorganics/Microbiology: _____
Harvey Jacky

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

2.0 TABLE OF CONTENTS

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3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is an employee-owned professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, and other material.

Quality Management Systems are established, implemented and maintained by management. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory in involved.

This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities. Policies and procedures are established in order to meet the quality objectives of clients, accrediting authorities, and certifying organizations. Columbia Analytical Services, Inc. is committed to operate in accordance to: ISO/IEC 17025:2005 International Standards, The NELAC Institute (TNI) National Environmental Laboratory Accreditation Program (NELAP), and DoD Environmental Laboratory Accreditation Program. Quality Systems are established to meet the requirements of these standards.

Laboratory management is committed to continually improve the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set fourth in the *Columbia Analytical Services, Inc. Quality and Ethics Policy Statement March* 2009 and in this *Kelso Quality Assurance Manual* (QAM). We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

Columbia Analytical maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

Columbia Analytical is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, Columbia Analytical also operates laboratories in California, Florida, New York, Arizona, and Texas.

The information in this document has been organized according to the format described in *EPA Requirements for Quality Management Plans, EPA QA/R-2,* USEPA, 2001; *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* USEPA, 2001, and *ISO17025 International Standard.*

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at Columbia Analytical is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of Columbia Analytical:

> "The mission of Columbia Analytical Services, Inc. is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

4.1 Quality Management Systems

In support of this mission, the Kelso laboratory has developed Quality Management Systems to ensure all products and services meet our client's needs. These systems incorporate the requirements of ISO17025 standards. Quality Management Systems Include:

- Standard Operating Procedures
- Sample Management
- Chain of Custody Procedures
- Statistical Control Charting
- Standards Traceability
- Core Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the Quality Management System is assessed in several ways:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Complaints
- Internal and External Proficiency Testing

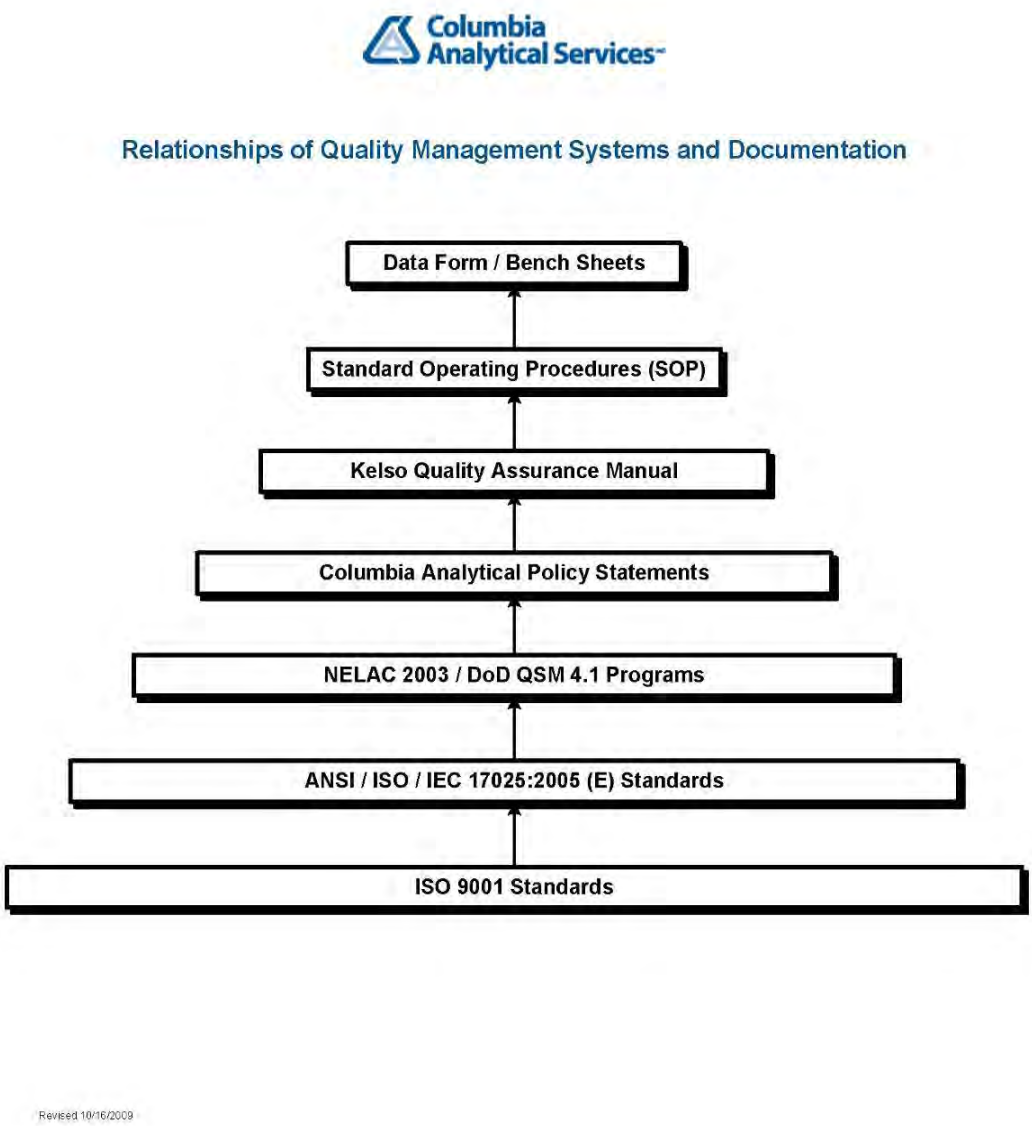


Figure 4-1

Kelso Quality Management Systems are based upon ISO 17025:2005 standards. Fundamental programs (NELAC 2003 and DoD QSM) are based upon these standards. Implementation and documentation against these standards are communicated in corporate policy statements, and Kelso's Quality Assurance Manual. Actual procedures, actions and documentation are defined in both administrative and technical SOP's.

4.2 Facilities and Equipment

Columbia Analytical features over 45,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, Columbia Analytical minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals "clean room" sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Metals R&D Laboratory
- Water Chemistry & General Chemistry Laboratories (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography/High Performance Liquid Chromatography Laboratories
- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Semi-volatile Organics Drinking Water Laboratories (2)
- Volatile Organics Laboratory
    - Separate sample preparation laboratory
    - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water Systems (2)
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.3 Technical Elements of the Quality Assurance Program

The laboratory's technical procedures are based upon procedures published by various agencies or organizations (See Section 18). The Quality Assurance Program provides to the laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained by the QA department. This includes method approvals and audit administration. In addition,

internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventative maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.4 Operational Assessments

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients including; On-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

Columbia Analytical utilizes a number of different methods to ensure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results. All Requests for Proposal (RFP) documents are reviewed by the Project Chemist and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Plans (QAPPs) if available. A weekly status meeting is also conducted with the laboratory staff by the Client Services Manager to inform the staff of the status of incoming work, future projects, or project requirements.

4.5 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, certificates of analysis, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled Columbia Analytical documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

Columbia Analytical maintains a records system that ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH).*

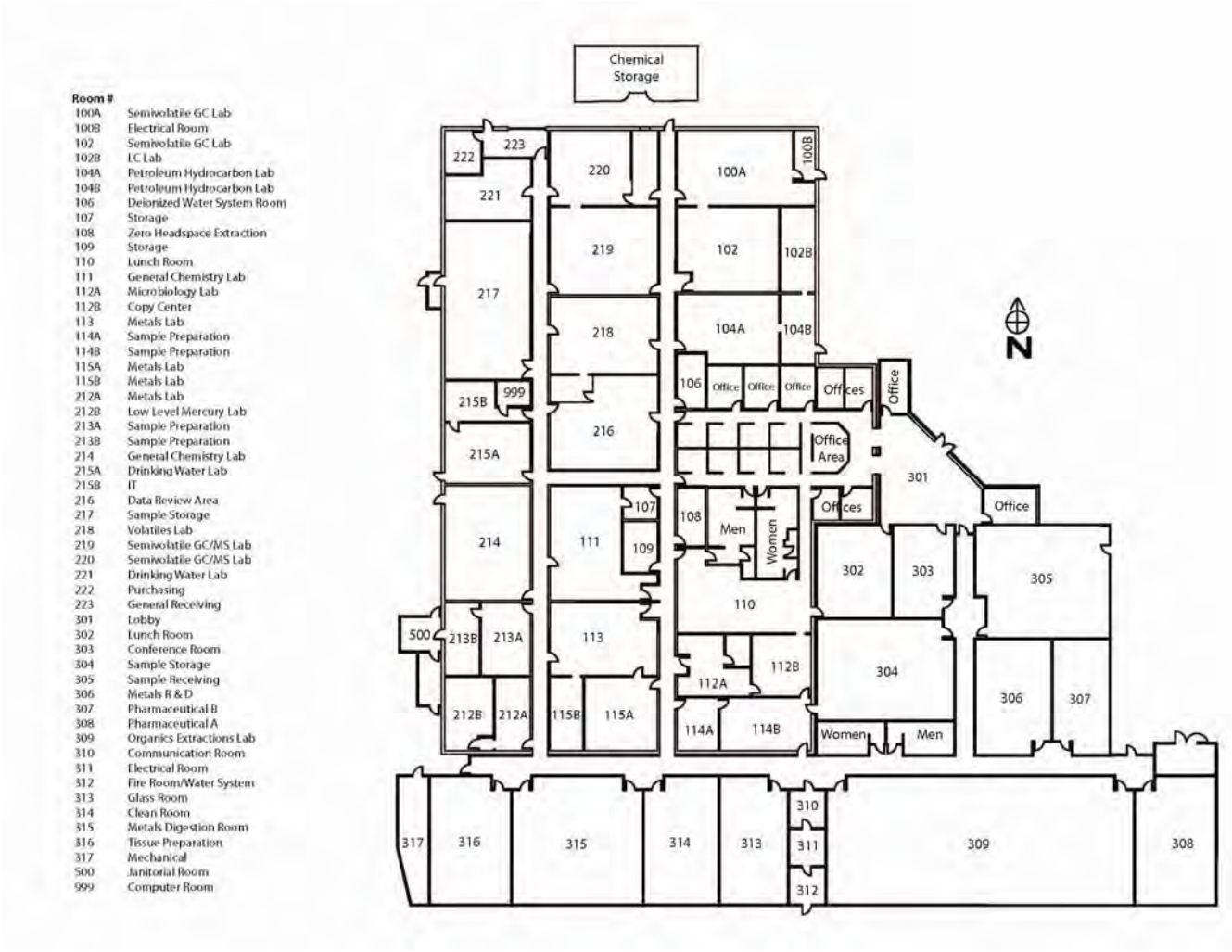
4.6 Subcontracting

Analytical services are subcontracted when Columbia Analytical/Kelso needs to balance workload or when the requested analyses are not performed by Columbia Analytical/Kelso. Subcontracting is only done with the knowledge and approval of the client and to qualified laboratories. Subcontracting to another Columbia Analytical laboratory is preferred over external-laboratory subcontracting. Further, sub-contracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories (ADM-SUBLAB).* The Corporate Quality Assurance staff is responsible for qualifying and oversight of subcontract laboratories.

4.7 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned (by the laboratory user) as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Purchasing through CAS Purchasing Department in Kelso (SOP ADM-PUR).* Also, refer to section 10.4 for a discussion of reference materials.

Figure 4-2
Columbia Analytical/Kelso Laboratory Floor Plan



5.0 PROFESSIONAL CONDUCT AND ETHICAL PRACTICES

One of the most important aspects of the success of Columbia Analytical is the emphasis placed on the integrity of the data provided and services performed. To promote product quality, employees are required to comply with certain standards of conduct and ethical practices. The following examples of Columbia Analytical policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action. Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible. Employee discipline is progressive in its severity and each situation is handled individually in that the discipline is designed to fit the circumstances. Potential disciplinary actions may include a verbal warning, written warning, a second written notice (more severe and more strongly worded than a warning), suspension without pay, demotion, or termination.
- It is the responsibility of all Columbia Analytical employees to safeguard sensitive company and client information. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action.

All employees are required to sign and adhere to the requirements set forth in the Columbia Analytical *Confidentiality and Conflicts of Interest Employee Agreement* and the Columbia Analytical *Commitment to Excellence in Data Quality Policy.* All employees receive in-house ethics training and are periodically reminded of their data quality and ethical conduct responsibilities.

Columbia Analytical makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the Columbia Analytical Employee Handbook. This includes the Columbia Analytical Ombudsman Program, the Columbia Analytical Open Door Policy, and the use of flexible work hours. Operational assessments are regularly made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads (Section 4.3). Procedures for subcontracting work are established, and within the Columbia Analytical laboratory network additional capacity is typically available for subcontracting, if necessary.

6.0 ORGANIZATION AND RESPONSIBILITIES

The Columbia Analytical/Kelso staff, consisting of approximately 130 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

Columbia Analytical is committed to providing an environment that encourages excellence. Everyone within Columbia Analytical shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 6-1 lists the Columbia Analytical/Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The responsibility of the **Quality Assurance Manager** (QAM) is to oversee implementation of the quality program and to coordinate QA activities within the laboratory. The QAM works with laboratory production units to establish effective quality control and assessment plans. The QAM has the authority to stop work in response to quality problems. The QAM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and coordinating the annual review of each SOP; maintaining QA records such as metrological records, archived logbooks, PT sample results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; performing internal QA audits; preparing QA activity reports; etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the Columbia Analytical Quality Assurance Director. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

  The Chief Quality Officer (CQO) is responsible for the overall QA program at all the Columbia Analytical laboratories. The CQO is responsible for ensuring that annual internal audits are performed at each Columbia Analytical laboratory; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of Columbia Analytical-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Organics Department Manager (for the Laboratory Director) and the CQO or Laboratory Director (for the QA Manager).

- The **Environmental Health and Safety Officer** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to Columbia Analytical's EH&S Director.

- The **Client Services and Sample Management Office Manager** is responsible for the Client Services Department (customer services/project chemists, and Electronic Data Deliverables group) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.

- The **Project Chemist** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the Columbia Analytical laboratory and administrative staff to ensure that client-specific needs are understood, and that the services Columbia Analytical provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within the department. Each **Department Manager and Supervisor** has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.

- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.

- **Information Technology** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 6-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	30	Laboratory Director
Julie Gish, M.S.	18	Quality Assurance Manager
Lynda Huckestein, B.S.	20	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	19	Metals Department Manager
Nicolas Bloom, M. S.	29	Metals R & D Manager
Harvey Jacky, B.S.	20	General Chemistry Department Manager
Gregory Salata, Ph.D.	9	Extractions Department Manager
Jeff Grindstaff, B.S.	20	Organics Chromatography & Mass Spectrometry Department Manager
Loren Portwood, B.S.	18	Organics Drinking Water Department Manager
Eileen Arnold, B.A.	27	Environmental Health and Safety Officer
Mike Sullivan, B.S.	8	Information Technology Director
Lee Wolf, B.S.	23	Chief Quality Officer
Steve Vincent, B.S.	33	President

7.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. Columbia Analytical management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major automated data processing equipment.

7.1 Software Quality Assurance Plan

Columbia Analytical has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the *CAS Software Quality Assurance Plan (SQAP)*. The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of this plan are policies for software validation and control.

7.2 IT Support

The local Columbia Analytical Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, Columbia Analytical corporate IT provides support for network-wide systems. Columbia Analytical also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

7.3 Information Management Systems

Columbia Analytical has various systems in place to address specific data management needs. The Columbia Analytical Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request. Included on the service request is a summary of client information, sample identification, required analyses, work instructions, deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats, including Staged Electronic Data Deliverable (SEDD) format.

7.4 Backup and Security

Columbia Analytical laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non- Columbia Analytical access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. Columbia Analytical uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. Email attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems.

8.0 SAMPLE MANAGEMENT

8.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. Columbia Analytical recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

Columbia Analytical uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 18 for complete citations). The container, preservation and holding time information for these references is summarized in Table 8-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

Columbia Analytical routinely provides sample containers with appropriate preservatives for our clients. Containers are purchased as precleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container.

Figure 8-1 shows the chain-of-custody form routinely used at Columbia Analytical and included with sample kits. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to Columbia Analytical. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. Columbia Analytical keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. Columbia Analytical also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When Columbia Analytical ships environmental samples to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures (SMO-GEN) for maintaining the samples' chain of custody, packaging and shipment. Dry ice gel ice is the only temperature preservative used by Columbia Analytical, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures (SMO-GEN) are established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received.

Once samples are delivered to the Columbia Analytical sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 8-2 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);

- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Chemist and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Chemist for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. Columbia Analytical has five walk-in cold storage units which house the majority of sample containers received at the laboratory. In addition, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. Columbia Analytical also has seven sub-zero freezers capable of storing samples at -20° C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

Columbia Analytical adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is printed or written on the analytical raw data. For analyses with a holding time prescribed in hours it is essential that the sample collection time is provided, so holding time compliance can be demonstrated. If not, the sample collection time is assumed as the earliest in the day (i.e. the most conservative).

Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature for 30 days, soil samples are retained at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Environmental Health and Safety Manual.* All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

8.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN)*. Figure 8-1 is a copy of the chain-of-custody form routinely used at Columbia Analytical.

Facility security and access is important in maintaining the integrity of samples received at Columbia Analytical/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The Columbia Analytical facility is equipped with an alarm system and Columbia Analytical employs a private security firm to provide nighttime and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC).*

8.4 Project Setup

The analytical method(s) used for sample analysis are chosen based on the client's requirements. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Chemist will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Chemist ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the service request. To communicate and specify project-specific requirements, a Tier V form (Figure 8-3) is used and accompanies the service request form.

Table 8-1
Sample Preservation and Holding Times

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert (Standard Methods)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total (Standard Methods)	W, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci (SM 9230B)	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia (SM 4500NH3)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Chlorine, Total Residual (SM 4500Cl F)	W, DW	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500CN E,G)	W, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable (SM 4500CN I)	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron (CAS SOP)	W, DW	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0)	W, DW	P,G	None Required	28 days
Fluoride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Hardness (SM 2340C)	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrite (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Orthophosphate (EPA 365.3)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Perchlorate (EPA 314.0)	W, DW	P,G	Protect from temp. extremes	28 days
Phenolics, Total (EPA 420.1)	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total (EPA 160.3 & SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS) (SM 2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500SiO2 C)	W	P Only	Cool, 4°C	28 days
Specific Conductance (EPA 120.1 & SM 2510B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Sulfide (SM 4500S2 F)	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite (SM 4500SO3 B)	W	P,G	None Required	24 hours
Surfactants (MBAS) (SM 5540C)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Metals				
Metals, except CrVI and Mercury	W, DW	P,G	HNO_3 to pH<2	6 months
(EPA 200.7, 200.8, 200.9, 6010, 6020)	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
(EPA 245.1, 7470, 7471)	S	P,G	Cool, 4°C	28 days
1631E	W	F	Cool, 4°C, HCl or H_2SO_4 to pH<2	90 days
1631E	S	F	Freeze < -15ºC	1 Yr
Methyl Mercury 1630	W	F	HCL to pH<2	6 months
Organic Tests				
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (EPA 415.1, 9060 & SM 5310C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (EPA 9020)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total (EPA 8015)	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction
Pharma Personal Care Products 1694	W	Amber G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	14 days until extraction; 40 days after extraction
Nitroaromatics and Nitramines 8330, 8330B	W,S	G, Teflon-Lined Cap	Cool, 4°C	S 14, W 7 days until extraction; 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Organic Test				
Methanol in Process Liquid NCASI 94.03	L	G, Teflon-Lined Cap	Cool, 4°C	30 days
HAPS – Condensates NCASI 99.01		G, Teflon-Lined Cap	Cool, 4°C	14/30 days
HAPS – Impinger/Canisters NCASI 99.02			Cool, 4°C	21 days
Perfluorinated Compounds HPLC/MS/MS	W	P	Cool, 4°C	14 days until extraction; 40 days after extraction
PBDE/PBB – ROHS GC/MS			RT	40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics) (EPA 8015)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days
Purgeable Halocarbons (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE) (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile (EPA 624, 8260)	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
EDB and DBCP (EPA 8260)	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Alcohols and Glycols (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270, 8310)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides (EPA 8141, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides (EPA 8151)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organotins (CAS SOP)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics (EPA 1653A)	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Resin and Fatty Acids (NCASI 85.02)	W	G, Teflon-Lined Cap	NaOH to pH $\geq$10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined Septum Cap	Ascorbic Acid, HCl to pH≤2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	DW	G, Teflon-Lined Septum Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Carbamates, Carbamoyloximes (EPA 531.1)	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH<3; 80 mg/L $Na_2S_2O_3$ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/4omL NaS; Cool, <6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$ if Res.Cl., Cool, 4°C,	7days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate (EPA 547)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$, Cool, 4°C	14 days
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Nitrosoamines (EPA 521)	DW	G, Amber, Teflon-Lined Cap	Dechlorinate at collection[g] Cool, 4°C	14 days until extraction; 28 days after extraction
Selected Pesticides and Flame Retardants (EPA 527)	DW	G, Amber, Teflon-Lined Cap	See method Cool, 4°C	14 days until extraction; 28 days after extraction
Explosives (EPA 529)	DW	G, Amber, Teflon-Lined Cap	See method Cool, 4°C	14 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Mercury (EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics (EPA 1311/8260)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction

a For EPA SW-846 methods the method number is listed generically, without specific revision suffixes.
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass, F- Fluoropolymer
d For chlorinated water samples
e The maximum holding time is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

Figure 8-1
Chain of Custody Form

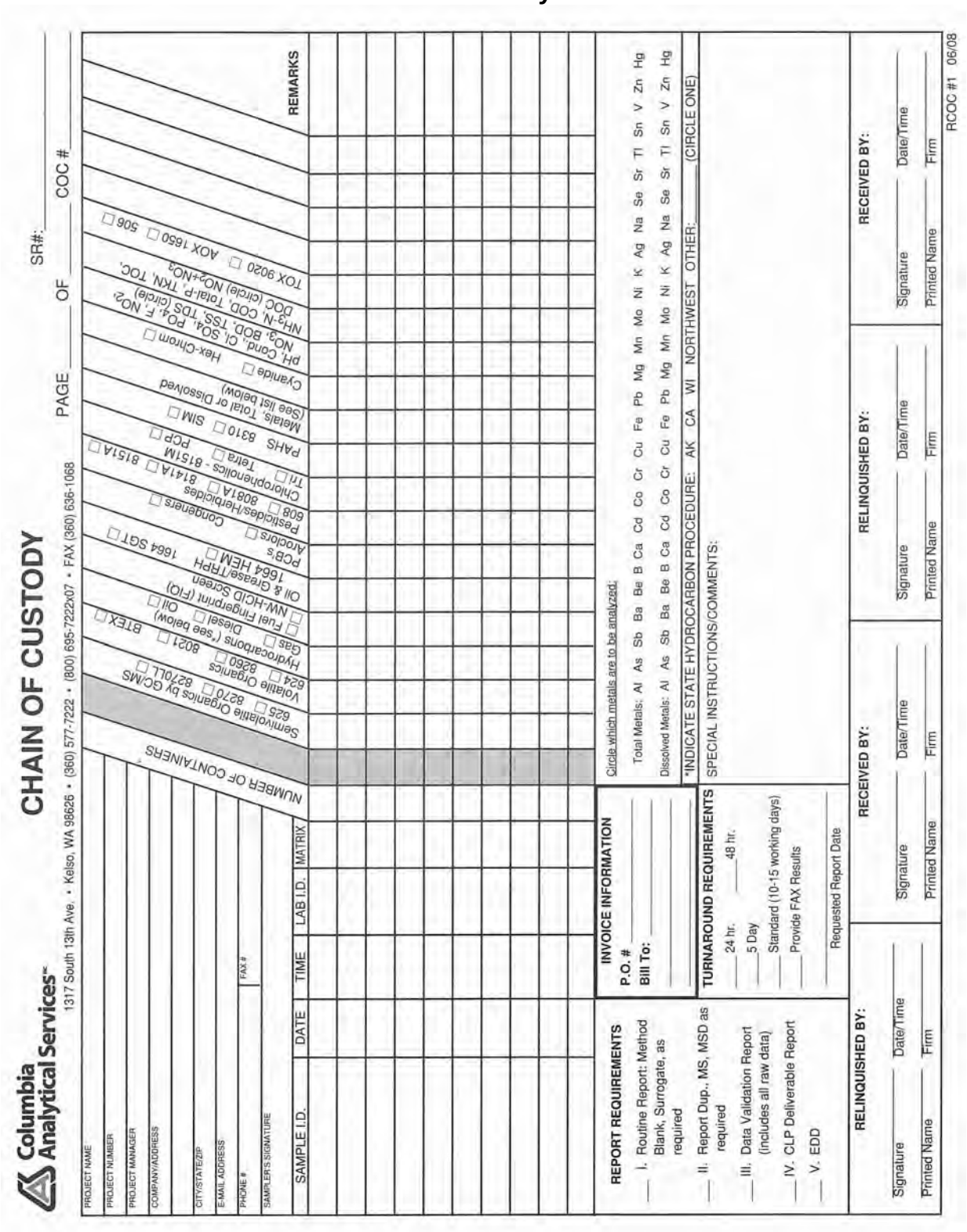


Figure 8-2

Columbia Analytical Services, Inc. PC_____
Cooler Receipt and Preservation Form

Client / Project:_____ Service Request *K09*_____

Received:_____ Opened:_____ By:_____

1. Samples were received via? ***US Mail*** ***Fed Ex*** ***UPS*** ***DHL*** ***GH*** ***GS*** ***PDX*** ***Courier*** ***Hand Delivered***
2. Samples were received in: (circle) ***Cooler*** ***Box*** ***Envelope*** ***Other***_____ ***NA***
3. Were custody seals on coolers? NA Y N If yes, how many and where?_____
   If present, were custody seals intact? Y N If present, were they signed and dated? Y N
4. Is shipper's air-bill filed? If not, record air-bill number:_____ NA Y N

5. **Temperature of cooler(s) upon receipt (°C):** _____ _____ _____ _____
   Temperature Blank (°C): _____ _____ _____ _____
   Thermometer ID: _____ _____ _____ _____
6. If applicable, list Chain of Custody Numbers: _____ _____ _____ _____
7. Packing material used. ***Inserts*** ***Baggies*** ***Bubble Wrap*** ***Gel Packs*** ***Wet Ice*** ***Sleeves*** ***Other***_____
8. Were custody papers properly filled out (ink, signed, etc.)? NA Y N
9. **Did all bottles arrive in good condition (unbroken)?** *Indicate in the table below.* NA Y N
10. Were all sample labels complete (i.e analysis, preservation, etc.)? NA Y N
11. Did all sample labels and tags agree with custody papers? *Indicate in the table below* NA Y N
12. **Were appropriate bottles/containers and volumes received for the tests indicated?** NA Y N
13. Were the pH-preserved bottles tested* received at the appropriate pH? *Indicate in the table below* NA Y N
14. Were VOA vials and 1631 Mercury bottles received without headspace? *Indicate in the table below.* NA Y N
15. **Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?** NA Y N
16. Was C12/Res negative? NA Y N

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume added	Reagent Lot Number	Initials

**Does not include all pH preserved sample aliquots received. See sample receiving SOP (SMO-GEN).*

Additional Notes, Discrepancies, & Resolutions:_____

Figure 8-3
Tier V Form

Client :

Project Chemist :

Project Name :

Service Request :

Project Number :

SMO LimsTemplate ID :

Project Description :

QAPP/SOW Information :

Reporting

Tier Level :

PDF:

Report to :

In result field use :

EDD :

Flagging Requirements :

Other Requirements :

Sample Considerations

Sample Limitations :

Sample Prep/Analysis :

Non-Standard Holdtimes :

Historical Data :

Comments :

9.0 ANALYTICAL PROCEDURES

Columbia Analytical employs methods and analytical procedures from a variety of external sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 18.0. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by Columbia Analytical is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix E. Further details are described below.

9.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

Columbia Analytical maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

9.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

9.3 Modified Procedures

Columbia Analytical strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

9.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that Columbia Analytical has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.
2) All (field) samples in a batch are of the same matrix.
3) The QC samples to be processed with the (field) samples include:
   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b) Laboratory Control Sample

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.
4) A single lot of reagents is used to process the batch of samples.
5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.

7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

9.5 Specialized Procedures

Columbia Analytical not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are trace-level Mercury and Methylmercury analyses, reductive precipitation metals analysis, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs).

9.6 Sample Cleanup

Columbia Analytical commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods (3620, 3630, 3640, 3660, and 3665) for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

10.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at Columbia Analytical are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files.

Records are maintained to provide traceability of reference materials.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standards. All analytical measurements generated at Columbia Analytical are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type. This information is provided to the client when requested.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective; is taken out of service until it is repaired. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

10.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators. Bound record books are kept which contain daily-recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC).* The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standard on an annual basis.

10.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC. The weights are recertified using NIST traceable standards by an accredited metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by an accredited metrology organization.

10.3 Water Purification Systems

Columbia Analytical uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Following a written SOP, the status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily in a bound record book. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked on a daily basis at a point downstream of the purification system at a tap in the laboratory.

10.4 Source and Preparation of Standard Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. Columbia Analytical relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

10.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

10.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

10.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be “dropped” from the resulting calibration curve.

10.8 GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working

range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.10 LC/MS Systems

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.11 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be “dropped” from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at Columbia Analytical include total phenolics, phosphates, surfactants and tannin-lignin.

10.12 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be “dropped” from the resulting calibration curve. Standard Columbia Analytical acceptance limits are used to evaluate the calibration curve prior to sample analysis.

10.13 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be “dropped” from the resulting calibration curve. A correlation coefficient of $\geq$ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and drinking water inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

10.14 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

10.15 Ion-selective electrode

The method-prescribed numbers of standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

10.16 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP Checking Volumetric Labware (ADM-VOLWARE)*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

10.17 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

11.0 QUALITY CONTROL

A primary focus of Columbia Analytical's Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. Columbia Analytical has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

11.1 Quality Control Objectives

11.1.2 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria is met.

11.1.3 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination). Columbia Analytical utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

11.1.4 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

11.1.5 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the Quality Assurance Manager. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

11.1.6 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. Columbia Analytical has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample.

These include the *SOP for Subsampling and Compositing of Samples* and the *SOP for Tissue Sample Preparation*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

11.1.7 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using Columbia Analytical or project-specified data qualifiers.

11.2 Method Detection Limits and Method Reporting Limits

Method Detection Limits (MDL) for methods performed at Columbia Analytical/Kelso is determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation (ADM-MDL),* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation- LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

11.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

11.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

11.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

11.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

11.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

11.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in the calibration. Once it is determined that there is no reference material defect or systematic error in preparation of the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

11.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

11.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

11.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

11.3.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in

the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

11.3.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$Relative\ Percent\ Difference\ (RPD) = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

11.3.13 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

11.3.14 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

11.3.15 Control Charting

The generation of control charts is routinely performed at Columbia Analytical. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

11.3.16 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at Columbia Analytical undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at Columbia Analytical; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

Columbia Analytical reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the *SOP for Laboratory Data Review Process.*

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the “before” and “after” integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (SOP ADM-INT).

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.
    2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
    2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS Methods - Two criteria are used to verify identification:
    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.
- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures as described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are

performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including alternative analysis.

- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP *Confirmation Procedure for GC and HPLC Analysis (SOC-CONF).* If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including additional cleanup.
- Surrogate Results (Organic) – Following sample analysis and data reduction, the percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.
- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 are used to determine if the LCS is effective in validating the analytical system and the associated samples.

- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. Columbia Analytical maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Reporting and Report Generation* addresses the flagging and qualification of data. The Columbia Analytical-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the project chemist to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Chemist verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Chemist accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the Columbia Analytical client.

12.5 Documentation

Columbia Analytical maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the *SOP for Data Archiving*.

12.5.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

12.6 Deliverables

In order to meet individual project needs, Columbia Analytical provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) to following specialized deliverables:

- ADEC – Alaska Department of Conservation specified data package
- ACOE/HTRW – Army Corps of Engineers specified data package and reporting requirements (HTRW, CERP, FUDS, etc.)
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, Columbia Analytical provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. Columbia Analytical is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 12-1
Descriptions of Columbia Analytical Standard Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results and acceptance criteria for applicable organic methods
5. Chain of custody documents
6. Dates of sample preparation and analysis for all tests

Tier II and IIA. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Tier IIA also includes Laboratory Control Sample (LCS) result(s) with calculated recovery and including associated acceptance criteria

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or Quality Control check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Summary forms for associated QC and calibration parameters
6. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Results and spectra of TIC compounds will be included upon request.

Tier IV. CLP-Level Data Validation Package.

A complete Data Validation Package containing all sample results, quality control and calibration results, and raw data necessary to fulfill all deliverable requirements of an EPA Contract Laboratory Program (CLP) data package.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of Columbia Analytical/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of Columbia Analytical/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which Columbia Analytical/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of Columbia Analytical/Kelso are conducted regularly under the direction of the Quality Assurance Manager. The internal audit procedures are described in the *SOP for Internal Audits*. The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that systems, technical operations, hardcopy data, and electronic data are assessed.
- Hardcopy report audits – minimum of 3 per quarter.
- Electronic audit trail reviews – each applicable instrument per quarter.

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

13.2 Performance Audits

Columbia Analytical/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. Columbia Analytical routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the Columbia Analytical Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformity (NCAR) procedures.

Table 13-1
Current Columbia Analytical Performance and System Audit Programs

Federal and National Programs

- The TNI (The NELAC Institute) National Environmental Laboratory Accreditation Program (NELAP) Accredited Drinking Water, Non-Potable Water, Solid & Hazardous Waste, and Biological Tissue Laboratory
- ANSI-ASQ National Accreditation Board/ACLASS ISO 17025:2005
- DoD- ELAP Environmental Laboratory Accreditation Program
- Naval Facilities Engineering Service Center
  Validated Laboratory for NFESC Parameters
- U.S. Army Corps of Engineers
  Approved Laboratory for USACE Projects
- U.S. EPA Region 8
  Approved Drinking Water Laboratory

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory, Lab I.D. UST040
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of Arkansas, Department of Environmental Quality
  Certified Environmental Laboratory, Lab I.D. 88-0637
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Colorado, Department of Public Health and Environment
  Certified Drinking Water Laboratory
- State of Florida, Department of Health
  Primary NELAP Accreditation No. E87412
- State of Georgia, Department of Natural Resources
  Certified Drinking Water Laboratory
- State of Hawaii, Department of Health
  Certified Drinking Water Laboratory
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Indiana, Department of Health
  Certified Drinking Water Laboratory, Lab I.D. C-WA-01
- State of Louisiana, Department of Environmental Quality
  Accredited Environmental Laboratory, Lab I.D. 3016
- State of Louisiana, Department of Health and Hospitals
  Accredited Drinking Water Laboratory, Lab I.D. LA080001
- State of Maine, Department of Human Services
  Certified Environmental Laboratory, Lab I.D. WA0035
- State of Michigan, Department of Environmental Quality
  Certified Drinking Water Laboratory, Lab I.D. 9949

Table 13-1 (continued)

State and Local Programs (continued)

- State of Minnesota, Department of Health
  Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nevada, Division of Environmental Protection
  Certified Drinking Water Laboratory, Lab I.D. WA35
- State of New Jersey, Department of Environmental Protection
  Accredited Environmental Laboratory, Lab I.D. WA005
- State of New Mexico, Environment Department
  Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
  Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
  Accredited Environmental Laboratory, Lab I.D. WA200001
- State of South Carolina, Department of Health and Environmental Control
  Certified Environmental Laboratory, Lab I.D. 61002
- State of Utah, Department of Health, Division of Laboratory Services
  Accredited Environmental Laboratory
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
  Accreditation No. C1203
- State of Wisconsin, Department of Natural Resources
  Accredited Environmental Laboratory, Lab I.D. 998386840

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at Columbia Analytical (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at Columbia Analytical contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at Columbia Analytical before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at Columbia Analytical. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D.

This inventory or “parts list” also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix D for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form. The laboratorys procedure and responsibilities for addressing nonconforming work is defined in the SOP ADM-CA *Corrective Action.*

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives outlined in Section 11, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist is notified in order to ascertain if the client needs to be notified.

In the event that analyses produce nonconformances with data or results, the problem and the corresponding corrective actions taken are documented on a *Nonconformity and Corrective Action Report* (See Figure 15-1) following the requirements in the *SOP for Corrective Action* (SOP No. ADM-CA). This form is utilized to determine the root cause of the nonconformity and to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the project chemist) is responsible for documenting the complaint. If the project chemist is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (ADM-FDBK).

Figure 15-1

Nonconformity and Corrective Action Report

NCAR No: *Assigned by QA*

PROCEDURE (SOP or METHOD): EVENT DATE: _____

EVENT:
- ☐ Missed Holding Time
- ☐ Method Blank Contamination
- ☐ Equipment Failure
- ☐ SOP Deviation
- ☐ QC Failure
- ☐ Login Error
- ☐ Unacceptable PT Sample Result
- ☐ Other (describe):
- ☐ Lab Error (spilled sample, spiking error, etc.)
- ☐ Project Management Error

INCLUDE NUMBER OF SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED

DETAILED DESCRIPTION

ORIGINATOR: _____ DATE: _____

PROJECT MANAGER(S): _____ NOTIFIED BY: _____ DATE: _____

ROOT CAUSE OF NON-CONFORMITY (POTENTIAL CAUSES COULD BE TRAINING, COMMUNICATION, SPECIFICATIONS, EQUIPMENT, KNOWLEDGE)

What is the cause of the error or finding:

CORRECTIVE ACTION AND OUTCOME

Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent its reoccurrence. Include Project Manager Instructions here.

Is the data to be flagged in the Analytical Report with an appropriate qualifier? ☐ **No** ☐ **Yes**

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____ Date: _____
Comments:

QA PM Verification and Approval of Corrective Action _____ Date: _____
Comments:

Project Manager Verification and Approval of Corrective Action _____ Date: _____
Comments:

Customer Notified by ☐ Telephone ☐ Fax ☐ E-mail ☐ Narrative ☐ Not notified

(Attach record or cite reference where record is located.)

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by Columbia Analytical personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists as required (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QAM provides the Laboratory Director with quarterly reports that summarize the various QA/QC activities that occurred during the previous quarter. The report addresses such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

The Laboratory Director also performs an annual management review of the quality and management systems to identify any necessary changes or improvements to the quality system or quality assurance policies. This review is documented in a report *Management Quality System and Testing Review* and sent to senior management.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at Columbia Analytical are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at Columbia Analytical when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at Columbia Analytical. Safety training begins with the reading of the *Environmental Health and Safety Manual.* Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer.

Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s). Quality Systems training begins with Quality Assurance orientation for new employees and reading the *Quality Assurance Manual.* During the employees first year, the employee attends *Core Ethics* training and learns about Columbia Analytical Services quality systems. Each employee participates in annual *Ethics Refresher* training, which is part of the Columbia Analytical Improper Practices Prevention Program.

Columbia Analytical also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The “CAS University” education system, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained in the QA department. Training requirements and its documentation are described in the SOP (ADM-TRANDOC) *Documentation of Training.* A training plan is developed whenever an employee starts a new procedure to new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

17.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible, special requirements are as follows:
    - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
    - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
    - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 17-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

17.2 Continuing Demonstration of Proficiency

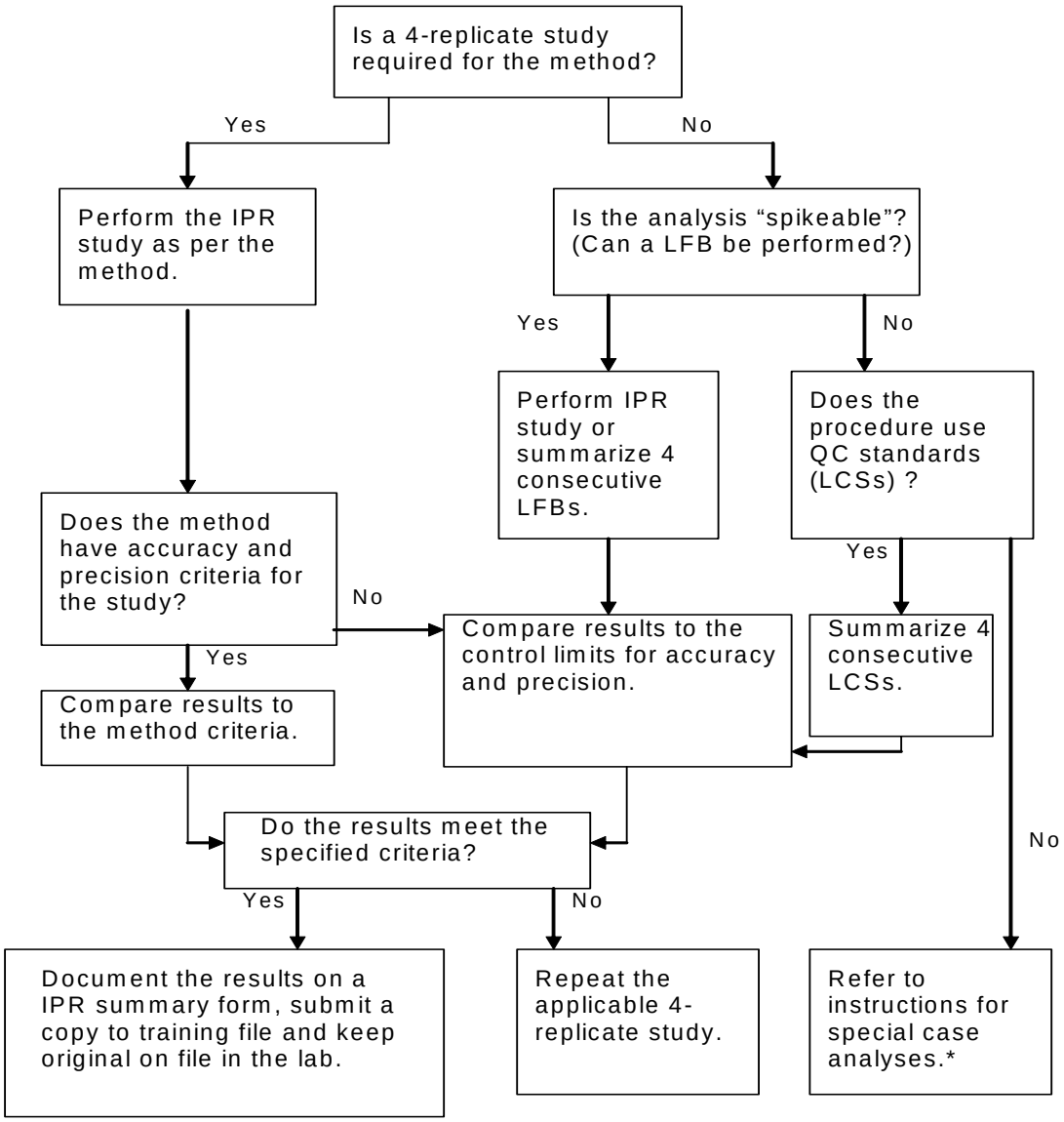
A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

17.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and Columbia Analytical resumes. QA maintains a database to record the various technical skills and training acquired while employed by Columbia Analytical. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Training (SOP No. ADM-TRANDOC).*

Figure 17-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per NELAP requirements
*Total Settleable Solids: Successful PT sample analysis and duplicate results with RPD<10%.
*Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
* Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES – EXTERNAL DOCUMENTS

The analytical methods used at Columbia Analytical generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at Columbia Analytical are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E)
- *Department of Defense Quality Systems Manual for Environmental Laboratories*, Final Version 3 (January 2006).
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 4.1, 4/22/2009
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).
- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4.
- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 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) and Supplements.
- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.
- *Standard Methods for the Examination of Water and Wastewater*, 18th Edition (1992); 19th Edition (1995), 20th Edition (1998). See Introduction in Part 1000.
- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.
- 40 CFR Part 141, National Primary Drinking Water Regulations.

- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.
- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).
- Annual Book of ASTM Standards, Part 31, Water.
- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM03.1, OLM03.2, OLM04.2, and OLM04.3.
- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0, ILM04.1, and ILM05.2.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).
- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).
- *Identification and Listing of Hazardous Waste,* California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).
- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

and

STANDARD OPERATING PROCEDURES

QA Program Files

Quality Assurance Manual	10/2/2009
Software Quality Assurance Plan	7/11/05
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	AppSignatories.pdf
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
Quality Control Acceptance Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001
Standard Operating Procedure Database	TrainDat.mdb

Corporate – Policies

POLICY TITLE	POLICY DATE	DATE APPROVED	DATE EFFECTIVE
CAS Quality and Ethics Policy Statement	March 2009	3/19/09	3/19/09
Policy for Data Review and Validation	May 2009	5/5/09	7/1/09
Policy for Internal Quality Assurance Audits	May 2009	5/5/09	7/1/09
Policy for Standards and Reagents Expiration Dates	September 2009	Final draft	9/28/09
Policy for Quality Assurance for Non-Regulated Testing	Draft	-	-
Policy for Use of Accreditation Organization's Name, Symbols, and Logos	Draft	-	-
Policy for Conducting Research, Technical Investigations, and Method Development	In development	-	-

Administrative SOP Corporate

SOP TITLE	SOP Code	Rev	SOP Date
SOP for Checking New Lots of Chemicals for Contamination	ADM-CTMN	4	1/26/09
SOP for Control Limits	ADM-CTRL_LIM	6	9/28/07
SOP for Corrective Action	ADM-CA	5	9/12/07
SOP for Data Recall	ADM-DATARECALL	0	9/21/07
SOP for Document Control	ADM-DOC_CTRL	7	1/27/09
SOP for Documentation of Training	ADM-TRANDOC	10	12/6/07
SOP for Estimation of Uncertainty of Measurements	ADM-UNCERT	4	12/30/08
SOP for Handling Customer Feedback	ADM-FDBK	4	12/10/07
SOP for Making Entries into Logbooks and onto Benchsheets	ADM-DATANTRY	8	9/8/09
SOP for Managerial Review of the Laboratory's Quality Systems	ADM-MGMTRVW	2	11/7/07
SOP for Manual Integration of Chromatographic Peaks	ADM-INT	3	8/28/07
SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	ADM-MDL	9	9/8/09
SOP for Preparation of Electronic-data for Organic Analyses for Electronic-data Audits	ADM-E_DATA	3	8/29/07
SOP for Preparation of SOPs	ADM-SOP	8	11/14/08
SOP for Preventive Action	ADM-PA	0	11/14/08
SOP for Proficiency Testing Sample Analysis	ADM-PTS	1	9/28/07
SOP for Purchasing Through SOP Purchasing Agent in Kelso	ADM-PUR	2	12/10/07
SOP for Qualification of Subcontract Laboratories Outside of SOP Network	ADM_SUBLAB	4	12/29/08
SOP for Significant Figures	ADM-SIGFIG	8	1/28/09

Administrative SOP Kelso

SOP Title	FILE NAME
CHECKING PIPETTE CALIBRATION	**ADM-CPIP**
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	**ADM-ECP**
CONTROL CHARTING QUALITY CONTROL DATA	**ADM-CHRT**
DATA ARCHIVING	**ADM-ARCH**
DATA REPORTING AND REPORT GENERATION	**ADM-RG**
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	**ADM-DOD**
ELECTRONIC DATA BACKUP AND ARCHIVING	**ADM-EBACKUP**
INTERNAL QUALITY ASSURANCE AUDITS	**ADM-IAUD**
LABORATORY BALANCE MONITORING AND CALIBRATION	**ADM-BAL**
LABORATORY DATA REVIEW PROCESS	**ADM-DREV**
PROJECT MANAGEMENT	**ADM-PCM**
REAGENT LOGIN AND TRACKING	**ADM-RLT**
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	**ADM-SEMC**
SAMPLE BATCHES	**ADM-BATCH**
SAMPLE MANAGEMENT SOPS	**FILE NAME**
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC

Technical SOP Kelso

SOP Title	FILE NAME
COLIFORM, TOTAL (DRINKING WATER)	BIO-9221DW
COLIFORM, FECAL	BIO-9221FC
COLIFORM, TOTAL	BIO-9221TC
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D
COLILERT® and COLITAG	BIO-9223
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B
COLILERT® COMPLETED TEST VERIFICATION OF E. COLI IN MUG CULTURES	BIO-CCT
ENTEROLERT	BIO-ENT
HEPTEROTROPHIC PLATE COUNT	BIO-HPC
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN
EPA CLP ORGANICS ANALYSES	CLP_ORGA
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510
CONTINUOUS LIQUID - LIQUID EXTRACTION	EXT-3520
SOLID PHASE EXTRACTION	EXT-3535
SOXHLET EXTRACTION	EXT-3540
AUTOMATED SOXHLET EXTRACTION	EXT-3541
ULTRASONIC EXTRACTION	EXT-3550
WASTE DILUTION EXTRACTION	EXT-3580
SILICA GEL CLEANUP	EXT-3630
REMOVAL OF SULFUR USING COPPER	EXT-3660
REMOVAL OF SULFUR USING MERCURY	EXT-3660M
SULFURIC ACID CLEANUP	EXT-3665
CARBON CLEANUP	EXT-CARCU
DIAZOMETHANE PREPARATION	EXT-DIAZ
FLORISIL CLEANUP	EXT-FLOR
ORGANIC EXTRACTIONS GLASSWARE CLEANING	EXT-GC
PREPARATION OF REAGENTS AND BLANK MATRICES USED IN SEMIVOLATILE ORGANICS ANALYSIS	EXT-REAG
ADDITION OF SPIKES AND SURROGATES	EXT-SAS
SOLID PHASE DISPERSION IN TISSUES	EXT-SPD
MEASURING SAMPLE WEIGHTS AND VOLUMES FOR ORGANIC ANALYSIS	EXT-WVOL
FACILITY AND LABORATORY CLEANING	FAC-CLEAN
OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS	FAC-WATER
FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020
COLOR	GEN-110.2
HARDNESS, TOTAL	GEN-130.2
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-160.1
SOLIDS, TOTAL SUSPENDED (TSS)	GEN-160.2
TOTAL SOLIDS	GEN-160.3
SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES	GEN-160.4
SETTEABLE SOLIDS	GEN-160.5
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300.1
ACIDITY	GEN-305.2
ALKALINITY TOTAL	GEN-310.1

PERCHLORATE BY ION CHROMATOGRAPHY	GEN-314.0
CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)	GEN-325.3
CHLORINE, TOTAL/FREE RESIDUAL	GEN-330.4
TOTAL RESIDUAL CHLORINE - METHOD 330.5	GEN-330.5
TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION	GEN-335
AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1
AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE	GEN-350.3
NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS	GEN-353.2
NITRITE BY COLORIMETRIC PROCEDURE	GEN-354.1
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3
DISSOLVED SILICA	GEN-370.1
GRAVIMETRIC SULFATE	GEN-375.3
SULFIDE, TITRIMETRIC (IODINE)	GEN-376-1
SULFIDE, METHYLENE BLUE	GEN-376-2
PHENOLICS, TOTAL	GEN-420.1
MBAS	GEN-425.1
HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION	GEN-5050
BIOCHEMICAL OXYGEN DEMAND	GEN-5210B
HALIDES, ADSORBABLE ORGANIC (AOX) - SM 5320B	GEN-5320B
TANNIN AND LIGNIN	GEN-5550
CYANIDE EXTRACTION OF SOLIDS AND OILS	GEN-9013
HALIDES, TOTAL ORGANIC (TOX)	GEN-9020
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030
TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY	GEN-9076
CARBON, TOTAL ORGANIC IN SOIL	GEN-ASTM
AUTOFLUFF	GEN-AUTOFLU
SULFIDES, ACIDS VOLATILE	GEN-AVS
HEAT OF COMBUSTION	GEN-BTU
CYANIDE, WEAK ACID DISSOCIABLE	GEN-CNWAD
CHEMICAL OXYGEN DEMAND	GEN-COD
CONDUCTIVITY IN WATER AND WASTES	GEN-COND
CORROSIVITY TOWARDS STEEL	GEN-CORR
HEXAVALENT CHROMIUM - COLORIMETRIC	GEN-CR6
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109
FERROUS IRON IN WATER	GEN-FeII
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26
MERCURY IN COAL SAMPLE PREPARATION BY PARR BOMB COMBUSTION	GEN-HGPREP
HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD
TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS
ION CHROMATOGRAPHY	GEN-IONC
COLOR, NCASI	GEN-NCAS
OXYGEN CONSUMPTION RATE	GEN-O2RATE
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU
Ph IN SOIL AND SOLIDS	GEN-Phs

Ph IN WATER	GEN-Phw
PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM
PARTICLE SIZE DETERMINATION	GEN-PSP
SULFIDES, REACTIVE	GEN-RS
TOTAL SULFIDE BY PSEP	GEN-S2PS
SULFITE	GEN-SO3
SPECIFIC GRAVITY	GEN-SPGRAV
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS
THIOCYANATE	GEN-THIOCN
NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN
POST DIGESTION DETERMINATION OF TOTAL KJELDAHL NITROGEN BY SEMIAUTOMATED COLORIMETRY	GEN-TKNAA
TOTAL ORGANIC CARBON IN WATER	GEN-TOC
TURBIDITY MEASUREMENT	GEN-TURB
ULTIMATE BOD	GEN-UBOD
GLASSWASHING FOR INORGANIC ANALYSES	GEN-WASH
Quantitative Determination of Carbamate Pesticides by High Performance Liquid Chromatography/Tandam Mass Spectrometry (HPLC/MS/MS)	LCP-8321
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY(HPLC)	LCP-8330B
QUANTITATION OF NITROAROMATICS AND NITRAMINES IN WATER, SOIL, AND TISSUE BY LIQUID CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY (LC-MS/MS)	LCP-LCMS4
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	LCP-NITG
QUANTITATION OF NITROPHENOLS IN SOLIS BY LIQUID CHROMATOGRAPHYAND TANDEM MASS SPECTORMETRY (LC-MS/MS)	LCP-NITRO
METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630S
METHYL MERCURY IN TISSUE BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630T
METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630W
MERCURY IN WATER BY OXIDATION, PURGE&TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY	MET-1631
MERCURY IN WATER	MET-245.1
METALS DIGESTION	MET-3005A
METALS DIGESTION	MET-3010A
METALS DIGESTION	MET-3020A
METALS DIGESTION	MET-3050B
CLOSED VESSEL OIL DIGESTION	MET-3051M
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020
ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7062
METALS DIGESTION	MET-7195
MERCURY IN LIQUID WASTE	MET-7470A
MERCURY IN SOLID OR SEMISOLID WASTE	MET-7471A/B
SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7742
CATION-EXCHANGE CAPACITYOF SOILS (SODIUM ACETATE) - METHOD 9081	MET-9081
SAMPLE PREPARATION OF AQUEOUS SAMPLES BY "CLEAN" TECHNIQUES	MET-ACT
BIOACCESSIBILITY OF METALS IN SOIL AND SOLID WASTE	MET-BIOACC
METALS DIGESTION	MET-DIG
FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRIC ANALYSES	MET-FAA
SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT
METALS LABORATORY GLASSWARE CLEANING	MET-GC

DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)	MET-GFAA
DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES	MET-ICP
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICP.MS
MULTIPLE EXTRACTION PROCEDURE	MET-MEP
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC
METALS AND SEMIVOLATILES TCLP EXTRACTION (EPA METHOD 1311)	MET-TCLP
SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS	MET-TDIG
TISSUE SAMPLE PREPARATION	MET-TISP
GRAVIMETRIC DETERMINATION OF HEAXANE EXTRACTABLE MATERIAL (1664)	PET-1664
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	PET-GRO
ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS	PET-SVF
ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS	PHC-WIDRO
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND INTERNAL CHAIN OF CUSTODY	SMO-SCOC
SAMPLE DISPOSAL	SMO-SDIS
CHLORINATED PHENOLICS BY IN-SITU ACETYLATION AND GC/MS	SOC-1653A
PHARMACEUTICALS, PERSONAL CARE PRODUCTS AND ENDOCRINE DISRUPTING COMPOUNDS IN WATER BY HPLC/TANDEM MASS SPECTROMETRY (HPLC/MS/MS)	SOC-1694
1,8-DIHYDROXYANTHRAQUINONE BY GC/MS SIM	SOC-18DHYDRAQ
GEL PERMEATION CHROMATOGRAPHY	SOC-3640A
ACETAMIDE HERBICIDE DEGRADATES IN DRINKING WATER BY SPE AND HPLC/MS/MS	SOC-535
ORGANOCHLORINE PESTICIDES AND PCBs (METHOD 608)	SOC-608
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-625
GLYCOLS	SOC-8015M
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081
PCBS AS AROCLORS - METHOD 8082A	SOC-8082AAr
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD - METHOC 8082A	SOC-8082ACo
PCBS AS AROCLORS	SOC-8082Ar
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD	SOC-8082C
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141
CHLORINATED HERBICIDES	SOC-8151
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - METHOD 8270D	SOC-8270D
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE	SOC-8270L
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SOC-8270P
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SOC-8270S
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	SOC-8310
ALDEHYDES BY HPLC	SOC-8315A

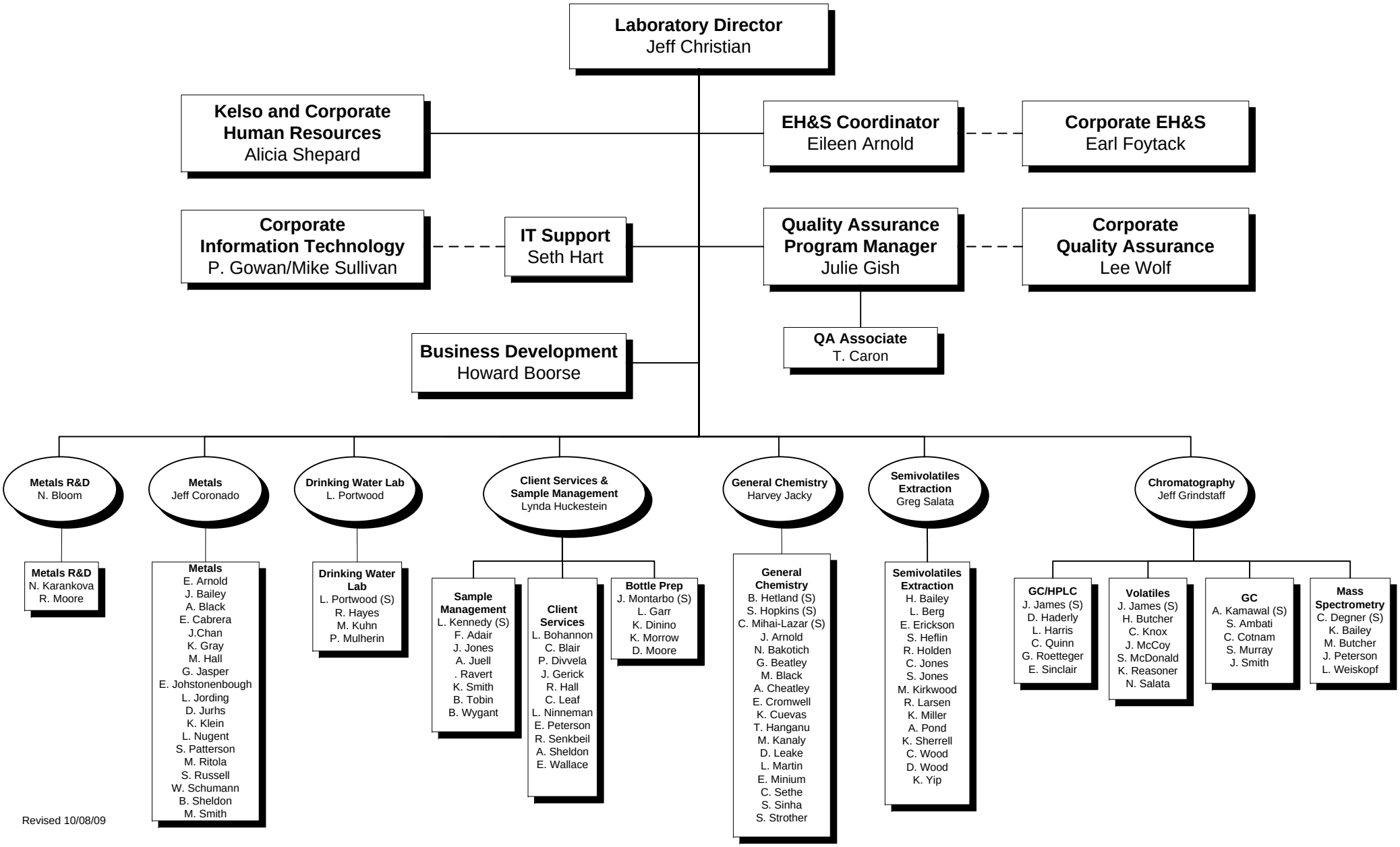
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8330
NITROGLYCERIN AND PETN BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8332
RESIN AND FATTY ACIDS BY GC/MS - NCASI METHOD 85.02 MODIFIED	SOC-85.02
METHANOL IN PROCESS LIQUIDS AND STATIONARY SOURCE EMISSIONS	SOC-9403
HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901
HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES	SOC-9902
BUTYLTINS	SOC-BUTYL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES USING EPA 8000C	SOC-CAL8000C
CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES	SOC-CONF
CPSC PHTHALATES BY GC/MS SELECTIVE ION MONITORING	SOC-CPSC
DIMP	SOC-DIMP
DMD SYNTHESIS	SOC-DMD
TOTAL OLEANOLIC ACID SAPONINS IN WATER BY ACID HYDROLYSIS AND HPLC/MS/MS	SOC-LCMS3
PERCENT LIPIDS IN TISSUE	SOC-LIPID
MONOCHLOROACETIC ACID BY GC-ECD	SOC-MCA
NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES	SOC-NONYL
ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC	SOC-OALC
EXTRACTION METHOD FOR ORGANOTINS IN SEDIMENTS, WATER, AND TISSUE	SOC-OSWT
CHLORINATED PESTICIDES BY GC/MS/MS, EPA METHOD 1699 MODIFIED	SOC-PESTMS2
PERFLUORINATED COMPOUNDS BY HPLC/MS/MS	SOC-PFC
PICRIC ACID AND PICRAMIC ACID BY HPLC	SOC-PICRIC
POLYBROMINATED DIPHENYL ETHERS (PBDEs) AND POLYBROMINATED BIPHENYLS (PBBs) BY GC/MS	SOC-ROHS
SEMI-VOLATILE ORGANICS SCREENING	SOC-SCR
1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC	SVD-504
ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515_4
N-NITROSAMINES BY GC/MS/MS	SVD-521
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)	SVD-525
SELECTED PESTICIDES AND FLAME RETARDANTS IN DRINKING WATER BY GC/MS (EPA METHOD 527)	SVD-527
DETERMINATION OF EXPLOSIVES AND RELATED COMPOUNDS IN DRINKING WATER BY GC/MS	SVD-529
CARBAMATES AND CARBAMOYLOXIMES IN WATER BY POST-COLUMN DERIVITIZATION HPLC	SVD-531 -1
GLYPHOSATE IN DRINKING WATER BY HPLC	SVD-547
ENDOTHALL IN DRINKING WATER BY GC/MS	SVD-548
DIQUAT AND PARAQUAT BY HPLC	SVD-549
HALOACETIC ACIDS IN DRINKING WATER	SVD-552
PURGE AND TRAP FOR AQUEOUS SAMPLES	VOC-5030
PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM	VOC-5035
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-524.2
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 602	VOC-602BTEX
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-624
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 8021	VOC-8021BTEX
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-8260
VOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING	VOC-8260S

VOA STORAGE BLANKS	VOC-BLAN
SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES	VOC-BVOC
ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)	VOC-ZHE

APPENDIX B

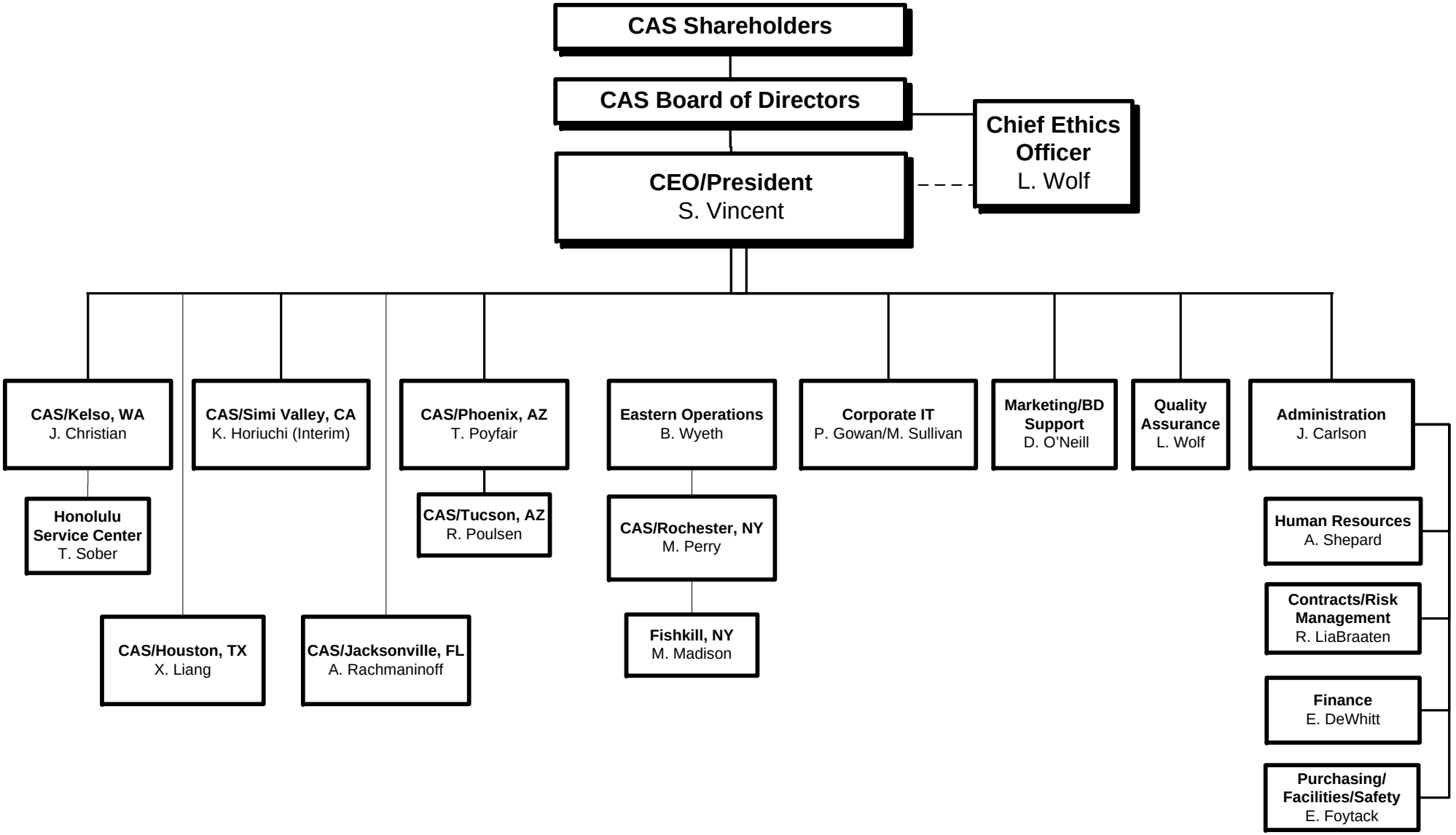
ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

Environmental and General Testing Division
Kelso, Washington
Laboratory Organization



Revised 10/08/09

Laboratory Division Organization



Revised .12/1/09

JEFFREY D. CHRISTIAN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**VICE PRESIDENT/NW REGIONAL DIRECTOR –** 1996 to Present
Responsibilities	Responsible for all phases of laboratory operations at the Kelso (WA) facility, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory (i.e. serves as the Kelso Laboratory Director, 1993-present). Also responsible for additional duties acquired as a member of the Columbia Analytical Services Holdings, Inc., Board of Directors.
Experience	**Laboratory Director, Kelso Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance. **Operations Manager, Kelso Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts. **Project Chemist and Manager, Metals Analysis Laboratory,** *Columbia Analytical Services, Kelso, Washington,* 1989-1992. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories. **Scientist,** *Weyerhaeuser Technology Center, Federal Way, Washington,* 1986-1989. Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods. **Lead Technician, Metals Lab,** *Weyerhaeuser Technology Center, Federal Way, Washington*, 1981-1986. Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing. **Research Assistant,** *ITT Rayonier, Olympic Research Division, Shelton, Washington,* 1978-1981. Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry.
Education	**B.S., Chemistry,** *Evergreen State College, Olympia, Washington,* 1993. **ICP/MS Training Course,** *VG-Elemental,* 1992. **Coursework,** *Pacific Lutheran University, Tacoma, Washington.* 1988-1989. **Coursework,** *Tacoma Community College, Tacoma, Washington.* 1970-1971, 1988-1989. **Perkin-Elmer Advanced Furnace,** *Norwalk, Connecticut,* 1986. **CERTIFICATION, Chemistry,** *L.H. Bates Technical, Tacoma, Washington,* 1978. **Coursework,** *Central Washington University, Ellensburg, Washington.* 1969-1970.
Publications/ Presentations	*Mr. Christian has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.*

JULIE GISH

1996 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER I, KELSO LAB QUALITY ASSURANCE MANAGER** – 2008 to Present
Responsibilities	Responsible for the overall implementation of the laboratory QA program. Responsible for the Quality Assurance Manual, certifications, documenting SOPs, and maintaining proficiency testing (PT) records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits and provides audit responses and initiates any corrective actions. Coordinate the analysis and reporting of PT samples. Conduct internal audits and make recommendations for corrective action.
Experience	**Scientist IV, Semi-Volatile Mass Spectrometry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Primary responsibilities were analysis, interpretation and report generation for semivolatile organics by GC/MS. Analyses included EPA 625, 8270, SIM, and other miscellaneous methodology.
	Technical Manager I, Semi-Volatile GC Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1999-2002. Primary responsibilities include supervision and oversight of semi-volatile GC department. This includes initiating new methods, staff training, workload management, and instrument maintenance/troubleshooting. Duties include departmental compliance with CAS QA and Safety policies. Responsible for analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, EPA 8141A, Organotins, and CLP Pesticides.
	Scientist III, Semi-Volatile Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-1999. Primary responsibilities were analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, and CLP-Pesticides. Secondary responsibilities include organics semi-volatile sample preparation.
	Scientist, Volatile Organics Sample Preparation, *Employer's Overload, Longview, Washington – assigned to the Columbia Analytical Services, Inc., Kelso, Washington facility,* 1996. Primary duties included the preparation of water, soil, sediment and tissue samples using EPA Methods 3510, 3520, 3540, 3550, and 3545. Other duties were the further clean up of extracts using EPA Methods 3620 (Florsil), 3610 (Alumina), 3630 (Silica gel), 3650 (Acid/Base Partitioning), and 3660 (Sulfur).
	Organics Chemist and GC/MS Chemist, *Coffey Laboratories, Portland, Oregon,* 1990-1996. Primary responsibilities included sample preparation and analysis for EPA FID, ECD, and HPLC using various EPA SW-846 and 500-series methods, as well as other methodology. Later, moved to GC/MS position which included sample preparation, analysis, and associated instrument maintenance for EPA Methods 625, 8027, and 525 BNA's. Also responsible for data review and approval of data packages.
	QC Manager/QC Supervisor and Product Manager, Corn Products, *Frito-Lay, Inc., Vancouver, Washington,* 1982-1990. Manager of the QC department overseeing three supervisors and approximately 30 technicians. Responsible for department cost, accuracy, timeliness of data and safety performance. Later, responsible for production oversight of brand name snacks. Responsible for cost, quality and safety performance over three shifts. Managed four supervisors directly and approximately 60 employees indirectly.
	Food Technologist, QA Department, *Kraft, Inc., Buena Park, California*, 1978-1981. Responsible for audits, formulations, finished product evaluation, batch reviews and technical support.
Education	**MS, Food Science, Minor in Industrial Engineering,** *Oregon State Univ. Corvallis, Oregon,* 1978. **BS, Food Science, Minor in Business Administration,** *Utah State University, Logan, Utah,* 1975
Publications/ Presentations	*Quality Improvement Team Leader,* Coffey Laboratories, Portland, Oregon. 1991
	Methods Improvement Program, Coffey Laboratories, Portland, Oregon. Seminars on Development and Implementation 1990.
	Statistical Process Control and Total Quality Management, Frito-Lay, Vancouver, Washington. Routine Training Classes 1986-1988.

GREGORY G. SALATA

2003 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**PROJECT/EXTRACTIONS MANAGER V** – 2003 to Present
Responsibilities	Responsibilities include Project Management, including quotation preparation and data reporting, as well as providing technical support to the laboratory as needed. Responsibilities also include oversight of the organic extractions lab, managing resources and providing technical support for all organic preparation work flows. 2003-Present.
Experience	**Project Manager,** *B&B Laboratories, College Station, Texas,* 1999-2003. Supervisor/responsible for analysis of TPH (waters, tissues, sediments), organotins (waters, tissues, sediments), Atterberg Limits (sediments), and total organic/inorganic carbon (sediments, waters). Also responsible for report generation on specific projects. Instrumentation operated included GCs with FID and FPD detectors, Combustion TOC, Water TOC, and Dionex Accelerated Solvent Extractor.
	Graduate Student, *Texas A&M University, College Station, Texas,* 1991-1999. While working toward MS in Oceanography, performed organic extractions for pesticides, PCBs, PAHs, and butyltins. While working toward Ph.D. in Oceanography determined stable carbon isotope ratios in sediments, waters, and bacterial phospholipid fatty acids. Other responsibilities included field sample collection, and operation/maintenance of FinniganMAT 252 isotope ratio MS.
	Analytical Chemist, *Science Applications International (SAIC), San Diego, California,* 1989-1990. Performed organic extraction and GC/FID analysis on sediment/rock samples for the Exxon Valdez oil spill.
	GC Chemist, *Analytical Technologies, San Diego, California,* 1987-1989. Responsible for analysis of volatile organics using purge and trap and GC/PID/ELCD.
Education	**Ph.D., Oceanography,** *Texas A&M University, College Station, Texas.* 1999 **MS, Oceanography,** *Texas A&M University, College Station, Texas.* 1993 **BA, Chemistry,** *University of California San Diego, Revelle College, La Jolla, California.* 1987
Publications/ Presentations	*Dr. Salata has a number of publications and published abstracts. For a list of these publications and published abstracts, please contact CAS.*
Affiliations	Society of Environmental Toxicology and Chemistry (SETAC)
	American Chemical Society

JEFFREY A. CORONADO

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER IV, INORGANICS DEPARTMENT MANAGER –** 2001 to Present
Responsibilities	Oversee the operation of the Metals Group. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Metals Department Manager,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-2001. Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence.. **Supervisor, GFAA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1989-1992. Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.
Education	**Field Immunoassay Training Course**, *EnSys Inc*., 1995. **Winter Conference on Plasma Spectrochemistry**, *San Diego, California,* 1994. **ICP-MS Training Course**, *VG-Elemental*, 1992. **BS, Chemistry,** *Western Washington University, Bellingham, Washington,* 1988. **BA, Business Administration,** *Western Washington University, Bellingham, Washington,* 1985.

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**CLIENT SERVICES MANAGER IV** – 1998 to Present
Responsibilities	Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Personally responsible for approximately 1.5 million dollars of client work annually performing technical project management and client service. Provides technical and regulatory interpretation assistance, as well as project organization of work received by the laboratory.
	Documentation of Demonstration of Capabilities is available for review.
Experience	**Project Chemist,** *Columbia Analytical Service, Inc., Kelso, Washington,* 1992-1998. Primary responsibilities included technical project management and client service in areas of pulp & paper, marine services, mining, and DOD. Also responsible for providing technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory
	Project Chemist and Department Manager, General Chemistry Laboratory, *Columbia Analytical Services, Inc.,* 1989-1992. Responsible for management of the General Chemistry laboratory for routine wastewater, bioassay, and microbiological analyses. Also responsible for supervision of staff, data review, and reporting.
	Analyst III, *Columbia Analytical Services, Inc., Kelso, Washington*, 1989. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and TKN, in addition to miscellaneous wet chemistry analyses.
	Microbiologist/Chemist, *Coffey Laboratories, Portland, Oregon,* 1983. Coliform analysis; water chemistry.
	Laboratory Assistant, *Oregon State University, Corvallis, Oregon,* 1983. Wheat spike dissection and tissue culture.
Education	**BS, Microbiology,** *Oregon State University, Corvallis, Oregon,* 1983.

HARVEY L. JACKY

1999TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER II** – 2008 to Present
Responsibilities	Oversee the operation of the General Chemistry and Microbiology groups. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Project Manager III,** *Columbia Analytical Services, Inc., Kelso, WA*, 1999-2008. Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies. **Director of Project Management,** *Coffey Laboratories, Portland, Oregon*, 1997-1999. Responsible for technical project management. Communicated with clients to determine needs and expectations. Monitored laboratory production and ensured the timely completion of analytical projects. Technical consultant for clients regarding environmental compliance. Supervised and managed other members of the project management team. Served as a member of the senior management team for oversight of general operations, strategic planning, finances, and policy. **Project Manager/Chemist,** *Coffey Laboratories, Portland, Oregon*, 1997-1999. Served as primary liaison between Coffey Laboratories and major clients. Ensured that work was completed in a timely manner and done to client specifications. Served as technical consultant regarding environmental chemistry, soil remediation, and waste water industrial compliance. Clients included the Oregon Department of Transportation, Hazmat Unit, Portland, Oregon; Raythion Demilitarization Co., Umatilla, Oregon; Hydroblast - Wastewater Evaporator Systems, Vancouver, Washington; and Union Pacific Railroad, Northwest Region, Klamath Falls, Oregon. **Technical Sales Representative,** *Coffey Laboratories, Portland, Oregon*, 1995-1997. Responsible for marketing and sales, including actively prospecting for new potential clients. Additional responsibilities included procurement and preparation of all major project bids; ensuring that client expectations were met; and maintaining customer satisfaction. Served as consultant regarding industrial compliance issues, environmental remediation projects, and hazardous waste management. **Senior Chemist/Laboratory Chemical Hygiene Officer,** *Coffey Laboratories, Portland, Oregon*, 1988-1995. Performed analytical tests including Anions by Ion Chromatography (EPA 300.0), PAHs by HPLC (EPA 8310), Cyanides (EPA 335), and other inorganic, wet chemistry, and organic analytical tests on a wide variety of sample matrices. Responsible for the initial quality assurance review of work performed, supervised and managed personnel. Developed and implemented Laboratory Chemical Hygiene Plan. Directed personnel in regards to safety issues and hazardous waste management. Served as consultant and teacher regarding analytical methodology, environmental compliance, and industrial hygiene.
Education	**40-Hour Hazmat Certification,** *PBS Environmental,* 1996. **Industrial Emergency Response,** *SFSP Seminar,* 1991 **BS, Zoology,** *Oregon State University, Corvallis, Oregon,* 1988. **BS, General Science,** *Oregon State University, Corvallis, Oregon,* 1988. **COURSEWORK, General Studies,** *Linfield College, McMinnville, Oregon,* 1981-1982.
Publications/ Presentations	*Biochemical and Physical Factors Involved in the Application and Measurement of a Soil Bioremediation System.* Biogeochemistry, Portland State University, 1996
Affiliations	American Chemical Society, Member since 1988

JEFFERY A. GRINDSTAFF

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER III, PHARMACEUTICAL, GC/MS VOA AND SEMI-VOA LABORATORIES, –** 1997 to Present
Responsibilities	Primary responsibilities include leadership of the Pharmaceutical, GC/MS VOA and Semi-VOA staff, management of method development, training, data review, tracking department workload, scheduling analyses. Responsible for ensuring data quality and timeliness. Also responsible for project management and coordination for pharmaceutical clients. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Manager, GC/MS VOA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1997. Responsible for supervision of GC/MS VOA staff, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems. **Scientist III, GC/MS VOA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1994. Responsibilities included scheduling workload, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and customer service. **Chemist,** *Enseco-CRL, Ventura, California,* 1990-1991. Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses. **GC/MS Chemist, VOA Laboratory** *Coast-to-Coast Analytical Service, San Luis Obispo, California,* 1990-1991. Responsible for standard preparation for VOA analyses, instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants.
Education	**Sampling and Testing of Raw Materials***, PTI International, 2004*. **Leadership Training,** *Richard Rogers Group, 1996* **Mass Selective Detector Maintenance,** *Hewlett Packard Education Center, 1993* **Interpretation of Mass Spectra I,** *Hewlett-Packard Analytical Education Center,* 1992. **B.S., Chemistry**, *California Polytechnic State University, San Luis Obispo, California,* 1989. **A.A., Liberal Arts,** *Allan Hancock College, Santa Maria, California.* 1986
Publications/ Presentations	*Low Level Analysis of 1,4-Dioxane by GC/MS SIM using Large Volume Injection,* with J. Peterson and R. Holden. SETAC National Meeting Poster Session, Portland, OR 2004. *Low Level Determination of N-nitrosodimethylamine by Chemical Ionization GC/MS with Large Volume Injection*, with C. Degner and J. Peterson. SETAC National Meeting Poster Session, Portland, OR 2004. *Analysis of Polybrominated Diphenyl Ethers by GC/MS with Large Volume Injection,* J. Peterson and M.Thompson SETAC National Meeting Poster Session, Portland, Oregon, 2004. *Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater*, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, FL 1996
Affiliations	American Chemical Society. 1989

NICOLAS BLOOM

2008 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position **Scientist VII** – 2008 to Present

Responsibilities **Senior Research Scientist**

Mr. Bloom has been involved in research on the biogeochemistry of trace metals in the environment for 30 years. After graduating from the University of Washington in 1979, he entered the graduate program in the Civil Engineering Department, where he worked as a full time researcher, investigating the sorption behavior of ultra-trace concentrations of cations and anions on ferric hydroxide suspensions. In 1980, Mr. Bloom was hired by the Battelle Marine Research Laboratory to develop sampling and analytical techniques to quantify a wide range of trace metals in sea water at ambient levels and apply those methods to the biogeochemical cycling of Hg, As, Ag, Pb, Cd, and Cu in Puget Sound. In 1984 Mr. Bloom returned to graduate school at the University of Connecticut, where he developed analytical techniques to allow the speciation of Hg at the sub-picogram level by GC-CVAFS. These methods have since been applied to investigate the cycling of Hg and its various compounds in lacustrine and marine systems throughout the world.

In 1991, Mr. Bloom founded Frontier Geosciences Inc., where he continued research into ultra-low level metals speciation in sediments, air, and fossil fuels, as well as mentored the development of IC-ICP/MS and IC-HG-AFS methods for most other trace metals and for Se, As, and Cr speciation. From 2001-2005, Mr. Bloom collaborated extensively with the Universita Ca'Foscari di Venezia in a study of Hg speciation and dynamics in the Venice Lagoon. In 2004, Mr. Bloom founded Studio Geochimica LLC, continuing his studies of the biogeochemistry of trace metals in the environment and industry. In 2008, Mr. Bloom joined Columbia Analytical Services, as Director of the Trace Metals Research and Development Department. In this position, Mr. Bloom is responsible for the development and validation of new trace metals speciation methodologies as well as working with clients and staff having biogeochemical questions or particularly perplexing analytical issues.

Experience

Research Scientist, *Battelle Pacific Northwest Laboratory, Marine Sciences Lab, Sequim, WA,* 1980-1989. As an analyst, developed and validated ultra-clean sampling methods and techniques for the analysis of all 13 EPA priority trace metals in water, sediment, and tissues with detection limits below the ambient background concentrations. As a researcher, emphasized biogeochemical processes of trace metals, particularly at the air/sea and sediment/water interfaces. Supervised two technicians.

Owner/Manager/Sr. Scientist, *Studio Geochimica LLC, Seattle, WA*, 2004 - 2008. Set up the scientific agenda, marketing, sales, inventing new analytical methods, mentoring, working in the lab as scientist and analyst, etc. Staff varied from 4 to 9 people.

Owner/Manager/Sr. Scientist, *Frontier Geosciences Inc., Seattle, WA*, 1991 – 2004. Set up the scientific agenda, marketing, sales, inventing new analytical methods, mentoring, working in the lab as scientist and analyst, etc. Staff varied from 3 in 1991 to 87 people in 2003.

Education

BS, Chemistry, *University of Washington, Seattle, WA*, 1979.

MS, Chemical Oceanography, *University of Connecticut, Storrs, CT* 1986.

Publications/ Presentations

Nicolas Bloom Mr. Bloom has approximately 120 publications on the biogeochemistry and analysis of trace metals in the environment (please inquire for publication list or copies of key papers), and has over 400 presentations at conferences and symposia world-wide.

Affiliations

ASTM, ACS (past member), ASLO (past member)

LOREN E. PORTWOOD

1992 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

Technical Manager I, DRINKING WATER LABORATORY – 2008 to Present

Responsibilities

Responsible for the overall operation and supervision of the Organic Drinking Water department. Also responsible for implementation and oversight of UCMR2 analyses. Perform method development. Project management of drinking water accounts. Development of Standard Operating Procedures for Drinking Water methods. Operation of Varian GC/MS, Agilent GC/ECD and Agilent HPLC.

Documentation of Demonstration of Capabilities is available for review.

Experience

Scientist IV, Drinking Water Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Plan, conduct, and, as lead analyst, supervise analyses using advanced instrumentation such as HPLC with post column derivatization, GC/MS, and GC/ECD. Responsible for data interpretation, quality control and data reporting. Additional responsibilities include preparation of SOPs and specifications for processes and tests; handling routine and advanced maintenance and troubleshooting of instrumentation; and assisting in the training of staff department analysts. Assists the department manager and/or other senior scientists in setting up more complex procedures. Serves as senior technical advisor for teams and projects.

Technical Manager I, Petroleum Hydrocarbon Laboratory Supervisor, *Columbia Analytical Services, Inc., Kelso, Washington,* 1998-2002. Primary responsibilities include organizing and prioritizing the workload for the petroleum hydrocarbon team, initiating new methods and process improvements, and staff development and training. Other duties include department wide compliance with CAS quality assurance guidelines, routine system checks, assist and encourage staff in troubleshooting equipment and procedural problems, and lead by example in a manner that is consistent with company, state and federal guidelines. Also responsible for duties listed below under Scientist II and Scientist III.

Scientist III, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1997-1998. Duties primarily as listed below.

Scientist II, **Petroleum Hydrocarbon Laboratory,** Columbia Analytical Services, Inc., Kelso, Washington, 1996-1997. Primary responsibilities included analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons and miscellaneous FID tests. Methods of analysis include EPA methods 8100, 8310, 8315, 8330, 8040, 8015 and various state modifications of 8015 (OR, WA, CA, AK). Additional analyses include solvent scans, alcohols, glycols, and EPA methods 413.2 and 418.1. Other responsibilities include sample preparation and instrument maintenance.

Scientist I, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1993-1996. Primary responsibilities included the analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons. Methods of analysis include EPA method 8015 and various state modifications thereof (OR, WA, CA, AK). Additional responsibilities include sample preparation, instrument maintenance, and assistance with other departmental analyses, including EPA methods 413.2 & 418.1.

Bench Chemist I, Organic Extractions Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1993. Primary responsibilities included the performance of a full range of semi-volatile sample preparations for water, soil, and oil to be analyzed in the GC, GC/MS, and Petroleum Hydrocarbon Laboratories. These extraction methods included hazardous waste, wastewater, and drinking water procedures. Other responsibilities included extract cleanup via Florisil®, GPC, and Hg.

Chemist, Treclen Laboratories, Spokane, Washington, 1990-1992. Primary responsibilities included inorganic water and soil testing by EPA methods. As Chemist, I developed the testing which was accredited by the EPA, which included everything from metal digestions, to phosphates, to TSS and TDS.

Education

Comprehensive HPLC Training, *Restek,* 2002.
Purge & Trap Theory and Troubleshooting, *Full Spectrum Analytics, Inc.,* 2001.
HP5890 GC Advanced Operations, *Hewlett Packard,* 1996.
HP6890 Fast GC, *Hewlett Packard,* 1996.
Quality Training, *Roger Tunks,* 1996.
Capillary Chromatography Training, *Restek,* 1993.
HP5890 GC Maintenance and Troubleshooting, *Hewlett Packard,* 1993.
BS, Chemistry, Emphasis in Biochemistry, *Whitworth College, Spokane, Washington*, 1990.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFTEY OFFICER –** 1994 to Present
Responsibilities	Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Project Chemist, Client Services Group, Kelso Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1994. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Scientist IV, Metals Laboratory, Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington, 1987-1992.* Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Chemist,** *Dow Corning Corporation, Springfield, Oregon,* 1986-1987. Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon. **Chemist,** *Ametek, Inc., Harleysville, Pennsylvania,* 1982-1985. Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques. **Chemist,** *Janbridge, Inc., Philadelphia, Pennsylvania*, 1978-1982. Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.
Education	**BA, Chemistry,** *Immaculata College, Immaculata, Pennsylvania,* 1977.
Affiliations	American Chemical Society, Member since 1987.

APPENDIX C

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (10):			
Precisa and Mettler models	1988-2008	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autotitrator – Thermo Orion 500	2007	LM	3
Calorimeters (2):			
Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	15
Colony Counter - Quebec Darkfield	1988	LM	4
Conductivity Meters (2):			
YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5):			
COD (4)	1987, 1989	LM	5
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	5
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	7
Drying Ovens (11):			
Shel-Lab and VWR models	1988 - 2003	LM	15
Flash Point Testers (2):			
ERDCO Setaflash Tester	1991	LM	4
Petroleum Systems Services	2005	LM	4
Flow-Injection Analyzers (2):			
Bran-Leubbe	2002	LM	4
Lachat 8500	2007	LM	4
Ion Chromatographs (4)			
Dionex 2000i with Peaknet Data Systems	1988	LM	3
Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Dionex ICS-2000 with Chromchem Data System	2006	LM	3
Ion Selective Electrode Meters (5)			
Fisher Scientific Accument Model 50	1997	LM	6
Fisher Scientific Accument Model 25	1993	LM	6
Fisher Scientific Accument Model 20	2000	LM	6
Orion Model 920A	1990	LM	6
Corning pH/ion Meter Model 135	1992	LM	6
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	15
pH Meters (2):			
Fisher Scientific Accument Model 20	1993	LM	6
Fisher Scientific Accument Model AR25	2005	LM	6

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Shatter Box - GP 1000	1989	LM	5
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	7
Total Organic Carbon (TOC) Analyzers (2)			
Coulemetrics Model 5012	1997	LM	3
O-I Corporation Model 1010	2002	LM	3
Total Organic Halogen (TOX) Analyzers (3):			
Mitsubishi TOX-Sigma	1995	LM	4
Mitsubishi TOX-100 (2)	2001	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	8
UV-Visible Spectrophotometers (3):			
Hitachi 100-40 Single Beam	1986	LM	5
Beckman-Coulter DU520	2005	LM	5
Perkin Elmer Lambda 25	2008	LM	5
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991	LM	13
Water Baths/Incubators (6):			
Hach Model 15320 Incubator	1986	LM	15
Precision Model L-6 (2)	1989, 1990	LM	15
VWR 1540	1991	LM	15
Fisher 11-680-626M Incubator	1992	LM	15
Fisher Isotemp Incubator	2001	LM	15

METALS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (6)			
Mettler AE 200 analytical balance	1990	MM	12
Various Mettler, Sartorius, and Ohaus models (5)	1988	MM	12
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA Zeeman/220 AA w/Data Systems (2)	2000	LM	3
CETAC Mercury Analyzer	2000	LM	2
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	2
Atomic Fluorescence Spectrophotometer			
Brooks-Rand Model III (2)	1996, 2005	LM	3
Leeman Mercury Analyzer (1)	2006	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (2) - Labconco	1992, 2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (3)			
Thermo Jarrell Ash Model 61E	1988	LM	4
Thermo Jarrell Ash, Model IRIS	2000	MM	4
Thermo Scientific Model iCAP 6500	2007	MM	3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	2
Muffle Furnace - Thermolyne Furnatrol Model 53600 (2)	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5

SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (4)			
Mettler PM480, AE166, BB300	1999 - 2005	MM	18
OHaus EP613	2006	MM	18
Centrifuge - Sorvall Model GLC-1	1988	LM	18
Drying Ovens (2)			
Fisher Model 655G	1991	LM	18
VWR Model 1305U	1999	LM	18
Evaporators (14):			
Organomation N-Evap (7)	1989-98, 2001, 2006	LM	18
Organomation S-Evap (7)	1989-1991, 2006	LM	18
Extractor Heaters: Lab-Line Multi-Unit Models for Continuous Liquid-Liquid and Soxhlet Extractions (102)	1987-1992, 2007	LM	12
Extractors (52):			
Branson Model 450 Sonifier (2)	1991	LM	6
Tekmar Sonicator	1994	LM	6
Fisher Scientific Sonicator	1994	LM	6
Soxhtherm (48)	2000, 2008	LM	8
Extractors, TCLP (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	2
TCLP Extractor - Tumbler (12 position)	1989	LM	2
Gel Permeation Chromatography (GPC) (5)			
ABC single column (3)	1998, 1999, 2007	LM	4
ABC Autoprep 1000	1995	LM	4
J2 Scientific	2005	LM	4
Muffle Furnace - 4	1994-2006	LM	4
Solid Phase Extractors (8) – Horizon SPE-Dex 4790	2003, 2006	LM	4
Ultrasonic Water Bath – VWR 550D	2007	LM	18
Vacuum Pump – Edwards	1992	LM	8

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler AT 250	1989	MM	7
Chromatography Data Systems (12)			7
HP Enviroquant (8)	1994-2002	LM	
Thruput Target (4)	1998-2000	LM	
Gas Chromatographs (11):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (4)	1990 – 1995	LM	7
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (5)	2001, 2005, 2007	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	LM	7
Agilent 7890A Dual ECD Detectors Agilent 7683B autosampler	2008	LM	7

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	5
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	5
Semivolatile GC/MS Systems (9):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	5
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	5
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler	1994	LM	5
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	LM	5
Agilent 6890/5973 with ATAS Optic3 LVI and 7683 Autosampler	2004	LM	
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler	2007	LM	4
Semivolatile GC/MS/MS – Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	1

PETROLEUM HYDROCARBONS GC/HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB240	1994	MM	6
Aspirator pump – GAST	2004	LM	6
Drying Oven - Fisher Model 630F	1991	LM	6
Evaporator - Organomation N-Evap	1990	LM	6
HP Enviroquant Chromatography Data Systems (8)	1994-2002	LM	6
Gas Chromatographs (6):			
Hewlett-Packard 5890 Series II with PID/PID/FID(2)	1991	LM	4
EST-ENCON Purge and Trap Concentrator	1991	LM	4
Dynatech Archon 5100 Autosampler	1992	LM	4
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector	1995	LM	4
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (3)	2001, 2005	LM	4
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	4
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	4
High-Performance Liquid Chromatograph/Mass(2) Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS and Autosampler	2005	MM	2
API 5000 LC/MS/MS and SIL-20AC Autosampler	2008	MM	2

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	5
Drying Ovens (2):			
Narco 420	1989	LM	5
VWR 1305 U	1991	LM	5
Sonic Water Bath - Branson Model 2200	1989	LM	5
Volatile GC/MS Systems (7):			
Agilent 5890/5970	1989	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 5890/5971	1991	LM	5
Tekmar 3000 Purge and Trap Concentrator	2001	LM	5
Dynatech ARCHON 5100 Autosampler	1995	LM	5
Agilent 5890/5972A	1993	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 6890/5973	2001	LM	5
Tekmar 3100 Purge and Trap Concentrator	2001	LM	5
Varian Archon Autosampler	2001	LM	5
Agilent 6890/5973	2005	LM	5
Tekmar Velocity Purge and Trap Concentrator	2005	LM	5
Tekmar Aquatech Autosampler	2005	LM	5
Agilent 6890/5973 (2)	2007	LM	5
Tekmar 3000 Purge and Trap Concentrator	2007	LM	5
Varian Archon 5100 Autosampler	2007	LM	5

DRINKING WATER ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB300	1991	MM	2
Extractors (10) – Horizon SPE-DEX Solid Phase Extractor	2003/2008	LM	2
Aglinet Enviroquant Chromatography Data Systems (2)	2003	LM	2
Varian Saturn Chromatography Data System	2003	LM	2
Evaporator - Organomation N-Evap	2003	LM	2
Agilent 1100 HPLC w/post-column derivitization: UV/Fluoescence detectors Pickering PCX-5200 Post-column derivitization unit	2003 2003 2003	LM LM LM	2 2 2
Agilent 6890N GC/Dual ECD system w/ autosamplers Agilent 7890 GC/Dual ECD w/autosamplers	2003 2008	LM LM	2 2
Varian Ion trap GC/MS: Varian 3800 GC w/CP8400 autosampler Varian Saturn 2100T mass spectrometer	2003 2006 2003	LM LM LM	2 2 2
Thremo Ion Trap GC/MS w/TriPlus autosampler	2008	LM	2

Metals Method Development Laboratory			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Perkin-Elmer ICP/MS Elan 9000 w/ Perkin-Elmer AS-93+ Autosampler	2008	LM	2
Perkin-Elmer Series 200 IC	2008	LM	2
Brooks Rand III Atomic Fluoresence Spectrophotometer - 2	2008	LM	2
Oriel Atomic Fluoresence Spectrophotometer – Lab Designed	2008	LM	2
Balances - 4	2008	LM	2
Ovens - 2	2008	LM	2
Buck AA Spectrophotometer Model 205	2008	LM	2
Forma Scientific Bio Freezer	2008	LM	2
Digital Shaker SK-71	2008	LM	2

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMS Sample Manager using Oracle 10g DBMS running on Redhat Advanced Server 3.0 (Linux) platform connected/linked on a frame relay WAN environment	1994-2004	LM	NA
1 - Network Server Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 Advanced Server, 1 for Applications running Windows 2003 Advanced Server. Data acquisition capacity at 65GB with redundant tape and disk arrays.	2004	LM	NA
Approximately 50+ HP and Dell Laserjet printers (various types including models III, 4, 5, 8150, 4000, 4050, 4250, 8150, 1720dn, W5300)	1991 - 2007	LM	NA
Approximately 180 Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2004	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97.	1996 - 2004	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Outlook Web Access interface. Microsoft Outlook 2003.	1994 - 2006	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Facsimile Machines - Brother 4750e (2); Brother SuperG3 (1); Canon CFX-L4000 (1)	1991 - 2007	LM	NA
Copiers/Scanners: Konica BizHub 420 (1), BizHub 600 (1), BizHub 920 (2), BizHub Pro 1050 (3). The 920s and 1050s are accessible via LAN for network scanning.	2000 - 2007	LM	NA
Dot Matrix Epson FX-880, LQ-1050, LX-300	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, StarLIMS reporting software systems.	1998 - 2004	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures	Daily
	Clean coils	Annually
	Check coolant	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured	Quarterly
	Sash operation	As needed
	Change filters	Annually
	Inspect fan belts	Annually
Ovens	Clean	As needed or if temperature outside lim.
	Record temperatures	Daily, when in use
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures	Daily, morning and evening
	Wash with disinfectant solution	When water is murky, dirty, or growth appears
Autoclave	Check sterility	Every month
	Check temperature	Every month
	Clean	When mold or growth appears
Analytical Balances	Check alignment	Before every use
	Check calibration	Daily
	Clean pans and compartment	After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride ISE	Store in storage solution	Between uses
Ammonia ISE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero	Weekly
	Check digestion/condensation vessels	Each use
	Clean digestion chamber	Every 2000 hours, or as needed
	Clean permeation tube	Every 2000 hours, or as needed
	Clean six-port valves	Every 200 - 2000 hours, or as needed
	Clean sample pump	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	Every 200 - 2000 hours, or as needed
	Clean IR cell	Every 2000 - 4000 hours, or as needed

Instrument	Activity	Frequency
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed
Flow Injection Analyzer	Check valve flares	Each use
	Check valve ports	Each use
	Check pump tubing	Each use
	Check light counts	Each use
	Check flow cell flares	Quarterly
	Change bulb	As needed
	Check manifold tubing	Each use
	Check T's and connectors	Each use
Ion Chromatographs	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro-photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency

Instrument	Activity	Frequency
ICP - MS	Check argon dewar	Daily
	Check water level in chiller	Daily
	Complete instrument log	Daily
	Replace peristaltic pump tubing	Daily
	Clean sample and skimmer cones	As needed
	Clean RF contact strip	As needed
	Inspect nebulizer, spray chamber, and torch	Clean as needed
	Clean lens stack/extraction lens	As needed
	Check rotary pump oil	Monthly
	Change rotary pump oil	Every six months
Gel-Permeation Chromatographs	Clean and repack column	As needed
	Backflush valves	As needed
High Pressure Liquid Chromatographs	Backflush guard column	As needed
	Backflush column	As needed
	Change guard column	As needed when back pressure too high
	Change column	Annually or as needed
	Change in-line filters	As needed
	Leak check	After column maintenance
	Change pump seals	As needed
	Change pump diaphragm	Annually
	Clean flow cell	As needed
	Fluorescence detector check	Daily
	Diode array absorbance check	Daily
Gas Chromatographs, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean FID	Weekly or as needed
	Clean ECD	Quarterly or as needed
	Leak test ECD	Annually

Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Annually or as needed
	Change septum	Daily, when in use
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications
Purge and Trap Concentrators	Change trap	Every four months or as needed
	Change transfer lines	Every six months or as needed
	Clean purge vessel	Daily
Gas Chromatographs, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean PID lamp	As needed
	Clean FID	As needed
	Change ion exchange resin	Every 60 days
	Replace nickel tubing	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Annually or as needed
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications

APPENDIX E

CORPORATE POLICY STATEMENTS

Columbia Analytical Services™

Policy for Data Review and Validation

May 2009
Effective July 1, 2009

The purpose of this policy is to identify the requirements for performing data review and validation prior to releasing data and reports to customers of Columbia Analytical Services. It is a requirement of NELAC (TNI) quality system standards and Department of Defense (DoD) agencies to have data review procedures established.

This policy is applicable to the review of raw and reported data generated in all laboratories. Specific data review and validation processes or logistics may vary somewhat from facility to facility, or vary for data generated using different methodologies however; the policies described here are to be followed. The documentation practices should be consistent within the facility. Automated validation processes are encouraged, but must be sufficiently described in an SOP.

In general, the data review and validation practices used at each facility will meet the requirements of NELAP quality system standards, the DoD Quality System Manual (QSM), and ISO 17025. Specific data review and validation policies are as follows:

1. Each laboratory facility will have a written and approved standard operating procedure (SOP) for conducting data review/validation that meets the standard CAS requirements for administrative SOPs. The SOP will list details of data review practices for the facility. The SOP will also give a detailed explanation of the review documentation procedures for each type of data.

2. Data review will be performed by qualified personnel who have documented training on either the analysis itself or training specific to the data review SOP. Personnel preparing reports who may do some level of clerical review or proofreading do not need technical knowledge of the test, but must be knowledgeable of reporting systems and requirements.

3. All data will be reviewed by a minimum of two persons. Data generated or reported by one person may not be released without another person's review.

4. However defined, one review (typically a "primary" technical review) must focus on the validity of the analysis and raw data generated, the technical accuracy and correctness of the analysis (the analytical procedure is in control), use of valid and approved procedures and methods, and interpretation of sample results.

5. The secondary review will be performed by someone other than the technical reviewer. The secondary review will make the same assessments as the primary reviewer, and check the interpretations, data manipulations, and decisions made by the primary reviewer. Additionally, the secondary reviewer will review the outputs from the initial review to the raw data. This includes such things as data processing results/outputs, calculations, runlogs, bench sheets, QC analyses, etc. The secondary review verifies the completeness and validity of the data to be reported.

6. All client-ready final reports will be reviewed in the format, and as presented to, the client; either by analysis fraction or in their entirety. This review will include verification of the accurate and correct reporting of sample and QC results; including accurate translation of results from data to report forms, report format, use of qualifiers and flags, and method citations. This review will also include verification of the correct project information; such as client name, project name, sample I.D.s, etc. The report review should ensure that the report is error-free and contains no inconsistencies. For upper tier deliverables, this review will verify that all deliverables are included in the report package.

7. The Project Manager will review all complete reports prior to signing the report and submitting to the client. The review of the reported data will focus on the following items:
    a. Consistency with client, contract, and/or project specifications.
    b. Acceptability of any data qualifiers or footnotes.
    c. Accuracy and completeness of explanations or discussion in the report cover letter or case narrative.
    d. As needed depending on the scope of testing, an additional level of technical review of all data generated.
    e. A general overview of the completed service request file with respect to overall reasonableness, and if available, with historical project information.

8. Data review must be documented. Persons performing data and report review must sign (or initial) and date the applicable data reviewed. Checklists or review summaries should be used for guidance and documentation. Documentation processes must be described in the laboratory SOP.

Lee Wolf, Corporate Director of Quality Assurance — Date: 5-5-09

Steve Vincent, President — Date: 5-5-09

Columbia Analytical Services™

Policy for Conducting Research, Method Development, and Method Investigations

December 2009

Columbia Analytical Services (CAS) often develops test procedures internally by conducting research and development or method development based on published procedures. This type of testing may not fall under common laboratory regulations which describe benchmarks, or minimum requirements, for procedure development and implementation. Also, it may be necessary at certain times to conduct investigations into the quality of existing methods. Therefore, a policy is necessary to identify and establish those minimum requirements.

The purpose of this Policy is to identify the CAS requirements for performing internal research and subsequent method development, performing method development from published references, and performing investigations into method performance.

For the purpose of this policy, the following Definitions are provided:

> *Research and development (R&D)* – The practice of independently evaluating analytical options and procedures and applying them to a sample analysis challenge; resulting in an internally developed analysis method. For this policy, R&D is limited to that performed by CAS personnel.
>
> *Method development* – The practice of implementing a CAS analysis procedure based on published references.
>
> *Method investigation* – For the purpose of this policy, this is defined as the evaluation of major changes in methodology outside the scope of published methods or SOPs. This is generally done to improve method performance or troubleshoot a significant analytical problem; and done outside of the routine maintenance, troubleshooting, and nonconformance/corrective action process.

The intent of this policy is to ensure that CAS R&D, method development, and method investigations are performed in an unbiased manner, ensure data integrity, use common scientific practices; and ensure that these activities are peer reviewed.

General Provisions

- When conducting any of the activities covered by this policy, employees will follow standard CAS procedures for maintaining documentation and analysis records.
- Initial and final review of statements, plans, and summaries will be done by two persons; the applicable Technical Director (TD) and the Laboratory Director (LD). If the TD is the LD, then a Peer will conduct the second review.
- Once development is concluded, the adoption of Standard Operating Procedures (SOPs), conducting personnel training, etc., will be done following routine CAS QA protocols.

Research and Development

When conducting research on new analyses and developing in-house procedures not based on reference methods or published methods, the research and development effort will include the following components:

1) There will be a written *Development Statement* detailing the intent of the research and development effort. This will state the purpose of the work, the resources and references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the statement:
   a) Equipment to be used.
   b) Quality Control measures to be incorporated into the analysis.
   c) Method Performance (validation) measures to be taken and expectations.
2) There will be an initial internal review and acceptance of the statement by the Technical Director and the Laboratory Director.
3) The person leading a R&D effort will gather information, references, and resources as described in the Development Statement and document those resources.
4) The experimentation will be performed and documented.
5) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
6) The developer will draw conclusions, and if successful, summarize the results in a brief R&D summary.
7) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
8) Following approval, an SOP will be written for subsequent implementation.

Method Development

When developing and implementing new methods based on reference or published methods, the method development effort will include the following components:

Non-certified (nor certifiable) methods

1) There will be a written *Development Statement* detailing the method development effort. This will state the purpose of the work, the reference method, references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the statement:
   a) The reference method being implemented and the application(s).
   b) Equipment to be used.
   c) Quality Control measures to be incorporated into the analysis.
   d) Method Performance (validation) measures to be taken and expectations.
   e) Modifications to the reference method.
2) There will be an initial internal review and acceptance of the statement by the applicable Technical Director and the Laboratory Director.

3) The experimentation will be performed and documented.
4) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
5) The developer will draw conclusions, and if successful, summarize the results in a brief method development summary.
6) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
7) Following approval, an SOP will be written for subsequent implementation on the stated applications.

Certified (certifiable) methods

1) There will be a written *Experimental Plan* detailing the method development effort. This will state the method being implemented, references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the Plan:
   a) The reference method being implemented.
   b) Equipment to be used.
   c) Quality Control measures to be incorporated into the analysis.
   d) Method Performance (validation) measures to be taken and expectations. This will include method and certification requirements for accuracy and precision, sensitivity, selectivity, calibration/linear range, etc. For methods where NELAC accreditation is being pursued, the requirements of the NELAC Standard (2003 Standard, Quality Systems section 5, Appendix C.3) will be met.
   e) Modifications to the reference method.
2) There will be an initial internal review and acceptance of the Plan by the applicable Technical Director and the Laboratory Director.
3) The method will be set up and run following the procedural steps of the method and the Plan; and will be documented.
4) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
5) The developer will draw conclusions, and if the results meet the method performance criteria in the method and/or Experimental Plan, the results will be summarized in a brief method development summary.
6) The summary report will include a documented approval the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
7) Following approval, an SOP will be written for subsequent implementation.

Method Investigations

1) There will be a written *Investigation Statement* detailing the method investigation effort. This will state the purpose of the investigation, the CAS procedure, the targeted problem, the experimentation that will be performed, and the desired improvement result. The following items will be included in the statement:
   a) The CAS procedure being investigated and the equipment used.
   b) A brief discussion of the problem, the solutions being investigated, and the impact on method compliance and data quality.
   c) The experimentation used to perform the investigation.
   d) The Method Performance (validation) measures that will be taken to re-establish conformity to QA/QC criteria.
2) There will be an initial internal review and acceptance of the statement by the applicable Technical Director and the Laboratory Director.
3) Once data is collected, it will be interpreted objectively using the assessments applicable to that analysis and CAS SOP.
4) The investigator will draw conclusions, and if the results meet the method performance criteria in the method and SOP, the results will be summarized in a brief method investigation summary.
5) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
6) Following approval, the CAS SOP will be revised to implement the changes to procedure.

Documentation

The developer or investigator will generate the written Development or Investigation statements, or Experimental Plan, and provide them for initial review prior to beginning experimentation and data collection. The initial review and acceptance of the Statement will be documented. The laboratory QA PM will keep this documentation on file.

The developer or investigator will generate the written summary report and validation package, and will submit supporting data for review. The approval of the development or investigation (and SOP changes) will be documented and the laboratory QA PM will keep this documentation on file.

Steve Vincent

Steve Vincent, President/CEO

12-15-09

Date

Lee Wolf

Lee Wolf, Chief Quality/Ethics Officer

12-15-09

Date

Columbia Analytical Services™

Policy for Standards and Reagents Expiration Dates

September 2009
Effective September 28, 2009

The purpose of this policy is to state the standardized requirements for assigning expiration dates to standards and reagents used in the laboratories of Columbia Analytical Services. It is a requirement of NELAP Quality System standards, the DoD Quality System Manual (QSM), and ISO 17025 to have written protocols to ensure the use of standards and reagents of appropriate quality. Additionally, documentation of the expiration date of reagents and standards is required. This policy is intended to meet the requirements of NELAC, DOD, and ISO 17025.

This policy is applicable to all purchased and prepared standards and reagents used by the laboratory to generate reported data. This includes raw (neat) materials, stock, intermediate, working, and calibration standards and/or reagents. This does not include solvents and acids.

In general, the expiration date is the date after which a standard or reagent shall not be used. It is either the date assigned by the manufacturer, the date (duration) specified by the applicable reference method, or it is a date assigned by the laboratory under this policy.

General Policies:

1. All standard and reagent expiration dates/periods shall be listed in the applicable laboratory SOP.

2. When establishing an expiration date, the following hierarchy will be used:

   - If the cited analytical method specifies the expiration date/period, that date shall be used.
   - If the cited analytical method does not specify the expiration date/period, then the date assigned by the manufacturer will be used.
   - If the cited analytical method does not specify the expiration date/period, and an expiration date is not assigned by the manufacturer, then the laboratory will assign the expiration date according to the CAS Standardized Expiration Dates tables below.

CAS Expiration Dates for Reagents	
Chemical	Expiration Date
Purchased neat reagents	5 years after receipt
Inorganic reagent solutions	1 year from preparation or receipt
Organic reagent solutions	6 months from preparation or receipt

CAS Expiration Dates for Standards	
Chemical	Expiration Date
Purchased neat standards	5 years after receipt
Inorganic stock standard solutions	1 year from preparation or receipt
Inorganic secondary, intermediate, or working standard solutions	6 months from preparation or receipt
Purchased semivolatile organic stock standard solutions	1 year from receipt
Prepared semivolatile organics stock standards	1 year from preparation
Semivolatile organic secondary, intermediate, or working standard solutions	6 months from preparation or receipt
Purchased volatile organics stock standards – **unopened** ampules	1 year from receipt
Purchased volatile organics stock standards – **opened** ampules	≤2000 mg/L 1 month after opening >2000 mg/L 3 months after opening
Prepared volatile organics stock standards	1 year from preparation
All volatile organics secondary, intermediate, or working standards* * note: common 'gases' standards and standards used for calibration should not be older than 7 days	≤20 mg/L 7 day expiration date >20 and ≤200 mg/L 1 month expiration date >200 mg/L 3 month expiration date
Dioxin/Furan and PCB stock standards	5 years from receipt
Dioxin/Furan and PCB working standards	1 year from preparation or receipt
Derivatized (prepared) semivolatile organics standard solutions	1 year from date of derivatization

3. The expiration date of a prepared reagent or standard cannot exceed the expiration date of the starting material, with the exception of standards prepared via in-lab derivatization to yield a different compound. The expiration date of a reagent or standard cannot be extended by preparing a dilution of it. For example, a purchased standard has an expiration date of July 15, 2009. A standard prepared on February 20, 2009 from this purchased standard would ordinarily have an expiration date of six months (namely, 8/20/2009), but since the purchased standard expires before six months, the prepared standard would be assigned an expiration date of July 15, 2009.

4. A multicomponent prepared reagent or standard will be assigned an expiration date not to exceed the expiration date of any of the components' expiration date. For example, a prepared standard is made from purchased standard A (with an expiration date of August 5, 2009) and from purchased standard B (with an expiration date of December 15, 2009). Consequently, the prepared standard will have an expiration date of August 5, 2009.

5. The stability and concentration of the reagent or standard are to be taken into account when assigning the expiration date. Certain solutions, depending on use and storage, may have shorter usable life time than defined by the method, manufacturer, or this policy; and should be assigned expiration dates accordingly. Reagents and standards must be stored under conditions specified by the test method and outlined in the analytical SOP.

6. Expiration dates can be extended under the following conditions:
   - A new, replacement reagent or standard is not readily available from vendors and,
   - The cited analytical method does not specify the expiration date/period and,
   - The material has been stored under conditions specified by the analysis method and outlined in the analytical SOP and,
   - The material is not reactive, volatile, or prone to degradation under the specified storage conditions and,
   - The suitability of the material is verified by the laboratory as follows, under the same valid analysis conditions used for sample analysis, and meet the following criteria:

     a. For reagents:
        i. Perform a blank and LCS pair of analysis three times using three different subaliquots of the reagent.
        ii. Each LCS result must be within the specified control limits for the test.
        iii. The %RSD for the three LCS's must be <10%.
        iv. Each blank result must be < 1/2MRL for every compound to be reported from subsequent analysis.
     b. For standards:
        i. Analyze three separate dilutions of the standard at a concentration near the midpoint of the calibration range. (Note that standards below this concentration cannot be re-verified).
        ii. The average result must be within ± 5% of the original true value.
        iii. The %RSD for the three results must be <10%.

     If these conditions and criteria are met and documented, the material may be assigned a new expiration period the same as newly prepared material.

Lee Wolf, Corporate Director of Quality Assurance — Date: 9-10-09

Steve Vincent, President — Date: 9-15-09

Policy for the Use of Accreditation Organization Names, Symbols, and Logos

September 2009

Effective October 1, 2009

The purpose of this policy is to state Columbia Analytical Services' (CAS) requirements and restrictions for the company use of the name, symbols, and logos of accreditation organizations. In general, the names, symbols, and logos used by these organizations are the property of the organization. Therefore, it is a policy that CAS will comply with the requirements and policies of the organizations that accredit our laboratories.

The NELAC Institute (TNI): The TNI Board of Directors approves and oversees the use of TNI logos and marks (TNI, NELAC, NELAP) by programs, members, and other entities. In consideration that CAS is a member of TNI, CAS will abide by the following TNI policy and be subject to the TNI Consequences of Misuse.

All persons and entities that use or reproduce TNI logos and marks:

1. *Shall restrict access to them by unauthorized parties.*
2. *Shall use them only for purposes and activities authorized by the TNI Board of Directors.*[a]
3. *Shall endeavor to avoid statements in relation to their use that the TNI Board of Directors may consider misleading or unauthorized.*
4. *May not imply endorsement or approval by TNI in communication media such as the Internet, documents, brochures, or advertising without the expressed consent of the TNI Board of Directors.*
5. *May not imply an association or partnership with TNI when such an arrangement has not been authorized by the TNI Board of Directors.*

[a] Authorized uses and activities are listed in the 2003 NELAC Standard, Section 6.8

American Association for Laboratory Accreditation (A2LA): CAS will comply with A2LA policy *P101 – Reference to A2LA Accredited Status – A2LA Advertising Policy*[b].

- CAS will only use the A2LA logo and symbol/phrase "A2LA Accredited" at individual CAS laboratory locations which have demonstrated to be in compliance with A2LA quality system requirements for the applicable A2LA accreditation program (e.g. Testing Laboratory).
- The "A2LA Accredited" symbol will not be used by a CAS laboratory that is not A2LA accredited and the symbol will not be used by a CAS laboratory that has only applied for accreditation.

- When promoting A2LA accreditation, CAS will follow the requirements of the A2LA policy.
- Where the "A2LA Accredited" symbol is used to endorse results on reports, it will always be accompanied by the A2LA certificate number and an indication of the type of laboratory (i.e., testing laboratory).

[b] The A2LA policy can be found at http://www.a2la.org/policies/A2LA_P101.pdf

International Organization for Standardization (ISO): ISO does not perform assessments and therefore is not a certification or accreditation organization. ISO is a standards development organization and compliance with an ISO standard does not imply ISO endorsement. ISO's statement on the use of the name and logo is listed below, and can be found at the following URL: http://www.iso.org/iso/support/name_and_logo.htm ISO has also provided a guide for how to publicize certification to an ISO standard: http://www.iso.org/iso/publicizing2005-en.pdf

Use of ISO's name®

Within the context of international standardization or related activities (such as consultancy, training or conformity assessment including certification) "ISO" (or "iso") is the short name of the International Organization for Standardization. The name is registered within this context as the sole property of ISO and the Organization will protect its name on behalf of all ISO's members - the national standards institutes of some 150 countries. In particular, ISO will not authorize the use of the name "ISO" (or "iso") by any organization other than its members in Internet domain names, names of Web sites, trademarks, companies / organizations, products, etc. Such use could mislead third parties into believing that the domain name / Web site / trademark / company / organization / product concerned represents ISO, or has been approved or authorized to act on behalf of ISO or belongs to ISO.

Therefore, ISO will take whatever actions it considers necessary to prevent the misuse of its name.

Use of ISO's logo®

The ISO logo is a registered trademark. Unless authorized by ISO, use of its logo is prohibited. Notably, ISO will not allow its logo to be used in connection with conformity assessment activities. These include the certification of management systems, products, services, materials or personnel, even when these certifications attest conformity to an ISO standard, such as one of the ISO 9000 or ISO 14000 series. Examples of unacceptable use of the ISO logo would include use on products, in publications, on Internet sites, in marketing materials, advertisements and company letterheads.

Allowing the ISO logo to be used would give the false impression that ISO carries out certification activities, or has approved or authorized the organization using its logo. These activities are not business functions of ISO.

ISO is not an auditor, assessor, registrar, or certifier of management systems, products, services, materials or personnel, nor does ISO endorse any such activities performed by other parties. ISO develops International Standards but does not operate any schemes for assessing conformance with them.

Therefore, ISO will take whatever actions it considers necessary to prevent the misuse of its logo.

The organizations specifically discussed in this policy do not comprise a complete list of organizations to which the policy applies. It is reiterated that, with regards to the use of names, symbols, and logos; it is a policy that CAS will comply with the policies of the organizations that accredit our laboratories.

Lee Wolf, Corporate Director of Quality Assurance — Date: 9-21-09

Steve Vincent, President — Date: 9-21-09

Columbia Analytical Services™

Policy for Internal Quality Assurance Audits

May 2009
Effective July 1, 2009

The purpose of this policy is to identify the requirements for performing internal systems audits and data audits in the laboratories of Columbia Analytical Services. Internal audits are necessary to ensure that laboratory operations work within the quality systems and that these systems yield data of high quality. Internal audits are also necessary in order to meet certification and accreditation requirements. The internal auditing practices used at each facility will meet the requirements of NELAC quality system standards, the Department of Defense (DoD) Quality System Manual (QSM), and ISO 17025.

For systems audits, the concept of this policy is that corporate quality assurance audits will evaluate the laboratory QA systems and operation horizontally, or as an overall 'umbrella' assessment, whereas local QA audits will be 'drill down' audits focused on technical correctness and data validity. It is practical to verify related systems implementation as these audits are conducted.

For electronic data auditing, the concept is to assess critical data from high liability steps of procedures, from all applicable instruments, in a frequent manner (quarterly) so as to identify any potential problems relatively quickly. This is in contrast to performing 100% data assessment from a subset of instruments quarterly and taking a long period of time to assess all instruments.

Definitions

- System audits are audits used to evaluate quality system implementation, policies, procedures, laboratory practices, and testing activities of the laboratory.

- Data audits are used to assess reported laboratory data. This includes all data used to generate the reported results and the final report itself. These are performed as 'desk audits' of reported data packages, and supporting data if not included in the reported data.

- Electronic data audits are used to assess laboratory data that is processed, interpreted, used by the analyst in electronic format. This is generally limited to electronic chromatographic data.

    - "Critical" and "high liability" data – Data related to the tuning, calibration, calibration verification, and QC analyses for an analysis; as well as data vulnerable to improper manipulation (improper processing/reprocessing of files, clock changes, poor interpretation of control data, peak integrations, etc., as described in CAS Ethics policies).

Specific internal auditing policies are as follows:

1. A comprehensive internal audit will be conducted annually (approximately every 12 months) at each laboratory. The audit will address all elements of the quality system and will include environmental testing activities, and used to meet the annual internal audit requirements of NELAC, DoD, and ISO 17025. In general, the comprehensive audit will be conducted and lead by the Quality Assurance Director (QAD), with assistance from the laboratory Quality Assurance Program Manager (QA PM).

   The laboratory QA PM will not be required to conduct an additional comprehensive audit. While performing the system and data audits described below, the QA PM will verify ongoing implementation of many QA systems.

2. Each laboratory QA PM will conduct three technical systems audits per calendar quarter. These audits will be technically-focused audits of three different test procedures and technologies.
   a. The three procedures will be varied throughout the year such that analytical disciplines (e.g. digestion, extraction, ICP, ICP/MS, titrimetric, colorimetric, GC, GC/MS, HPLC, microbiology, etc) from all sections of the laboratory are assessed in a year (for laboratories with fewer than 12 tests performed, the same tests will be audited more than once).
   b. The audits will assess SOP and method compliance.
   c. The audits will assess the use of sound analytical techniques and practices.
   d. The audits will assess the analyst(s) training and documentation of /proficiency.
   e. The audit will assess all aspects of the test being evaluated, including sample handling/preparation, calibration, sample batching/run sequences, standards, quality control, instrument operation/maintenance, data interpretation, data review/reporting, and applicable quality assurance.

3. Each laboratory will conduct two complete hardcopy data audits per quarter. These audits will focus on data validity, accuracy, and completeness. Data audits will be performed on hardcopy raw and reported data (or electronic version of) and on a 'Service Request basis'.
   a. The audits will be performed on data generated no earlier than three months prior to the audit.
   b. Service requests are to be chosen at random to encompass various analytical disciplines of the laboratory over the course of a year.
   c. The audit will assess the validity of the laboratory procedures used to generate the results reported, from sample receipt to analysis to data reporting, and the accuracy and completeness of the final report.
   d. The audit may be used as a convenient way to assess training documentation for the analysts who performed the analyses.

4. DoD report reviews will be conducted quarterly at the frequency required by the DoD QSM.

5. Electronic data auditing

    a. Each laboratory will conduct random screening of chromatographic data using Mint Miner software (where analytical software is compatible) every quarter on every instrument on data generated that quarter.
    b. Mint Miner software will be adequately configured in order to make screening effective.
    c. Using the screening results, data files will be selected for auditing from each instrument each quarter. Two sequences will be audited, one an initial calibration and one a typical sample analysis sequence. Test methods are to be chosen at random to encompass various methods performed.
    d. The audits will focus on calibration and QC data, including the evaluation of proper processing of files, interpretation of data, peak integrations, and comparison of raw electronic data to 'interpreted' and approved data.
    e. If screening results indicate significant potential problems, additional files should be inspected. The QA PM will conduct these added audits as needed.
    f. If Mint Miner software is not compatible with instrument software, auditing will be performed manually by the QA PM by auditing the data from two sequences per quarter, including one initial calibration sequence, per instrument.

6. As with any audit, additional auditing and investigation may be necessary based on the audits performed and magnitude of findings.

7. Each laboratory facility will have a written and approved standard operating procedure (SOP) for conducting their internal audits. The SOP will include detailed procedures for technical system audits, data audits, and electronic data audits as defined in this policy. In addition to meeting the standard CAS requirements for administrative SOPs, the SOP will include details of the audit processes, use of checklists, documentation, audit reporting, corrective action, and resolution of audit findings.

Lee Wolf, Corporate Director of Quality Assurance — Date: 5-5-09

Steve Vincent, President — Date: 5-5-09

Columbia Analytical Services

CAS Quality and Ethics Policy Statement

March 2009

Columbia Analytical Services (CAS) vision is simple. We strive to be the best in everything we do. This includes ethics and professional practice where CAS is committed to the highest standards of ethical behavior and quality of its analytical testing.

Unethical behavior carries a heavy price - one that we do not want to bear. This includes loss of reputation, loss of business, civil and criminal penalties, and government and customer sanctions.

CAS is committed to excellence and superior performance in everything we do. We will not sacrifice our ethical principles in order to achieve business success. This means we will always strive to conduct business honestly and with integrity. We will always follow and obey the law of the land in which we are operating our business. We will always follow, to the best of our ability, standard operating procedures, rules and regulations that apply to our industry and specifically to our laboratory operations. Our customers, employees, suppliers and communities that we serve expect and deserve nothing less than the highest standards of conduct and compliance.

The following are the critical elements of the Quality and Ethics program at CAS.

- The Executive Management and Board of Directors of CAS sponsor and support the Quality and Ethics program through their personal commitment and by providing the necessary resources to promote this program throughout the organization.
- Chief Quality and Ethics Officer. The position is responsible for the quality and ethics program, ensures that appropriate resources are provided, reviews and recommends changes in the program, and resolves ethical and quality issues brought to management attention. This Officer reports directly to the Board of Directors Audit Committee on quality and ethics.
- Core Values. The CAS Statement of Core Values was developed internally with input from the entire company. We are committed to ensuring the integrity and quality of data, and meeting the needs of our clients, while conducting business with high ethical standards. We hold strong to the core values of Honor, Truth, and Fairness. We are committed to these values and rely on them when confronted by difficult choices.
- Ethical Code of Conduct. As a member of the American Council of Independent Laboratories (ACIL) and part of the laboratory industry, CAS subscribes to and supports the core values and ethical codes established by this industry organization.

- CAS Code of Conduct. CAS requires its employees to be introduced to and to sign the "CAS Commitment to Excellence in Data Quality" statement and to comply with standards outlined in Section 6, Employee Conduct, of our Employee Handbook. All personnel concerned with analytical testing activities within the laboratory are required to acquaint themselves with the quality documentation and to implement these policies and procedures in their work.
- Open Door Policy. Employees have the right and obligation for open communications to ask questions, seek guidance, and report incorrect practices and wrong doing without fear of retribution. As described in the CAS Open Door Policy; CAS believes in using the chain-of-command channels for this dialogue. However, if there is fear or a concern that using this approach is not appropriate, employees are free to take their concerns to the President, the Director of Human Resources, the Chief Administrative Officer, the Chief Quality Officer, or the company Ombudsman. Employees may do so without fear of retribution.
- Ombudsman Program. CAS has implemented an external ombudsman/hotline program through EthicsPoint, a phone and internet-based reporting system, to enhance communication and empower employees to promote safety, security, and ethical behavior. Employees can file a report anonymously to address issues in the workplace and to cultivate a positive work environment.
- Internal Audits. Policies are established to ensure that internal systems and data audits are conducted periodically in addition to external agency and client audits. The data audits include a detailed in-depth review of hardcopy data and electronic data to ensure compliance with the CAS Quality program and on-going data integrity.
- NELAP Accreditation. CAS management is committed to compliance with the NELAP standards. CAS maintains NELAP accreditation and as such includes quality systems documented in QA Manuals, documented procedures in Standard Operating Procedures (SOPS) and policies, and documented training for demonstration of capabilities.
- Ethics Training. CAS has the obligation to provide training to its employees with respect to company policies concerning business conduct. This includes introductory training on this, and related policies, at the time of hire; in-depth "core" training within one year of hire, and on-going refresher training on a semi-annual basis.

The CAS Quality and Ethics Program has been in place for several years. However, this is a "living" program that will change and improve as the company grows and changes.

Steve Vincent, President/CEO — Date: 3-19-09

Lee Wolf, Chief Quality/Ethics Officer — Date: 3-19-09

Quality Assurance Manual

Analytical Services

Release Date December, 2008

Prepared by:
TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court
Grand Rapids, MI 49512
616-975-4500

1.0 TITLE PAGE

QUALITY ASSURANCE MANUAL

Policies and Procedures Required of the Personnel Employed by TriMatrix Laboratories, Inc., Including the Organic, Inorganic, and Metals Laboratory Areas

Revision Number: 7.0

Effective Date: December 2008

Initial Approvals:

Quality Assurance Manager: _____ Date: 12/2/08

Technical Director: _____ Date: 12/5/08

Laboratory President: _____ Date: 12/4/2008

Subsequent Approvals:

Quality Assurance Manager: _____ Date: 2/16/10

Quality Assurance Manager: _____ Date: _____

Quality Assurance Manager: _____ Date: _____

Quality Assurance Manager: _____ Date: _____

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7.0 APPENDICES

3.0 QUALITY SYSTEM

3.1 INTRODUCTION: THE TriMatrix QUALITY SYSTEM

3.1.1 Manual Purpose

The purpose of this manual is to outline the organization, specify the procedures, and define the technical requirements utilized by TriMatrix Laboratories, Inc. The goal is to ensure that all data generated is of the required quality, is reproducible, and is generated in a timely manner. This manual details a Quality Assurance/Quality Control (QA/QC) program encompassing the entire analytical efforts at TriMatrix, from project initiation to report generation. Some areas are covered with only a cursory discussion, while others are covered in detail, or are included in more than one section, depending on their importance. This manual describes the realistic functions of the quality programs in place, with an understanding that not every situation is covered nor every contingency explored.

3.1.2 The Need for Analytical Quality Assurance/Quality Control

In the increasingly competitive business of environmental laboratory services, the primary tenet of continued success is to efficiently provide results of the necessary quality. TriMatrix agrees with this tenet, considers analytical quality assurance and quality control to be of prime importance, and has incorporated it as the central pillar of our efforts to remain on the leading edge of the environmental laboratory field. The requirements we place on ourselves are in concert with the needs and agendas of other organizations, such as the Environmental Protection Agency (EPA), governmental and industrial clients, and various state and local regulatory agencies.

Quality assurance and quality control (QA/QC) functions absorb nearly fifty percent of the available effort involved in routine analysis, and continues to evolve and grow in importance. This level of quality is absolutely essential for two reasons: 1) accurate analytical data is obtained only with the concurrent

use of extensive QA/QC to regulate and monitor the many process variables that can potentially introduce errors into chemical analyses, and 2) clients make crucial business decisions based on the data supplied by the laboratory. Lab data not properly supported by adequate quality assurance/quality control practices and procedures can be questionable at best, and can lead to faulty or erroneous decisions in the field. In the overall analytical effort the additional time spent for QA/QC is time necessarily spent.

3.1.3 Definition of Terms

3.1.3.1 Quality Assurance

Quality Assurance (QA) is defined as those operations and procedures undertaken to provide measurement data of documentable quality that have a stated probability of being accurate. The measurement system part of the quality assurance program must be in statistical control to justify this probability statement.

The operations and procedures established as part of the overall quality assurance program encompass all aspects of the laboratory operations, including but not limited to: organizational structure, human resources, physical resources, methodology, analyst training and certification, data reduction, data validation, and instrument maintenance and troubleshooting. All aspects of QA are organized, implemented, and monitored through written standard operating procedures.

3.1.3.2 Quality Control

Quality control is defined as the basic checks necessary to produce a good measurement program. These checks include but are not limited to: proper calibration and calibration verification, statistical monitoring of accuracy and precision, of quality control samples

(e.g. laboratory control samples, blanks, duplicates, spikes, etc.), interference monitoring, and reagent control.

Adequate records are maintained to support data quality, to locate assignable causes in measurement problems, to improve the accuracy and precision of the measurement system, and to provide a historical record of traceability.

3.1.3.3 Quality Assessment

Quality assessment is defined as those specific steps utilized to evaluate the quality of the measurement process. These steps include use of control charts to plot multiple data points over time, monitoring parameters by statistical control, internal performance audits, external performance audits, certification programs conducted by individual states, and performance evaluation sample programs.

3.2 QUALITY POLICY STATEMENTS FROM MANAGEMENT

As communicated from top management through the entire organization, TriMatrix Laboratories, Inc. is driven by the following quality objectives and commitments.

3.2.1 Corporate Quality Objectives

- To create and maintain a uniform and controlled pattern for performing routine tasks within the organization, based on standard operating procedures.

- To generate legally defensible, scientifically sound laboratory data of documented quality.

- To build quality into the workplace ensuring services contributing to successful relationships with our customers, employees, and vendors.

- To develop, deliver, and maintain, excellence in all operational areas.

- To provide a service that consistently meets or exceeds client expectations.

3.2.2 Corporate Quality Commitments

- To support quality by underwriting the substantial cost of the quality commitment even though such expenses do not result in increased productivity or a tangible product.

- To maintain a work environment in which all employees are free from commercial pressures in the performance of their duties.

- To maintain a work environment in which all employees are free from internal organization or external client related pressures that may influence the quality of their work.

- To educate all employees in fraud prevention and their ethical responsibilities associated with analytical and data reporting activities.

- To ensure that client confidentiality and information are strictly protected.

- To implement on-going improvement in every area of laboratory activity.

- To create and maintain a Quality Environment with an all-encompassing determination to meet the needs and quality objectives of our clients.

- To commit and adhere to the requirements specified in ISO/IEC 17025.

- To commit and adhere to the requirements specified by the NELAC Standards.

Included with these improvements and commitments is an annual review process where the management of TriMatrix Laboratories performs a comprehensive review of the quality system. This review monitors the effectiveness of the quality system and provides feedback for on-going improvement. Policy changes made as a result of the annual review will be reflected in the QA Manual.

3.3 ORGANIZATION AND RESPONSIBILITIES

An efficient organizational operation requires a quality control program facilitating a high level of multi-directional communication and information flow. Each person in the TriMatrix organization inputs and receives information from the quality system. This information flow optimizes management directives with minimum disruption, and provides the means for creating improvements.

3.3.1 Corporate Structure

Flow of both administrative and quality control information is presented in Figure 3-1. This diagram graphically displays the corporate philosophy concerning the interaction of QA/QC and the generation of analytical data. The general flow of data in this format gives QA/QC independence in fulfilling its function while still acting as a liaison with the administrative staff. To further explain this interaction, a detailed description of roles and responsibilities is presented for each key laboratory position.

3.3.2 Laboratory President

Responsibilities of the Laboratory President are directed at the overall operation and management of the laboratory. Primary responsibilities include, but are not limited to: 1) develop and meet budgets established for the laboratory, 2) manage analytical services productivity and quality, 3) oversee and develop new business activities including client relations development, 4) plan analytical services organization, leadership and management programs, 5)

develop and manage human resources including career path planning, and 6) performing duties as Deputy Technical Director when necessary.

3.3.3 Quality Assurance Manager

The Quality Assurance Manager is primarily responsible for the implementation, maintenance, reporting, and development of all QA/QC activities performed within the laboratory. Duties include, but are not limited to: 1) QA/QC systems development and monitoring, 2) coordination of all documentation procedures including the development and control of standard operation procedures, 3) monitoring method and quality control requirements as published by regulatory agencies ISO/IEC 17025, and the NELAC Standards, 4) performing internal lab audits, 5) maintaining in-house QA/QC monitoring procedures and policies, and 6) providing quality assurance guidance and training to all staff members. The Quality Assurance Manager has the authority to stop work as a result of poor data quality.

3.3.4 Technical Director

The Technical Director is responsible for the overall technical capabilities and direction of the laboratory. Specific responsibilities include: 1) organization and management of new analytical technologies developed by the laboratory, 2) adherence to ISO/IEC 17025 requirements and NELAC Standards, 3) equipment procurement management.

3.3.5 Health and Safety Officer

The Health and Safety Officer is responsible for implementation, monitoring, and maintenance of all laboratory safety and chemical hygiene programs. Specific responsibilities include the development and maintenance of health and safety programs and manuals.

3.3.6 Vice President of Laboratory Operations

The Vice President of Laboratory Operations is responsible for the overall supervision of the individual laboratory areas. General responsibilities include management of staff activities such as scheduling, budgeting, training, and general supervision. The Vice President of Laboratory Operations also is responsible for 1) the development and management of all chemists, analysts, technicians, 2) implementation of quality systems and controls within the laboratory, 3) scheduling analysis activities, 4) meeting productivity goals and project deadlines, 5) technical development of the laboratory staff, 6) approval of laboratory's SOPs, 7) coordination of methods development with the staff and Technical Director, 8) approval of laboratory data, or the delegation thereof, 9) Approval of procurement activities, 10) Overall laboratory performance, and 11) adherence to ISO/IEC 17025 requirements.

3.3.7 Client Services Manager

The laboratory Client Services Manager supervises both the Client Services and the Data Management Group. Responsibilities of the Client Services Manager include management of scheduling and method development needs, budgeting, training, and general supervision, with specific emphasis on the following activities: 1) development and management of all project chemists, project chemist technicians, log-in staff, bottle preparation staff, laboratory couriers, the Field Services Group, and Data Management Group, 2) project management, 3) coordination of proposal preparation and marketing activities for existing and new clients, 4) monitoring of final report turnaround times and, 5) monitoring client satisfaction with laboratory services.

3.3.8 Deputy Quality Assurance Manager/Deputy Technical Director

The Deputy Quality Assurance Manager/Technical Director has the responsibility of fulfilling an interim role as outlined in sections 3.3.3, 3.3.4, 3.5.1.2, and 3.5.1.3.

3.3.9 Sales and Marketing Staff

The Sales and Marketing Staff are responsible for all marketing, business development, and client maintenance activities. These activities include but are not necessarily limited to: 1) market research/gathering market intelligence, 2) consulting with company management to develop a corporate business strategy and plan, 3) development and implementation of a corporate image campaign, 4) development and distribution of marketing materials (corporate literature, etc.), 5) client prospecting, 6) presenting/introducing company services to prospective clients, 7) account development, management and maintenance (in conjunction with Project Chemists), 8) development of corporate pricing guidelines, 9) development of proposals, quotations, bids and qualifications summaries, and 10) contract review, negotiation and execution.

3.3.10 Organizational Chart

Presented in Figure 3-2 is an organizational chart illustrating the personnel structure within the laboratory.

3.4 RELATIONSHIPS

Relationships within the analytical laboratory are organized through management into three main categories: Technical Operations, Support Services, and the Laboratory Quality System. The relationships between management and these operations define and maintain the delicate balance in a cost-effective, highly-technical, quality laboratory operation. An overview of each relation is presented below:

3.4.1 Management-Technical Operations

The relationship between management and technical operations is illustrated in Figure 3-3. In this relationship, the main role of management is to provide guidance and financial support to the programs and directives of the Technical Director. Through this structure, technical operational enhancements and developments occur and are applied through the laboratory staff.

3.4.2 Management-Support Services

The relationship between management and support services is illustrated in Figure 3-4. In this relationship, management's role is substantial in the day-to-day operation of each service.

The primary laboratory support groups are Client Services, Sales and Marketing, and LIMS system support. These groups report directly to the Laboratory President for all aspects of their daily activities.

Secondary relationships are maintained with the Laboratory Administrative Assistant, Laboratory Receptionist, Accounting, and the Human Resources Department. Some groups within this secondary category maintain relationships not only with the Laboratory President, but also with other management groups within the TriMatrix organization.

A tertiary relationship has been developed between the Laboratory President and Vice President of Laboratory Operations. This relationship supports productivity monitoring, cost containment, equipment procurement, operations management, personnel/human resources activities, technical support, data validation, and method development.

3.4.3 Management-Quality System

The relationship between management and the laboratory quality system is illustrated in Figure 3-5. In this relationship, management plays a secondary role in the overall scheme. This secondary role provides the quality assurance manager with guidance, company perspective, and structured support in the development, implementation, and maintenance of quality system programs and activities.

This relationship is vital to the success of TriMatrix Laboratories. Without a cost-effective quality system, the overall caliber of laboratory data and the success of all laboratory operations would be jeopardized.

A relationship also exists between management, the quality system, the laboratory support, and the HR staff. This relationship includes but is not limited to: laboratory management directives, and human resources/personnel activities. These activities are implemented and maintained without disruption to the quality system, and are depicted via the dashed lines on Figure 3-5.

3.5 JOB DESCRIPTIONS

The strength of a laboratory lies in the experience and dedication of its employees. TriMatrix hires quality personnel based both on work attitude and past job experience. Job descriptions have been written to define the employee qualifications required for each position.

3.5.1 Management Staff Members

Managerial positions are responsible for the development of their respective employees. These positions have specific minimum requirements for years of experience.

3.5.1.1 Laboratory President

Job Description

The Laboratory President (LP) directs the laboratory. The LP works through the Vice President of Laboratory Operations to improve data quality, overall productivity, staff development, safety/training programs, and overall profitability. This position has profit/loss accountability. Budgets are developed annually with senior management. The LP is also directly involved in business development/sales activities, and the sales staff reports directly to him.

Background/Educational Requirements

The LP possesses minimally a bachelor's degree in science, preferably chemistry. The LP has a minimum of 10 years direct

work experience in the environmental testing industry. This work experience includes having conducted environmental analyses and several years of demonstrated supervisory experience.

Duties and Responsibilities

1. Development and fulfillment of budgets.
2. Management of total laboratory productivity and quality.
3. Management of proposal preparation.
4. Development of new business and maintenance of client relationships.
5. Development of laboratory organization, leadership, and management planning.
6. Working with the Human Resources department to develop staff members and their career paths.

3.5.1.2 Quality Assurance Manager

Job Description

The Quality Assurance (QA) Manager is responsible for the development, implementation, improvement, and maintenance of all quality systems at TriMatrix. The QA Manager monitors all the analytical methods and procedures performed by the laboratory, and assures compliance with regulatory agency requirements.

Background/Educational Requirements

The QA Manager possesses a B.S. in science, preferably chemistry, and suitable work experience. Work experience must include several years of analytical work and a demonstrated ability to work with and train staff members. A strong working knowledge of quality assurance and statistical quality control procedures, specifically as they apply to analytical protocols, is required.

Duties and Responsibilities

1. Development and implementation of systems to measure and monitor laboratory data quality.
2. Maintenance of the documentation system for generation, control, and archiving laboratory forms, SOPs, and protocols.
3. Approving SOPs and monitoring their compliance with regulatory agency requirements.
4. Maintaining and updating the laboratory Quality Assurance Manual.
5. On-going investigation for optimizing procedures to minimize out-of-control data.
6. Maintenance of federal, state, and industrial certifications and accreditations as required.
7. Monitoring internal quality programs within the laboratory and reporting their status to management.
8. Training and training documentation of all staff members in all aspects of the laboratory quality system.
9. Perform other duties as deemed necessary by management.

3.5.1.3 Technical Director

Job Description

The Technical Director (TD) is responsible for the development and improvement of technical operations within the laboratory division. The TD oversees the investigation of all new instruments and equipment, method development, and general technical advancement of the laboratory. The TD is also responsible for informing the Deputy TD of current and pending projects and activities.

Background/Educational Requirements

The TD possesses a B.S. in science, preferably chemistry, and suitable work experience. Such work experience includes several years of analytical work and a demonstrated ability to work with and train staff members. A strong working knowledge of

instruments and methodologies, specifically as they apply to analytical protocols, is required.

Duties and Responsibilities

1. On-going technical development of the TriMatrix Laboratory pertaining to current and future analytical practices.
2. Overseeing the technical development of TriMatrix staff in the areas of method comprehension and implementation.
3. Development of new analytical procedures within the laboratory.
4. Providing technical advice regarding all equipment and apparatus procurement, and acquisitions.
5. Performing technical review of all Quality Assurance Project Plans (QAPPs).
6. Perform other duties as deemed necessary by management.

3.5.1.4 Client Services Manager

Job Description

The Client Services (CS) Manager is responsible for the supervision of the project chemists, project chemist technicians, sample log-in staff, bottle preparation staff, laboratory couriers, field services group, and laboratory administrative staff. These responsibilities include meeting project due dates, preparing and reviewing quotations, project initiation and management, client satisfaction management, and supervision and training of staff. The CS Manager strives for improvement in the on-time delivery of laboratory projects.

Background/Educational Requirements

The CS Manager possesses a B.S. in science, preferably chemistry, and has 5-10 years of work experience. The work experience includes 3-5 years of laboratory experience, involvement in client

management activities, and a demonstrated ability to supervise and train laboratory staff.

Duties and Responsibilities

1. Responsible for the productivity and quality of the client services group.
2. Management of large Level 3 or higher projects.
3. Quality control program implementation and maintenance.
4. Supervision and technical development of employees.
5. Development and maintenance of standard operating procedures.
6. Assisting and coordinating marketing activities through proposal preparation and client visitation.
7. Perform other duties as deemed necessary by management.

3.5.1.5 Vice President of Laboratory Operations

Job Description

The Vice President of Laboratory Operations (VPLO) is responsible for the individual laboratory areas and the supervision of laboratory staff. These responsibilities include meeting project schedules, and the supervision and training of staff members. The VPLO continually works to improve the quality of data generated.

Background/Educational Requirements

The VPLO possesses a B.S. degree in science, preferably chemistry, and 5-10 years work experience. The work experience includes a minimum of 5 years in the laboratory utilizing a variety of techniques. The VPLO must also demonstrate an ability to supervise and train staff members.

Duties and Responsibilities

1. Responsible for the productivity and quality of the laboratory areas.

2. Operation and maintenance of instrumentation and apparatus.
3. Quality control program implementation and maintenance.
4. Reviewing and final approval of all organic data.
5. Scheduling in-house to allow on-time report generation.
6. Supervision of supply acquisition activities.
7. Supervision and technical development of employees.
8. Approval of standard operating procedures.
9. Methods development.
10. Perform other duties as deemed necessary by management.

3.5.1.6 Laboratory Computer Systems Administrator

Job Description

Provide technical review, guidance, and training in current and future laboratory computer applications.

Background/Educational Requirements

Requires a degree in computer sciences with an emphasis in a chemistry or general science curriculum.

Duties and Responsibilities

1. Developing a complete understanding of the Laboratory Information Management System (LIMS).
2. Reviewing laboratory computer applications and processes, including instrument computer interfaces, data transmission/archiving processes and document control.
3. Providing database maintenance support activities for the LIMS system.
4. Providing technical direction and orchestrating implementation of electronic storage systems for the laboratory.
5. Providing technical training of the laboratory staff in software applications and basic computer operational activities.
6. Perform other duties as deemed necessary by management.

3.5.2 Technical Staff Members

Technical staff members are classified into chemist or technician levels dependant on job type, education, and years of experience. Level Classifications are Chemist I-V and Senior Chemist, Project Chemist I-V and Senior Project Chemist, Technician I-V and Senior Technician. In addition, qualified candidates are also eligible for group leader status. Classification descriptions are provided in Appendix A. To aid the employee in identifying the different classification requirements, the differences are printed in bold italicized text. The various classifications are also used by the employee and by management for career path development at TriMatrix.

3.6 MANAGEMENT RESUMES

Laboratory President
Quality Assurance Manager
Vice President of Laboratory Operations
Human Resources Manager

DOUGLAS E. KRISCUNAS — Laboratory President

EDUCATION

B.S., Environmental Sciences, Grand Valley State University, 1976

PROFESSIONAL SUMMARY

Mr. Kriscunas is responsible for the accuracy and integrity of all analytical data finalized at this location. He is continuously available for client support to resolve analytical issues as they pertain to environmental problems.

PROFESSIONAL EXPERIENCE

- **Detroit, Michigan**. Laboratory Supervisor for a field laboratory established at the Detroit Wastewater Treatment Plant. The project involved a one-year pilot study of the overall operation and plant performance to upgrade and modify existing treatment processes to meet current and future discharge limits. Approximately 20,000 samples were analyzed by seven full-time analysts.

- **Edmore, Michigan. Hitachi Magnetics Corporation.** Participated in the development and implementation of an on-site, flow-through bioassay of the plant discharge. The study was performed in conjunction with the Michigan Department of Natural Resources, Water Quality Division.

- **Grand Rapids, Michigan. EDI Laboratory Certification.** Direct responsibility for the inorganic parameters analysis and quality control measures necessary for laboratory certification under the Safe Drinking Water Act (SDWA) of 1974. Certification involved both analysis of unknown control samples and corresponding on-site evaluation by the U.S. EPA Region V laboratory certification team.

- **Muskegon, Michigan. Uniroyal Chemical Company.** Participated in the soil survey and on-site evaluation of potential soil contamination from deposited chemical waste materials produced by a major chemical company. On-site sample analyses for select parameters were made to locate and detail the extent of contamination.

- **Edmore, Michigan. Hitachi Magnetics Corporation.** Participated in the implementation of a treatability study to effectively remove cobalt and samarium from industrial waste. The study results led to the design and installation of treatment facilities.

- **Columbia, Missouri. A.B. Chance Corporation**. Responsible for implementing a treatment study for effective removal of heavy metals from process wastewater in order to achieve acceptable discharge limits.

- **Kent County, Michigan. Mill Creek Watershed Management Project**. Participated in the collection, mapping, and interpretation of environmental characteristics to be used as prototype guidelines for the management of area wide streams in the Great Lakes Basin. The project was funded by the Environmental Protection Agency.

- **Three Rivers, Michigan. Hydramatic Division, General Motors Corporation.** Responsible for the analytical services conducted on a survey of process wastewater for an automotive transmission manufacturer. The project involved data collection and analytical services including grab samples, setting automatic samplers on an hourly basis for a seven-day period, and installing recording meters for continuous pH monitoring.

- **Grand Rapids, Michigan. Michigan Department of Public Health Laboratory Certification.** Supervised analytical, bacteriological, and quality control activities involved in achieving certification status for the analysis of potable water supplies in Michigan.

- **Higgins Lake, Michigan. Ralph MacMullan Conference Center**. Served on a three-member panel before a meeting of the Northern Michigan Environmental Health Association. The topic of discussion was an overview of organic chemicals now found in much of Michigan's ground waters. A representative from industry and the MDPH laboratory completed the panel.

- **Grand Rapids, Michigan. Haviland Chemical Company.** Coordinated a static bioassay performed on a water-based detergent utilizing fathead minnows in the 96-hour static test.

- **Sparta, Michigan**. Conducted a dendrological survey of a proposed oil drilling site. The survey was incorporated in an overall environmental assessment of the proposed drilling site.

- **Caledonia, Michigan**. Conducted a dendrological survey of riparian vegetation types located along the banks of the Thornapple River in the area of the Labarge Dam.

- **Grand Haven, Michigan**. Conducted a limnological investigation of the estuary waters of the Grand River watershed near Grand Haven. The collected limnological data were evaluated for potential eutrophication problems resulting from nutrient discharges upstream.

- **Kalamazoo, Michigan. American Cyanamid Company**. Supervised laboratory work required in assisting a major chemical manufacturer with a permit application for existing facility hazardous waste management operation to administratively complete four supplemental technical attachments, multidisciplinary services were required in the areas of hydrogeologic investigation, environmental assessment, failure mode assessment, and engineering review. Field work was completed in 19 days with a report to the client in 25 days to meet scheduled deadlines.

- **Kent County, Michigan**. Coordination of field and laboratory services in conjunction with Act 641 monitoring requirements at two county-owned and operated refuse sites.

Specialized studies were also conducted to identify possible use of landfill gases for electric power generation and the source identification of volatile organic contaminants typical of most municipal landfills.

- **Cascade Township, Michigan. Cascade Resource Recovery/Waste Management, Inc.** Implementation of two separate tracer studies aimed at pinpointing possible cracks or defects in the clay liners of four hazardous waste disposal trenches. The study utilized a low absorptivity fluoroscene water soluble dye introduced to each trench. Samples collected from each liner failure detection system were then analyzed for the fluorescent characteristics of the dye.

- **Cascade Township, Michigan. Cascade Resource Recovery/Waste Management, Inc.** Coordination of field and laboratory services in connection with Michigan Department of Natural Resources Act 64 and U.S. EPA RCRA monitoring requirements. Each sampling event involves collection of ground waters, surface waters, and leak detection monitoring sites.

- **Cascade Township, Michigan. Cascade Resource Recovery/Chemical Waste Management, Inc.** Acted as project chemist and field services coordinator for activities involved in the excavation and site decontamination of an Act 64/RCRA hazardous waste disposal facility. The decontamination program involved the analysis of soils collected in and around each disposal trench after the removal of approximately 20,000 cubic yards of waste materials.

- **Cincinnati, Ohio. Rumpke Waste Systems, Inc.** Acting project manager for a large waste disposal firm headquartered in Ohio, with 20+ landfills located in a 5 state geographical area. Mr. Kriscunas is responsible for coordination of laboratory activities in conjunction with all ground water, surface water, and NPDES monitoring requirements.

RICK D. WILBURN — Quality Assurance Manager

EDUCATION

B.S., Environmental Studies, Earlham College, 1985

PROFESSIONAL SUMMARY

Mr. Wilburn is responsible for all aspects of the laboratory Quality Control/Quality Assurance Program. Primary responsibilities include conducting internal and external auditing of the laboratory, procurement and maintenance of state and federal certifications, and ensuring that all facets of the quality control program remain at the highest level possible. Mr. Wilburn also manages the external and internal Quality Control check sample programs.

PROFESSIONAL EXPERIENCE

- **TRACE Analytical Laboratories, Inc. – Quality Assurance Manager, 12/95 – 10/96.** Responsible for designing, implementing, and monitoring a formal quality control program. The program included: conducting internal and hosting external audits, implementing corrective actions resulting from any deficiencies, scheduling and reporting performance evaluation sample results, and the review of all Level 5 data packages.

- **EARTH TECH – Organic Laboratory Manager, 10/95 – 12/95.** As Organic Laboratory Manager, Mr. Wilburn was responsible for the day-to-day operations of the organic laboratory, including volatile and semi-volatile analyses by gas chromatography and gas chromatography/mass spectrometry. His responsibilities included scheduling, instrument maintenance, the writing and implementation of standard operating procedures, quality assurance, analytical data review, the technical development of all the organic laboratory personnel, and project management. Mr. Wilburn was also responsible for research and development in the organic laboratory, focusing on ways to automate and improve sample analysis, data quality, and turnaround time.

- **EARTH TECH (Formerly WW Engineering & Science) – Semi-Volatile Laboratory Supervisor, 1/94 – 10/95.** Responsible for the daily operation of the semi-volatile laboratory. The semi-volatile laboratory utilizes gas chromatography, gas chromatography/mass spectrometry, and high performance liquid chromatography in the analysis of semi-volatile organic compounds.

- **WW Engineering & Science – Supervisor, Organic Extraction Laboratory, 4/93 – 1/94.** Supervisor of the staff of chemists responsible for all organic extractions. Accountable for the processing, quality, and turn around of a wide variety of samples involving many extraction techniques and methodologies. Continually experimenting with automation and new technologies to improve extraction quality and turn around time, including solid phase and supercritical fluid extractions.

- **WW Engineering & Science – Supervisor, Mass Spectrometry Laboratory, 9/89 – 1/94.** Supervisor of the staff of chemists analyzing samples for semi-volatile organics in the mass spectrometry laboratory. Oversee all analysis and daily activities involved with the mass spectrometry laboratory. Evaluate, recommend, and implement new technologies. Implementations of these include sub-ambient injections using a Varian SPI injector, sub-ambient temperature programs for optimized chromatography, and the use of ion trap mass spectrometers for lower operating detection limits

- **IT Corporation, (formerly PEI Associates, Inc.) – Chemist, Level 3, GC/MS Semi-Volatile Team Leader, 7/88 – 9/89.** Along with daily analysis of samples, responsible for coordinating the efforts of the three analysts and three instruments used for semi-volatile analysis. This included scheduling each instrument/analyst to make sure analyses were completed correctly and on time, training new personnel, instrument maintenance, data checking, and reporting project results to management for client distribution. Leader of GC/MS Quality Circle group.

- **PEI Associates, Inc. – Chemist, Level 2, GC/MS Analyst, 12/86 – 7/88.** Primary responsibilities included analyzing soil, water, and other media with an Extrel ELQ-400 mass spectrometer system. Analyses performed included semi-volatile and volatile organics listed on the EPA's Toxic Compounds List according to the Contract Laboratory Program protocol. Also analyzed various other non - Toxic Compounds List compounds using appropriate methods.

- **PEI Associates, Inc. – Chemist, Level 1, GC Analyst, 7/85 – 12/86.** Carried out a variety of organic analyses in a wide range of matrixes. Was a primary analyst conducting CLP testing for pesticides and PCBs, and was the primary analyst for routine and non-routine testing for herbicides, and volatile organics.

JEFFREY P. GLASER — Vice President of Laboratory Operations

EDUCATION

B.S., Biochemistry, Michigan State University, 1987

PROFESSIONAL SUMMARY

Mr. Glaser is responsible for the operation and management of the laboratory areas. Main functions include supervision and training of personnel, formulation of standard operating procedures, final approval of laboratory data, and laboratory purchase approval.

PROFESSIONAL EXPERIENCE

- **TriMatrix Laboratories, Inc., Muskegon – Laboratory Manager, 1994 – 1996.** Responsible for all aspects of laboratory performance. He was responsible for all aspects of laboratory performance including, analytical testing and reporting; business development; customer service; capital expenditures, quality control; quality assurance; laboratory safety; and laboratory profitability. He was responsible for the hiring, training, guidance, and evaluation of all laboratory personnel, and for direction of overall laboratory policies and practices.

- **Great Lakes Environmental Laboratories – Senior Chemist, 1992 – 1994.** Mr. Glaser's responsibilities included supervision and training of other laboratory personnel, coordination of sample workloads, data review and evaluation, and quality control. He was also responsible for analysis of pesticides, PCBs, and herbicides using an HP 5890 GC w/ECD detectors.

- **Anatech Analytical Laboratories – GC/MS Operator, 1990 – 1992**. Mr. Glaser was responsible for the mass spectrometry analysis of environmental samples in a variety of matrixes for both volatile and semi-volatile organics. For volatiles, Mr. Glaser operated and maintained a Finnigan Ion Trap GC/MS system consisting of a Varian GC and a Tekmar purge and trap autosampler. Primary methodology used was 624/8240. For semi-volatiles, he operated and maintained a Hewlett Packard GC/MSD UNIX-based Chem Station. Primary methodology used was 625/8270. He was also responsible for method development. He served as the Organic Supervisor for the first quarter of 1991.

- **Anatech Analytical Laboratories – Volatile Organic Chemist, 1989 – 1990**. Mr. Glaser was responsible for operation and maintenance of two volatile GC systems utilizing ELCD, FID, and PID detectors, and Tekmar and O.I. Analytical purge and trap autosamplers. Primary analyses were 601 and 602.

STACY K. VANDEN AKKER — Human Resources/Business Manager

EDUCATION

B.S. Business Management, Davenport Business College, 1996.

PROFESSIONAL SUMMARY

As Business Manager, Ms. Vanden Akker is responsible for the record keeping and review of all financial data for the company. She manages accounts payable, accounts receivable, cash flow, and the generation of financial statements and other management reports. She maintains accurate records for potential audit or other review.

Ms. Vanden Akker also manages all Human Resource functions for TriMatrix Laboratories. She processes payroll on a biweekly basis, coordinates employee benefits, handles internal employee questions and concerns, assures compliance with all federal, state, and local employment laws and regulations, and maintains complete and accurate personnel data files.

PROFESSIONAL EXPERIENCE

- **EARTH TECH – Environmental Laboratory Business Office, Administrative Assistant, 9/95 – 1/97.** Responsible for assisting the Business Office Manager with accounts receivable, accounts payable, and the daily input of purchases and invoices.

- **EARTH TECH – Lowell Wastewater Treatment Plant Operator/Laboratory Technician, 8/93 – Present.** Responsible for sample collection, equipment maintenance, and the daily laboratory analysis of suspended solids, CBOD, ammonia, zinc, fecal coliform, pH, residual chlorine, and phosphates. She is also responsible for the correct input of all results into the reports required by the State of Michigan Department of Environmental Quality.

- **EARTH TECH – Lowell Wastewater Treatment Plant Assistant Laboratory Technician, 8/90 – 8/93.** Assisted the Laboratory Technician in the laboratory analysis of suspended solids, CBOD, ammonia, zinc, fecal coliform, pH, residual chlorine, and phosphates.

3.7 APPROVED SIGNATORIES

Designated laboratory staff members have the responsibility of validating laboratory documents on behalf of the laboratory organization. General categories and documents requiring a valid signature are presented below.

3.7.1 Client/Invoice Reports

All laboratory reports compiled and mailed contain at least one representative signature validating the contents of the laboratory report. By default, a report is signed by the appropriate project chemist. Alternate and/or additional signatures include the Laboratory President, Client Services Manager, Technical Director, Quality Assurance Manager, and Vice President of Laboratory Operations. No other individuals are approved to perform signatory approval of client/invoice reports.

3.7.2 Proposals, Price Quotations, and Laboratory Contracts

Proposals or price quotations for laboratory services contain at least one representative signature, validating the pricing, terms, and conditions of the quotation. At least one representative signature is required. Approved signatures for proposals and price quotations include the Laboratory President, Client Services Manager, project chemists, and a sales or marketing representative.

Required signatures for laboratory contracts are the Laboratory President and a Sales or Marketing representative.

3.7.3 Quality Assurance Project Plans (QAPP)

Quality Assurance Project Plans contain representative signatures of several responsible parties outside the laboratory. The only laboratory signature generally found on a QAPP is that of the QA Manager. The QA Manager has designated QA/QC responsibilities that are fully documented in QAPP

documents. All QAPPs are signed prior to submission to a governing body or client.

Signatures on the QAPP ensure all procedures, materials, quality control practices and project reports meet the predefined goals of the plan.

3.7.4 Purchase Orders and Agreements

Because the laboratory spends a significant portion of its annual budget on supplies and equipment, guidelines have been established to document and control purchasing.

Purchasing of general supplies is handled through a contracted vendor within the budgetary guidelines established for each laboratory area.

For major purchases such as equipment, service assessments, or building renovations in excess of $500.00, purchase orders or agreements must be approved by the Laboratory President or CEO.

3.7.5 Binding Statements - Laboratory Certification Documents or Accreditation

Many certification or accreditation programs require the laboratory to provide items and statements regarding details on the laboratory's operations and staff. In some cases these statements must be presented to the certifying body accompanied by a binding signature of the laboratory president or CEO.

3.8 CAPABILITIES, CERTIFICATIONS, ACCREDITATIONS, AND PROFICIENCY TESTING PROGRAMS

3.8.1 Capabilities

TriMatrix conducts analytical laboratory services in support of all major environmental regulations, including CERCLA, RCRA, CWA, CAA, and TSCA.

The laboratory is capable of routinely analyzing a variety of sample matrices, including drinking water, surface water, wastewater, soil, groundwater, solid waste(s), and sludge(s). In addition, analyses have been performed on fish tissue, biota, and air samples by project request.

TriMatrix routinely performs a wide array of environmental and non-environmental, chemical and physical analyses. A list of methods currently utilized by TriMatrix is provided in Appendix B. To maintain a quality system of analytical protocols, TriMatrix uses written Standard Operating Procedures (SOPs) derived from methodology specified by the United States Environmental Protection Agency, other federal and state agencies, and professional compendia.

When requested by the client, samples for analyses outside the analytical scope of TriMatrix can be subcontracted to another laboratory. Unless otherwise specified or required by the client, samples will be subcontracted to a NELAP accredited or ISO-17025 certified laboratory.

3.8.2 Laboratory Certification - Federal, State, and Independent

TriMatrix has been formally recognized for its commitment to quality. The laboratory maintains certification through various federal agencies, as well as several state regulatory agencies and private entities. As required by most of the programs, including NELAP and A2LA, certification and accreditation claims must be made in such a manner as to not imply certification or accreditation beyond that given on the laboratory's actual scope of accreditation. Generic certification or accreditation claims must not be made. The use of symbols (such as the A2LA symbol) and other forms of accreditation must always be analyte and/or method specific. Certification

programs in which TriMatrix currently participates are listed in the subsections below:

3.8.2.1 Federal Certification/Approval Programs

U.S. Army Corps of Engineers – DoD QSM

U.S. Air Force – AFCEE

U.S. Army Center for Health Promotion and Preventative Medicine – NELAC/A2LA

U.S. Navy – Navy (IR/QA – DoD QSM)

NELAP – National Environmental Laboratory Accreditation Program

3.8.2.2 State Certification Programs

Arkansas	Department of Environmental Quality
Florida	Department of Environmental Protection
Georgia	Environmental Protection Division
Illinois	Environmental Protection Agency
Kansas	Department of Health and Environment
Kentucky	Petroleum Storage Tank Environmental Assurance Fund
Louisiana	Department of Environmental Quality
Michigan	Department of Environmental Quality
Minnesota	Department of Health
New York	Department of Health
Ohio	Ohio VAP Program
Wisconsin	Department of Natural Resources

3.8.2.3 Independent Certification Programs

The American Association for Laboratory Accreditation (A2LA)

3.8.3 Proficiency Testing Studies

An integral part of most certification programs are Proficiency Testing (PT) Studies. PT studies are analyzed periodically as external "blind" or "double blind" spiked samples containing specific (known only to the administrators of the study) concentrations of target analytes. The laboratory reports the results to the agency or firm administering the PT study. The administrator then evaluates the laboratory's performance based on a comparison of the reported values with the known analyte concentrations. Laboratory results are scored and reports are prepared by the study administrator. The reports are submitted to the laboratory, certifying programs, and agencies or private entities that subscribe to the program.

TriMatrix routinely participates in the following proficiency testing programs:

- Water Supply (WS) Study
- Water Pollution (WP) Study
- Soil PT Study
- USEPA DMRQA

3.9 LABORATORY FACILITIES, EQUIPMENT, AND SUPPLIES

3.9.1 Physical Plant

3.9.1.1 Laboratory Demographics

The current TriMatrix Laboratories facility, located at 5560 Corporate Exchange Court SE, Grand Rapids, Michigan, was constructed in 1999. The 20,000 square foot structure was designed predominantly by the laboratory staff, with careful consideration given to the strict analytical testing requirements of today's environmental marketplace. Special attention was given to the sample preparation areas and the segregation of non-

compatible areas such as semi-volatile and volatile organics. Samples are stored according to type, with a large centrally located walk-in cooler used for the storage of all non-volatile, non-hazardous waste samples, to which both the sample receiving personnel and the laboratory staff have ready access. Quiet office areas were also built in, to provide space for data review, report compilation, and technical review discussions. A breakdown of each general area of analysis and the space allocated is as follows:

Laboratory Area	**Space Allotted, ft^2**
Wet Chemistry/Microbiology	Approx. 2000
Atomic Absorption/Emission	Approx. 2000
Volatile Organics	Approx. 1600
Semi-Volatile Organics	Approx. 2300
Sample Processing & Storage	Approx. 2400
Administrative Offices	Approx. 4200
Organic Pretreatment	Approx. 1300
Miscellaneous Space	Approx. 4200

The attached facility layout (Figure 3-6) shows the general lab areas and other space allocations.

Access to all laboratory areas including sample storage, sample container preparation, sample preparation, sample disposal, documents storage and clients files are secured. Non-authorized personnel may enter these areas only if escorted by a laboratory staff member.

Project initiation, sample control, and analysis, are all controlled using a Laboratory Information Management System (LIMS).

Under the direction of the Laboratory President, TriMatrix is organized into the following operating areas and support services.

Laboratory Administration

Client Services
Data Management
Sales/Marketing
Project Management
Health and Safety
Quality Assurance
Computer Services

Analytical Operations

Inorganic Laboratory
- Metals Laboratory
- Non-Metals Laboratory

Organic Laboratory
- Volatile Organic Laboratory
- Semi-Volatile Organic Laboratory
- Organic Extraction Laboratory

(Refer to Figure 3-2 for a graphical representation of the Laboratory Organization Chart)

3.9.1.2 Reagent Water Systems

Laboratory water originates from the Grand Rapids potable water distribution system. At the laboratory, the water is softened and passed through an activated carbon filter to remove residual chlorine. The water then enters a reverse osmosis system where approximately 90% of the dissolved constituents are removed. The water is temporarily stored in a 120 gallon holding tank until demand activates a mechanical pump that transfers the water through two mixed bed deionizing canisters. This water meets the requirements of ASTM Type II, and is utilized for glassware cleaning and as a feed-water to a variety of polishing systems.

The polishing systems are comprised of a distillation unit and a Milli-Q 4 Bowl System. Distilled RO-Deionized water is used primarily for BOD and metals analyses. Mill-Q water, which is equivalent to an ASTM Type I designation, is primarily used for the preparation of standard solutions and reagents.

Each water system is periodically monitored for specific quality requirements. Monthly, heterotrophic plate count and total residual chlorine analyses are performed. Weekly, the water system itself is checked for operational readiness and a hardness test is performed. Daily, additional readiness checks including a conductivity test are performed.

Responsibility for monitoring the TriMatrix reagent water systems is carried out by the Quality Assurance Department and personnel in the inorganic wet chemistry laboratory.

3.9.1.3 Ventilation Systems

The laboratory ventilation system was specifically designed to minimize or eliminate airborne contamination. Externally, the air conditioning unit intakes were located taking into consideration prevailing wind patterns, positioning them upwind of the fume hood exhaust stacks. Taking into account wind-shifts, the exhaust stacks were equipped with high velocity fans to disperse potential contaminants well above the building. Internally, the air-handling systems controlling heating, cooling, and humidity, also maintain maximum cfm air turnover. Additionally, the air-handling systems are monitored and controlled via a NOVAR computer controller.

3.9.1.4 Compressed Air

Compressed air must be free of dirt, water, and oil. Compressed air purchased from vendors is high purity grade (breathing air).

Compressed air produced in the laboratory uses filters at the compressor to remove water from the delivery lines. For the gas chromatographs and atomic absorption spectrophotometers, additional filters are located on the instrument to remove any residual oil at the point of use.

3.9.1.5 Electrical Services

The electrical system in use at TriMatrix was designed specifically for a laboratory environment. Special attention was paid to instrument requirements, including the isolation of separate lines for critical applications like GC, GC/MS, atomic absorption, and automated analyzers.

All laboratory benches, hoods, and work areas were designed with sufficient outlets to accommodate a variety of laboratory applications, such as distillations, digestions, and extractions.

Surge protection devices are in place for all laboratory computing equipment. The laboratory LIMS system is also protected by an Uninterrupted Power Supply (UPS). This UPS allows for a sequenced shutdown of the LIMS system during a power failure. This sequenced shutdown provides excellent protection of the LIMS database during a power interruption.

3.9.2 Equipment, Supplies, and Chemical Procurement; Reception, Storage, and Inventory

For an environmental testing laboratory where trace analyses are routinely performed, certain specifications for laboratory equipment, supplies, and chemicals are critical to quality. A minimum specification for accuracy and precision of equipment such as analytical instrumentation, balances, glassware, and water baths is required for each analytical procedure. The Technical Director in conjunction with the Laboratory President and laboratory area

managers are responsible for determining minimum specifications before equipment is procured. The analytical specifications are based on a detailed review of the test methods. Purchasing is coordinated through the purchasing department. Records are maintained on all vendors exhibiting poor performance on either their service or product. Relationships will be terminated with any vendor whose records indicate sub-standard performance.

3.9.2.1 Equipment Management/Maintenance/Inventory

A sufficient inventory of equipment is maintained to prevent testing delays resulting from equipment failure. Service is performed on equipment on a scheduled basis. A stock supply of spare parts that are known to wear out regularly is maintained.

Adequacy of equipment for its intended purpose must be verified before use. Maintenance logbooks are kept to document maintenance procedures on major equipment, allowing preventive maintenance frequency and requirements to be determined. Maintenance procedures are discussed in the various analytical SOPs.

A complete listing of Laboratory Equipment is presented in Appendix C of this manual.

3.9.2.2 Glassware

Only glassware providing the required precision is used for a particular analytical procedure. TriMatrix purchases Class A pipets, burettes, and volumetric flasks, to meet this specification. A standard operating procedure is utilized for cleaning each type of glassware. Cleaning of glassware is performed according to the analysis being conducted and the sample matrix involved, but certain general rules apply to all glassware washing procedures:

- Use hot water to wash away water-soluble substances.
- Use detergent, dichromate solution, organic solvent, nitric acid, or aqua regia to remove other materials according to the specific glassware cleaning procedures.
- Avoid using detergents on glassware to be used for phosphate determinations.
- Use ammonia-free water for ammonia and kjeldahl nitrogen analyses.

For all analyses, it is advisable to rinse glassware with tap water followed by deionized water immediately after use, as residue allowed to dry on glassware is more difficult to remove.

3.9.2.3 Reagents, Solvents, and Gases

Purchasing of reagents, solvents, and gases are carefully controlled through an ordering system that maintains a minimum level of quality in the testing process. The Quality Assurance Department defines the suitable grades of ordered materials. Designates from each laboratory area verify upon receipt that incoming materials meet these requirements. Certificates of Analysis are forwarded to the Quality Assurance department where they are scanned and stored. Each laboratory area will monitor the proper storage and the eventual removal of reagents, solvents, and gases, when their shelf life has expired. All consumable reagents and chemicals must be labeled with the date received to ensure a First-In-First-Out (FIFO) system of use.

Reagents, solvents, and gases are available from vendors in a broad range of purity, from technical to ultra pure grades. The analysis, as well as the sensitivity and specificity of the method, must be considered when choosing a grade. Analytical reagent (AR) grade is suitable for most inorganic analyses. Trace organic analyses frequently require ultra pure grades. AR grade is the minimum

approved for reagents used in organic analysis. The absence of certain impurities is required for some GC detectors - notably sulfur and phosphorus in an FID detector. Trace metals analyses including atomic emission and atomic absorption spectroscopy usually requires spectro-quality reagents, although AR grade may be suitable in some cases. Florisil, silica gel, and alumina used as absorbents in organic extract cleanups, must be checked for interfering components and activated according to the analytical method. Compressed gases are available in various purities, usually expressed as a percent (e.g. 99.999). Gases are filtered in the laboratory delivery lines to remove moisture, oil, and other contaminants. Refer to the analytical method and instrument manufacturers operating manual for gas purity requirements.

Provided they are available, expiration dates of unopened chemicals are based on the date determined by the manufacturer. They may also be derived from the analytical method. A new expiration date may be required once the chemical is opened. The following guidelines are utilized in assigning expiration dates:

Unopened Reagents, Solvents, and Neat Chemicals
Manufacturers assigned expiration date or 5 years from date received, whichever occurs first.

Opened Reagents, Solvents, and Neat Chemicals
2 years from date opened or remainder of manufacturers assigned expiration date, whichever occurs first.

Prepared Solutions - Stock
Manufacturers assigned expiration date or 1 year from date opened, whichever occurs first.

Prepared Solutions - Working

Assigned expiration date of stock, or 6 months from date prepared, whichever occurs first.

Unpreserved ethers have an expiration date of 34 days due to the potential for peroxide formation.

In order to maintain expiration date accuracy it is critical that the date opened is recorded on all containers, and the expiration date originally entered into the LIMS system be updated based on the date opened.

3.9.2.4 Certified Standards

The purity and traceability of standards used in the analytical process is crucial to the quality of the data generated. Only high quality standards certified by established vendors are to be utilized. Calibration standards must be of the purity required by the method for a particular analysis.

Upon receipt all purchased standards are entered into the LIMS system and labeled with a unique identifier and an expiration date. The date received is also recorded on the container. Stock and working standards are likewise labeled.

All calibration standards are validated against a second source standard. A second source standard is analyzed with every initial calibration. The quantitated value is compared to laboratory established limits. Recovery must fall within these limits for the calibration and calibration standard to be considered acceptable. Stock and working standards are also monitored for visible signs of deterioration (precipitates, color change, volume change).

Vendor expiration dates for purchased stock standards must not be exceeded. Expiration dates for laboratory prepared standards are

based on guidelines in the analytical method, generally 6 months for working, and 1 year for stock standards.

3.9.2.5 Chemical / Reagent Storage

Bulk chemicals and reagents are stored in a several locations and under a wide variety of conditions within the laboratory. Specific storage conditions for many reagents are presented in each laboratory testing SOP. Additional storage information is referenced in both the TriMatrix Laboratory Safety Manual and the TriMatrix Chemical Hygiene Plan. For general purposes, the following storage conditions are used:

Chemical /Reagent Type	General Storage Requirements	Location/Lab Area
1) Bulk Dry Chemicals	Dry Chemical Storage Cabinets	Inorganic Laboratory
2) Inorganic Acids	Vented Acid Storage Cabinets	Metals Laboratory
3) Organic Solvents-Flammable	Vented Flammable Cabinets	Inorganic & Prep Laboratory
4) Organic Solvents-Nonflammable	Vented Storage Cabinets	Inorganic & Prep Laboratory
5) Compressed Gases	Secured Gas Storage Area	Garage & Outside Storage
6) Bacteriological Materials	Reagent Refrigerator	Inorganic Laboratory
7) Aqueous Standards	Reagent Refrigerators	All Laboratory Areas
8) Organic Standards-Flammable	Explosion Proof Refrigerators and Freezers	Organic Laboratory Areas
9) Organic Standards-Nonflammable	Standards Refrigerator & Freezers	Organic Laboratory Areas
10) Sample Extracts	Extract Freezers	Organic Laboratory Areas
11) Digestates-Metals	Vented Acid Storage Cabinets	Metals Laboratory

3.10 TRAINING

Proper training of laboratory personnel is an essential part of staff development. Training procedures include documentation of training activities completed and serve as a guideline for continual staff development. All testing personnel must familiarize

themselves with the laboratory's training procedure (TriMatrix SOP GR-10-109) and implement all associated policies and procedures.

Personnel files contain the training documentation related to the development of each laboratory employee. Included are in-house training, external training certificates, safety training, ethics training, and other materials specific to the analyst. The quality assurance department maintains the training file system.

3.10.1 Training Orientation

The human resources department initiates training orientation for each new employee on the first day of employment. Orientation includes completion of various training checklists (Appendix D). These checklists provide documentation of the orientation after being signed by the new analyst and the trainer and become a part of the employee's permanent training record.

3.10.2 Code of Ethics/Data Integrity Training

It is the intent of TriMatrix Laboratories, Inc. to consistently report data of the highest quality. For this to be possible, analysts are instructed in accordance with the level of data quality desired and are provided with an environment conducive to its achievement. Besides providing the analyst with all necessary supplies and equipment, the work environment is maintained as free from undue pressures as possible. Such pressures may be through internal peer pressure or deadlines, or through external customer complaints or priority requests. It is the responsibility of management to insulate the analyst from such pressures as much as possible. Data quality cannot be compromised without reason and the analyst will not be reprimanded for adhering to established quality protocols in the face of such pressures.

During the orientation with human resources, these policies will be explained and the employee asked to review and sign a Code of Ethics/Data Integrity Policy Agreement (Appendix E). This agreement documents the understanding between management and the new employee concerning management's

position on data quality, sample analysis and data reporting, and the consequences of improper actions. The signed agreement is retained as part of the employee's permanent record.

3.10.3 Document Storage

All essential laboratory documents are stored on the laboratory's intranet drive. During orientation, the new employee is shown how to access these documents and instructed on which ones are required reading. These include the Quality Assurance Manual, Chemical Hygiene Plan, Safety Manual, Employee Handbook, a memo containing instructions on TriMatrix error correction policies and standard operating procedures. Forms are signed documenting that the employee has read and understood these documents.

3.10.4 Demonstrations of Capability (DoC, IDC, CDC)

All analysts and instruments used for sample analysis must complete at least one type of Demonstration of Capability (DoC). Three types of demonstrations exist: a method/instrument DoC, an analyst Initial Demonstration of Capability (IDC) and an analyst Continuing Demonstration of Capability (CDC). All demonstrations of capability are documented, reviewed, and signed in accordance with the TriMatrix SOP for analyst training (GR-10-109). All supporting data necessary to reproduce the DoC, IDC, or CDC must be available. Sample analysis may not begin without the successful completion of an appropriate DoC and submission of all associated paperwork to the Quality Assurance Department.

3.10.4.1 Demonstrations of Method Capability

Prior to the acceptance and institution of any Standard Operating Procedure or the use of any new instrument, a satisfactory demonstration of method/instrument capability study is required. This DoC must be performed on all instruments used for the analysis. This is a one-time study, unless there is a significant

change in the instrument or methodology. This procedure must be successfully completed for all applicable matrices prior to sample analysis. The instrument DoC consists of a Demonstration of Accuracy and a Method Detection Limit Study (MDL); two separate studies demonstrating the instrument's capability of producing sufficiently accurate and sensitive results.

1) Demonstrations of Instrument Capability

For instruments that utilize an initial calibration, an acceptable initial calibration will serve as the demonstration of capability. The low point of the calibration must be at or below the lowest desired reporting limit. The high point defines the calibration range. Any sample with an analyte concentration above the high point in the calibration requires a dilution before quantitation. For procedures not using a calibration curve, seven standards at various concentrations covering the range of the analysis must be analyzed to demonstrate accuracy throughout the range. These standards must be prepared from the same source as that used for calibration. The relative standard deviation of the average recovery must be less than 20% and average percent recovery must be between 95 - 105%. The spreadsheet in Appendix F and the form in Appendix G must be completed to document the accuracy test. Return these completed forms to the Quality Assurance Department.

2) Method Detection Limit study (MDL)

A Method Detection Limit (MDL) study is performed in accordance with TriMatrix SOP GR-10-125. MDL studies must be completed for each matrix-specific preparative and/or analytical technique and must be updated annually or whenever a major change is made to the preparative and/or

analytical technique. The MDL procedure is described in section 3.11.2.

3.10.4.2 Initial Demonstrations of Analyst Capability

After orientation and training, each analyst must complete a successful IDC study. The IDC, unlike the DoC, is not instrument dependent. An IDC must be completed any time a significant change to a procedure occurs. Conduct the IDC study by preparing four replicate blank spikes (for any procedure with a pre-treatment) or four replicate second-source calibration verifications (for any procedure without a pre-treatment) at a concentration in the lower half of the calibration or analytical range. In either case, the spiking standard must be prepared from a source other than that used for calibration. For analyses where a spiking standard is not an option, the acceptable analysis of a single blind PT sample will suffice. Alternatively, the analyst may analyze four replicates of a client sample against four replicates of the same sample analyzed by an experienced analyst for statistical comparison.

Process the four spikes, PT sample, or replicates, following every step in the preparative and/or analytical procedure concurrently or over a period of no more than 72 hours. Enter all four results into the IDC spreadsheet (Appendix H) or all eight replicates into the IDC spreadsheet (Appendix I) as appropriate. The spreadsheet will calculate average percent recovery and relative standard deviation then evaluate against default acceptance criteria (which may need changed to fit the procedure). If all acceptance criteria pass, the analysis of actual samples may begin.

When one or more analytes fail any criterion, the study is unacceptable for the failed analyte. Locate and correct the source of the problem then repeat the study for the failing analyte successfully. If none of the options presented above are possible

(such as with the TCLP pre-treatment), the analyst must perform and submit an acceptable method blank with acceptance being that all analytes are at or below the method detection limit.

When complete, forward the IDC spreadsheet, the NELAC Demonstration of Capability Certification Statement (Appendix J), the Laboratory Training Checklist (Appendix K), the MDL study when necessary (Appendix L), and/or PT results to the Quality Assurance department for review and training documentation.

3.10.4.3 Continuing Demonstrations of Analyst Capability

A Continuing Demonstration of Capability (CDC) is required annually. In addition to the IDC study described in section 3.10.4.2, the CDC may be accomplished by inputting the last four results of an MDL study to the IDC spreadsheet if completed exclusively by the analyst, by inputting four *consecutive* SCV results obtained during the course of routine sample analysis if completed exclusively by the analyst or by exclusively running a PT study analysis successfully.

When complete, forward a copy of all applicable data necessary to reconstruct and validate the study to the Quality Assurance department for training documentation.

3.10.4.4 SOP Revision Checklist

SOPs are periodically reviewed and updated. When an update is released, the appropriate form from Appendix M must be completed to record that the applicable analysts have read, understood and agree to follow the revised SOP.

3.10.5 Continuing Training and Education

TriMatrix Laboratories, Inc. is committed to education and training on a continual basis for employees. There are various ways in which continuing education may occur, including:

- seminars
- cross-training for additional job responsibilities
- retraining
- method and technology updates

3.11 DETECTION LIMITS

The process of quantifying an analyte in an environmental matrix using specific analytical procedures must use detection limits as a point of reference. The three levels of analytical detection are described below.

3.11.1 Instrument Detection Limit - IDL

Most analytical instruments produce a signal even when a blank (matrix without analyte) is analyzed. This signal is referred to as the noise level. The IDL is the analyte concentration required to produce a signal greater than three times the standard deviation of the instrument noise level. The IDL can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. If the instrument does not give a signal for the blank, perform the study using standards at the expected IDL concentration. Each measurement should be performed as though it were a separate analytical sample followed by a rinse and/or any other analytical step normally performed between the analysis of separate samples. Where required by the method (for example, SW-846 method 6010B), IDLs need determined at least every three months or at a project-specific designated frequency and kept with the instrument.

The IDL only defines an instrument's limitations and does not take into consideration sample processing in preparation for the analysis. As such, it may not be used to estimate the method detection limit (Figure 3-7).

An IDL study is required only when specified by the analytical method reference.

The requirement for performing an IDL study does not negate the requirement of a method detection limit (MDL) study.

3.11.2 Method Detection Limit - MDL

Many times there is more to an analysis than a direct analysis such as a digestion, dilution, concentration, chemical treatment, extraction, and cleanup. Additional analysis steps propagate error from the uncertainty associated with each step. Since the method detection limit is defined in terms of error, all steps leading to analysis must be included to calculate the MDL (Figure 3-7).

The MDL is defined as the minimum concentration of a substance that can be detected and reported with 99 percent confidence (statistically) that the value is above zero. The MDL is calculated from spiked blanks which go through the entire sample preparation and analysis scheme. MDL studies are run for aqueous and solid methodologies for every analyte targeted. Although MDL studies are completed for all laboratory determinations, if any result were to be obtained without an associated MDL study, it must be reported as estimated. All calculated MDL values must be verified.

The MDL procedure used at TriMatrix Laboratories references 40 Code of Federal Regulations, Part 136, Appendix B where seven replicate aliquots of laboratory reagent water (for an aqueous methodology) are spiked with every analyte of interest at the estimated minimum practical quantitation limit (PQL). For a solid methodology, an inert substance or empty vessel is spiked. The PQL may be estimated using instrument noise, a series of method blanks, the

instrument calibration, or the preliminary MDL estimation as described in TriMatrix SOP GR-10-125.

It is essential that all sample preparative, cleanup, and analytical steps be included in the MDL study. Calculate MDL study results based on all computations required to achieve the final result in sample-designated units.

To calculate the MDL, input all seven results to the MDL spreadsheet located on the laboratory intranet library. The spreadsheet calculates the MDL by multiplying the standard deviation by 3.143 which is the one-sided t-distribution for seven samples (with six degrees of freedom) for a 99% confidence interval. There must be no zero percent recoveries in the dataset and the concentration spiked must be between 1 and 5 times the MDL value.

Repeat the study at a lower concentration if results in the MDL spreadsheet are flagged “Fails Too Good”. Repeat at a higher concentration if the MDL value is flagged “FAIL”. However, if the spiking concentration is chosen based on a good estimate of the actual MDL, the MDL study should not fail. Re-estimate the actual MDL based on the failed MDL value before repeating the study.

Note: Even if the MDL value appears acceptable, the MDL procedure is not complete until an MDL verification also has been successfully performed.

The MDL verification is accomplished by analysis of a method blank and blank spike. Prepare the blank spike at a concentration between 1 – 4 times the calculated MDL value. If the blank spike response is greater than or equal to three times that found in the method blank, the MDL verification passes and the calculated MDL value is acceptable.

If the blank spike response is less than three times that found in the blank, the MDL value is too low. Repeat the MDL study by estimating the concentration necessary to produce a response equal to or greater than three times the method blank and repeat the verification study. Repeat the MDL verification. Repeat

until the MDL verification is at least three times the method blank. Only the MDL value or MDL verification that passes the MDL verification criterion may be used as the calculated MDL.

Appendix L shows an example of the MDL spreadsheet used to calculate and verify MDL values and practical quantitation limits.

The MDL for all analytes in aqueous and solid methodologies must be determined annually or whenever a significant modification is made to the procedure.

3.11.3 Minimum Practical Quantitation Limit - PQL

The PQL is defined as the minimum concentration of an analyte that can be quantitatively reported (versus qualitatively detected) within specified precision and accuracy limits under normal laboratory operating conditions. (Figure 3-7).

The minimum PQL is the analyte concentration spiked in the MDL study or 3 times the concentration spiked in the MDL verification when the initial MDL value fails. The minimum PQL must be 3-10 times the MDL value.

Note: Practical quantitation limits actually achieved for any given sample analysis will be highly dependent on the matrix and/or required dilutions.

3.12 PROCEDURES FOR ACCEPTING NEW WORK/TESTS

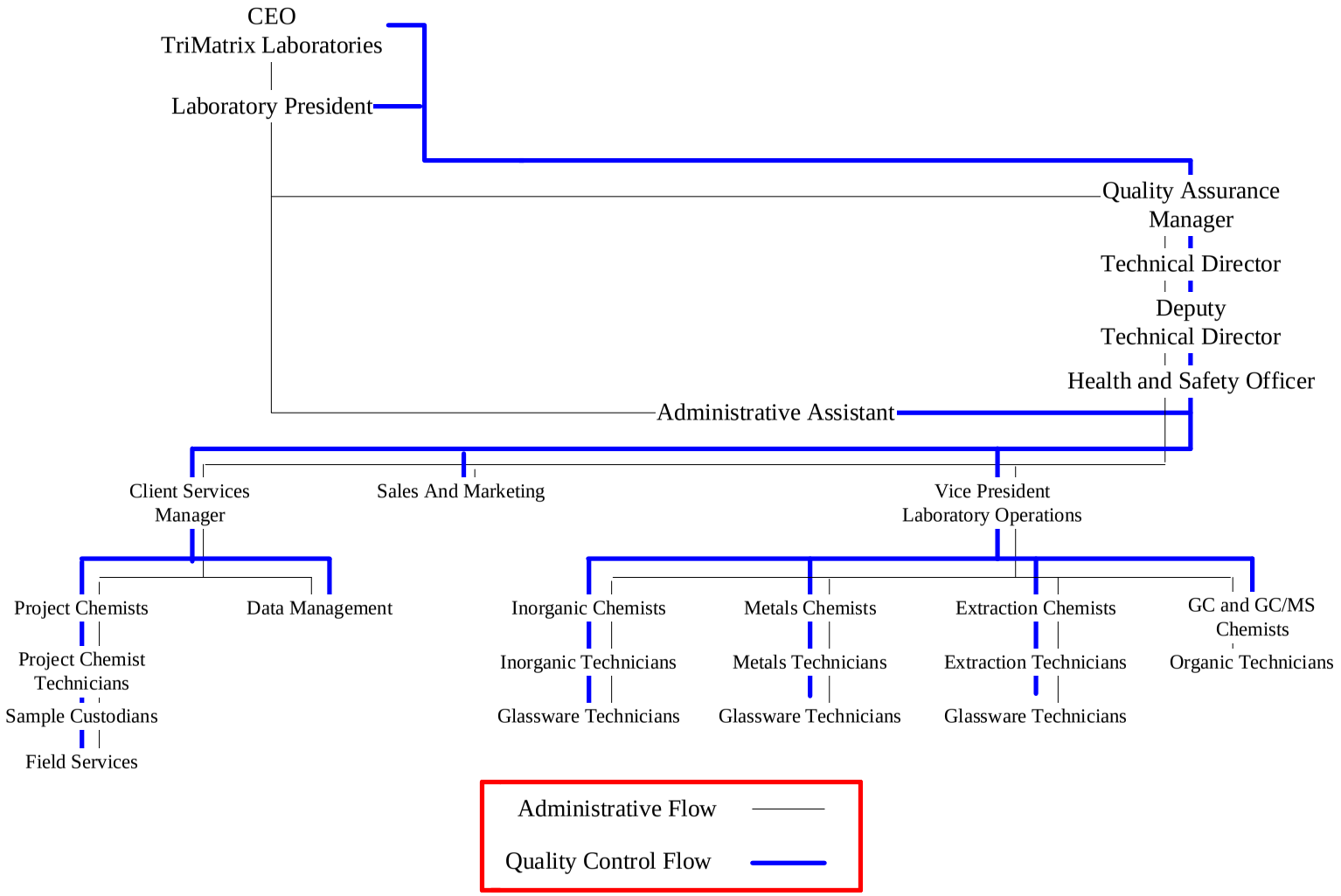
3.12.1 New Test Requests, Development, and Approval

Client Services must submit a request for new analyses to each impacted laboratory area where the request will be formally processed. Evaluation of the request will include the suitability of the analyte for quantitation, availability of existing test methods, instrumentation, capacity, standard materials, etc. The

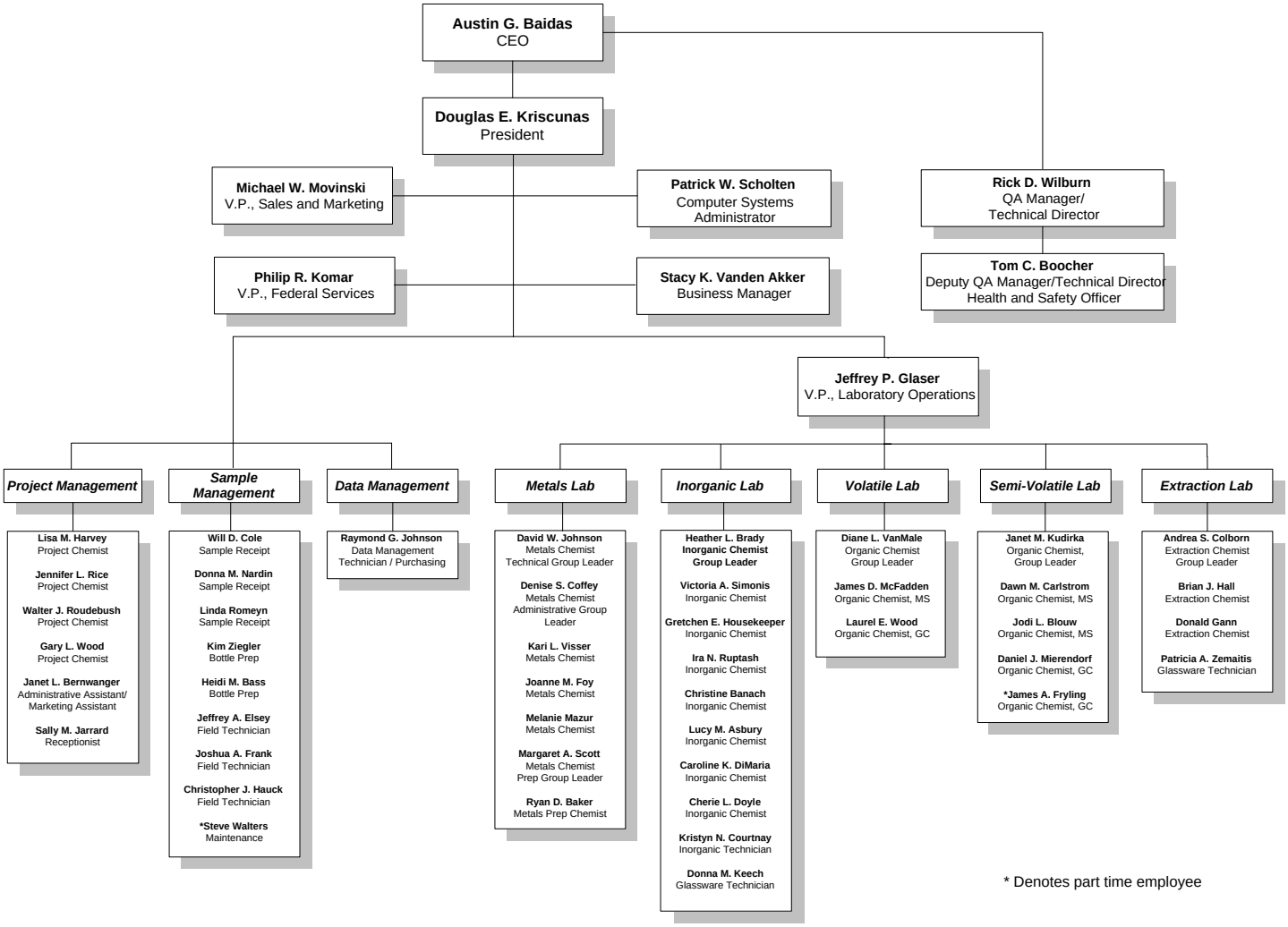
Vice President of Laboratory Operations, Technical Director, and/or Group Leader will provide a prompt response to client services to ensure client needs can be addressed.

All newly developed procedures are reviewed by the laboratory Technical Director and must comply with all requirements outlined in section 3.10.4.

Figure 3-1
Quality Control Chain of Command Flow Chart



TriMatrix Laboratories, Inc.



* Denotes part time employee

Figure 3-3
RELATIONSHIPS
Management to Technical Services

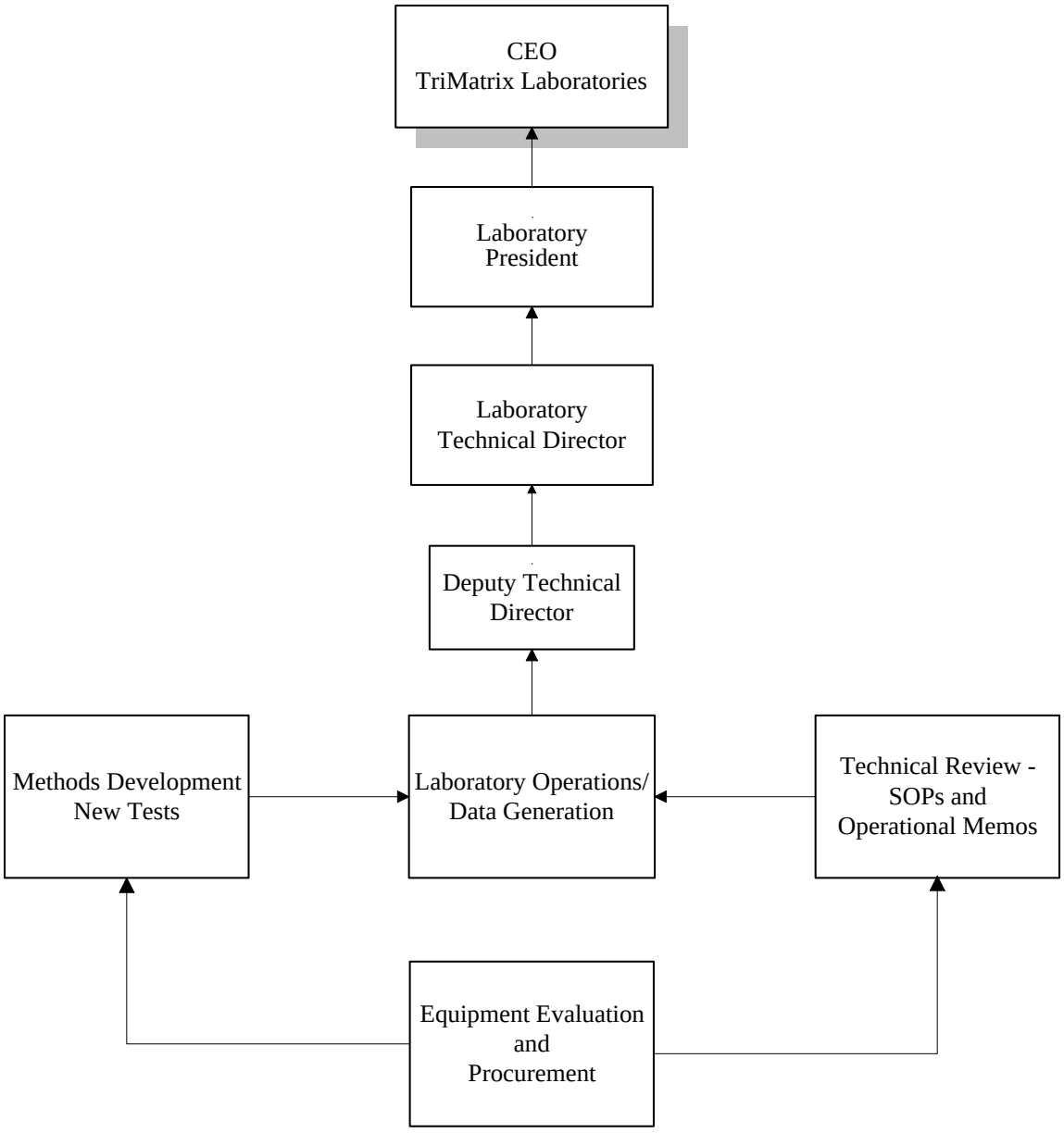


Figure 3-4
RELATIONSHIPS
Management to Support Services

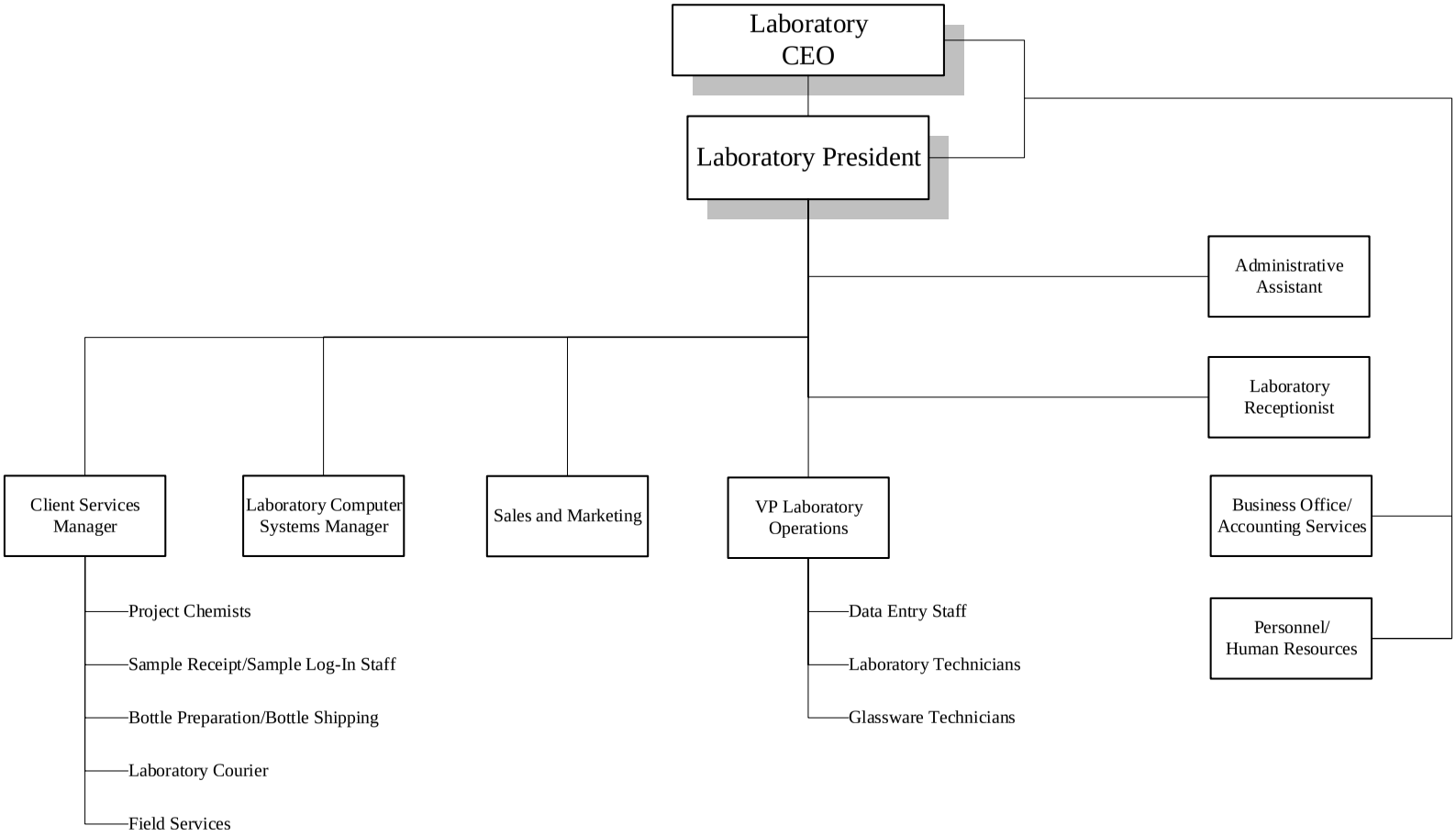


Figure 3-5
RELATIONSHIPS
Management to Quality System

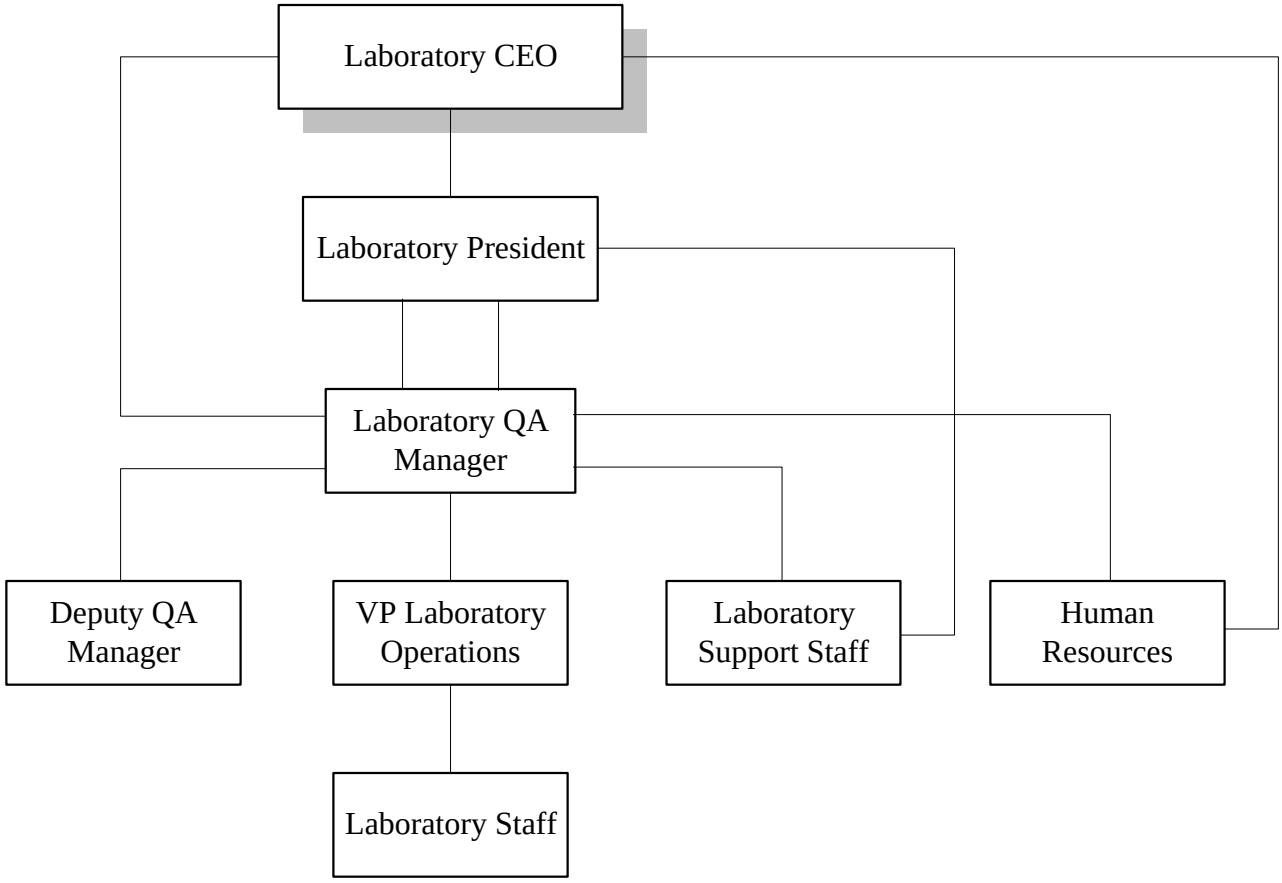


Figure 3-6
Laboratory Layout/Diagram

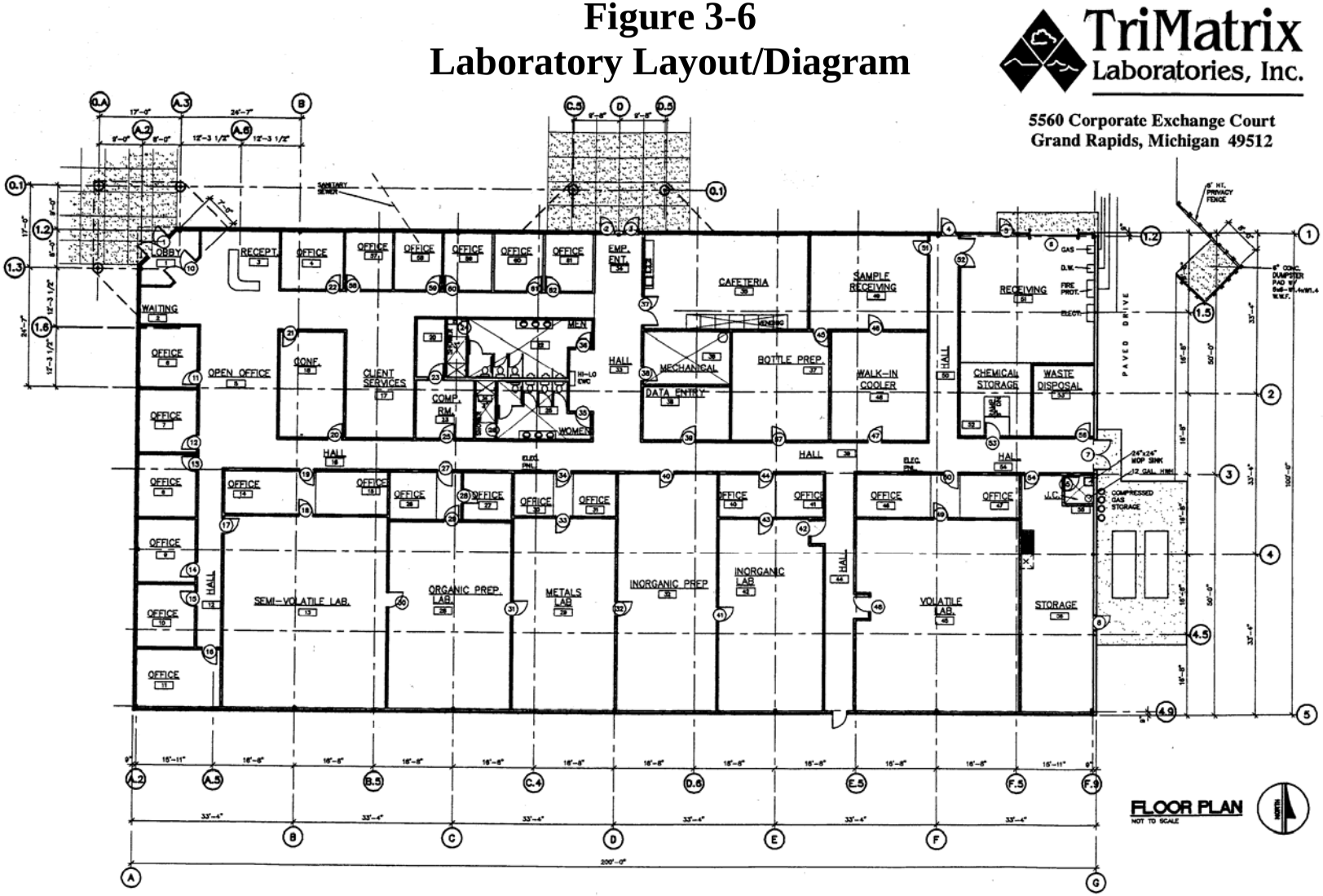
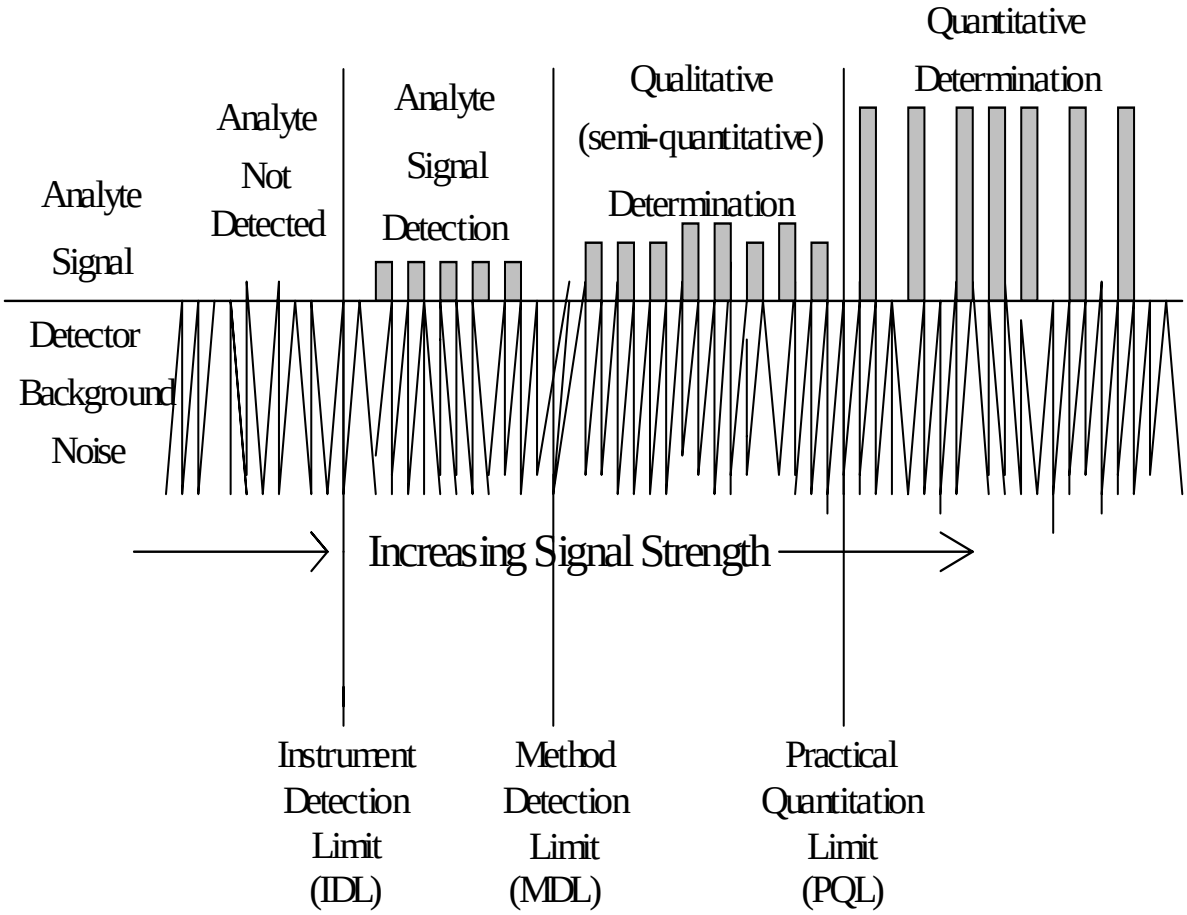


Figure 3-7
Regions of Analyte Signal



4.0 QUALITY CONTROL

4.1 DOCUMENT CONTROL AND MAINTENANCE

4.1.1 Procedures for the Control and Maintenance of Documentation

Documents utilized in the quality system are subject to strict control regarding their creation, revision, approval, use, and distribution. This applies to documents generated both internally, and those received from outside sources. Obsolete documents that are retained in circulation for either legal or knowledge preservation purposes are marked as "obsolete". The structure of the documentation used in the TriMatrix quality system is presented in Figure 4-1.

4.1.1.1 Internal Documentation

Examples of internal documentation include Standard Operating Procedures, the Quality Assurance Manual, miscellaneous forms, and logbooks. All documents must be reviewed and approved by one or more senior staff prior to their use. All documents will print with both the file name and revision number. Where possible, the document will contain the TriMatrix logo. All logbooks must be bound and paginated.

All approved documents are stored on the laboratory intranet read only drive designated as "Library." Document control is maintained through the use of the laboratory computer network. By maintaining only the current version of an approved document on the Library drive, document control and security is maintained. This procedure provides immediate access to the latest revision of all documents.

Document revisions may be made by any applicable, qualified, laboratory employee. Minor document revisions, such as those required to a Standard Operating Procedure, may be made by hand.

All hand amendments must be legible, dated and initialed, and recorded in ink. All hand amendments must be approved by, and distributed through, the Quality Assurance Department. Hand amendments will be incorporated into the next revision of the document. Hand amendments cannot be used for major document revisions. Extensive revisions require a formal document update.

Some documents, such as the QAM, require periodic reviews. The QAM is reviewed annually and updated as necessary.

Completed logbooks are numbered, scanned, and the resulting .pdf file stored on the Library drive.

4.1.1.2 External Documentation

Examples of external documentation include regulations, analytical methods, QAPPs, and client required standards. These documents are maintained by the quality assurance department. When possible, the documents are stored electronically on the Library drive. Instrument manuals are controlled by the individual laboratory areas.

4.1.2 Traceability of Measurements/Documentation Requirements

A properly designed and implemented documentation protocol will assure that all information presented in an analytical report can be traced back to its point of origin. The documentation protocol must also provide for traceability of non-reported information used to provide supporting value to the analytical report. These items include but are not limited to: stock standard records, test calibration records, data reduction and validation activities, sample custody, facilities monitoring, and final data reporting.

A more detailed review of the documentation procedure and traceability of information is presented in the following sections.

4.1.3 Paperwork/Information Flow

As displayed in Figure 4-2, document flow remains constant regardless of the quality control requirements of the sample. The general axiom is, a COC procedure will fail without a pre-existing scheme of rigid documentation control available. The records trace can provide for the following:

- Answers to questions of analytical integrity
- Assistance in finding and solving random and systematic problems
- Assistance in preventing long term degradation of the analytical process
- Assistance in ensuring continuity of analytical effort despite personnel and mechanical changes

The following subsections identify and describe the procedures followed, and the corresponding documents generated, from project initiation through completion.

4.1.3.1 Project Initiation

All samples or sample groups entering the analytical process must be accompanied by the appropriate documentation. This documentation is necessary to define the analytical goals and project objectives. Information concerning analytes, reporting limits, and reporting formats must be provided. An inventory of required sample containers must be prepared for each sampling event. This inventory is documented using the Container Packing List (Appendix N).

All projects are initiated through the LIMS system. All documents created during the project initiation phase are maintained and archived to the client filing system.

4.1.3.2 Sample Receipt/Examination

The receipt of all sample shipping coolers (empty or full) will be documented in the Sample Receipt Record Logbook (Appendix O). This logbook documents the delivery method, date and time, number of coolers received, client, and the name of the TriMatrix employee who received the cooler. This information is entered into the logbook immediately after drop-off.

Observations on the receipt of each sample delivery group, including sample temperatures, are documented on the "Sample Receiving/Log-in Checklist" (Appendix P). This form was designed in a step-by-step format to walk the log-in technician through all the steps required when receiving and logging-in samples. A supplemental "Sample Receiving/Log-in Checklist Additional Cooler Information" form is available when receiving projects consisting of more than four coolers (Appendix Q).

Additional forms to document sample preservation, "Sample Preservation Verification Form" (Appendix R), and non-conformances, "Sample Receiving Non-Conformance Report" (Appendix S), are also completed.

4.1.3.3 Sample Log-In

During log-in, a series of computer entry functions are performed in an effort to document and validate the log-in process. The remainder of the Checklist is also used to record the completion of the various steps that must be followed when logging samples into the LIMS system. Once complete, bottle tags are produced and a Work Order generated (Figure 4.3 and Appendix T). The log-in technician will initiate a project or submittal file for each sample delivery group received. This file is labeled with the LIMS system generated project-submittal sequence, and will contain all documents associated with the sample receiving/sample log-in process. These documents will include: all external chain-of-custody forms, sample preservation records, shipping records, any

client correspondence, and a copy of the actual log for each submittal. Upon completion of the analytical process, the project file becomes part of the permanent record of each project.

4.1.3.4 Worklists/Benchsheets

The worklists and benchsheets produced by the LIMS system are designed to provide the analyst with essential project information. This information not only includes client/project specifications, but also provides an avenue for communication of test specifications and parameter expiration dates and times. This up-front information enables the analyst to make vital decisions in their analytical scheme, and helps to minimize problems after samples are analyzed.

Examples of a laboratory worklists and benchsheets are presented in Appendix U.

4.1.3.5 Management Reports

Several reports are provided within the TriMatrix Laboratory system to help monitor operational conditions of the laboratory. These reports include: workload reports, on-time reports, and aging logs.

The flow of information from these various reports is geared to a variety of personnel within the management structure of the laboratory, and to specific persons outside the laboratory. Information is generally provided to employees' external of the laboratory for corporate management decisions or in providing information to a particular client about their project.

Examples of management reports are presented in Appendix V.

4.1.3.6 Quality Assurance Reports

Quality assurance reports play a vital role in the management of the quality system. Quality systems must be closely scrutinized in order to monitor, maintain, adjust, and add, procedures or systems to meet existing and new QA objectives of the laboratory.

Several quality assurance reports are created in this effort. These reports serve different functions and are designed to inform the ultimate user. In the case of a client/invoice report, the quality assurance data is presented to facilitate the objectives of the project requirements from data assessment through full 3rd party data validation.

Quality control reports are also used within TriMatrix to monitor the analytical process and to provide a means by which the analytical process can be viewed over time. Examples of efforts available for this monitoring process are presented in Appendix W.

Quality control reports are used extensively by the laboratory to access the analytical process. Many of these reports are utilized daily to monitor, for example – method accuracy, precision, completeness, and to provide the means for overall data assessment at the batch level. All QC reports are created through the LIMS system.

4.1.3.7 Project Files

The project file is the comprehensive record of every analytical project completed at TriMatrix. Project files are stored in secure filing cabinets. Items typically retained in a project file include:

- Initial project report/analysis plan/proposal
- All correspondence or documents mailed or received with the samples
- Written record of client phone conversations

- All sample receiving/log-in forms
- Chain-of-custody forms
- Laboratory worksheets
- Copy of the invoice

To save paper and file space, electronic, rather than paper, copies of final reports are typically retained, and can be regenerated on demand.

By default, project files are stored on-site for 1 year, followed by off-site storage at a secured limited access facility for an additional 6 years. Length of storage requirements are determined on a client/project specific basis. If the ownership of the laboratory changes, record storage will become the responsibility of the new owner. In the event the laboratory was to go out of business, each client will be contacted for instructions on record disposition. Client records will be transferred or destroyed as instructed.

4.1.3.8 Quality Control Documents

A) Instrument Logbooks

Two different instrument logbooks are maintained, an instrument run-log and an instrument maintenance log. Each log plays an important role in the documentation of daily instrument activities.

The Instrument Run Logbook is used to document all analytical determinations of a designated instrument. These determinations include not only sample analyses, but also recordings of all calibration and calibration runs, quality control analyses, and where applicable, any instrument tuning activities.

The Instrument Run Logbook also provides a chronology of each day's analyses. This chronology plays an important role in the data validation process. All run logs are identified by instrument

manufacturer name, model number, serial number, and the starting and ending dates encompassed. All completed run logs are issued document control numbers, inventoried, and properly archived.

The Instrument Maintenance Log is used to document instrument maintenance procedures, repairs, or modifications. All activities are documented by recording what was done, by whom, and why.

All completed maintenance logs are identified by instrument manufacturer name and model number, instrument serial number, and the dates encompassed. All maintenance logs are issued document control numbers, inventoried, and properly archived.

B) Controlled Temperature Units (CTU)

Each oven and incubator used for sample processing, and all cold sample and standard storage devices have their temperatures monitored and recorded on a daily basis. Within each CTU is a certified thermometer. Additionally, each CTU used for sample storage, and incubators used for BOD and bacteriological incubation, have their weekend temperature monitored via electronic data loggers. The calibration of liquid and digital thermometers is verified annually.

All temperature readings and thermometer calibrations are recorded in a CTU Logbook. This logbook contains a page for each unit with detailed information on unit identification, serial number, laboratory location, and designated operating temperature. All CTU logbooks are issued document control numbers, inventoried, and properly archived. An example of a Controlled Temperature Log is presented in Appendix X.

C) Balance Monitoring

Each analytical and top loading balance used at TriMatrix is monitored for accuracy. All daily checks are recorded in a TriMatrix Balance Log (Appendix Y). All balance logbooks are issued document control numbers, inventoried, and properly archived.

D) Standard and Reagent Preparation Logbooks

All standards and calibration solutions used at TriMatrix are prepared, when possible, from reagents or solutions traceable to national standards. Whether a stock, an intermediate, or a working concentration, each reagent and standard solution is traceable to its origin. This is accomplished using the laboratory's LIMS system and/or a Standard Preparation Logbook (Appendix Z).

Information available on each standard includes:

- The analyte or analytes contained in the standard
- The concentration
- The solvent used to prepare the standard
- The preservative (i.e., nitric acid)
- The date of preparation
- Initials of the preparer
- The expiration date
- The unique identification number

Unique identification numbers are generated by the LIMS system and/or a book, page, and line number system. All standard and reagent preparation logbooks are issued document control numbers, inventoried, and properly archived.

E) Pipet Logs

All autopipetors utilized for the delivery of standard solutions, diluents, and reagents, are periodically checked for delivery

accuracy. Because these pipetors contain mechanical parts they are subject to inaccuracies if not properly maintained and calibrated.

Daily calibrations (for pipets used to prepare standards), and weekly calibrations (for pipets used to prepare quality control samples) are recorded in a Pipet Calibration Logbook (Appendix AA). Each log is identified by manufacturer name and model number, the pipetor serial number (if available), and the starting and ending dates encompassed. All complete pipet logbooks are assigned document control numbers, inventoried, and properly archived.

4.1.3.9 Confidentiality and Proprietary Rights

Since significant amounts of information regarding the details of a client's operations are received in the laboratory, it is essential that strict confidentiality be maintained in the handling of all client information. Client data is protected in locked filing cabinets and in limited access computer files. Under no circumstances is the name of a client, or any information regarding that client, revealed to another client or to a regulatory agency without the client's written permission, under penalty of employment termination.

Any details of a client's operations that have necessarily been revealed to the laboratory for testing purposes are considered as proprietary and protected by patents, copyrights, infringement laws, or other legal constraints against disclosure.

4.1.3.10 Document Storage and Traceability

Archiving of information at TriMatrix has been designed to meet both short-term and long-term storage needs. Archives are maintained for a wide variety of data and documentation. These archives can be categorized into two main groups, a) document

archives (physical documents) and b) electronic archives (data files). Table 1 illustrates the current TriMatrix archival systems, their location, and duration.

Documentation records or logs are maintained for all archival systems to aid in the quick retrieval of information. Extended archival periods or special procedures are also in place for some projects and clients.

4.1.4 Standard Operating Procedures (SOPs)

Many of the methods published today by various agencies provide only general guidance in performing an analytical determination. A significant part of the variability observed in analytical data is in large part due to minor variations in the analytical process. A Standard Operating Procedure is a guide that clearly defines the exact steps to be followed while performing a procedure. The delineation of these exact steps in an SOP will improve the analytical conditions, which in turn will help the overall reproduction of analytical data.

4.1.4.1 SOP Categories

SOPs are written for nearly all laboratory activities. The categories utilized in the organization of SOPs are presented in Table 2.

4.1.4.2 SOP Development, Formatting, and Review

All standard operating procedures are developed and written to the specifications outlined in the TriMatrix guidelines for the preparation of a SOP. These guidelines are presented in SOP format and have been designed to accommodate analytical tests, non-tests such as extractions or digestions, and documentation or non-analytical activities. The guidelines were developed from both USEPA and ASTM protocols for the creation of standard operating procedures.

All SOPs developed by TriMatrix are subject to a review process where signatures or approvals are required from the appropriate area manager, the quality assurance department, and the Vice President of Laboratory Operations. In addition to this overall approval process, each page of an SOP is individually approved by both the laboratory area and quality assurance department (Appendix AB).

SOPs are reviewed and updated as necessary. Minor modifications can be hand edited on the SOP. These modifications must be made through the Quality Assurance Department. Depending on the modification, distribution of the edited SOP (as described below) may or may not be required. All minor modifications will be incorporated into the next revision of the SOP. Major modifications may require the SOP to undergo an immediate formal update.

4.1.4.3 SOP Documentation and Control

All SOPs are assigned a unique procedure identifier. Other information included in every SOP is the effective date, revision number, information on the author, total number of pages, and identification of any individual page revisions.

All original, approved paper copies of TriMatrix SOPs are controlled by the Quality Assurance department. Approved SOPs are scanned and stored on the network Library drive. This drive is accessible to all laboratory personnel. Copies of all outdated SOPs are destroyed (or marked as obsolete), and the scanned copy of the SOP is removed from the Library drive.

4.1.5 LIMS

TriMatrix utilizes the Element LIMS system developed by Promium Corporation. This system controls all aspects of laboratory operations. The main functions of the LIMS system are:

- Project Management
- Sample Management
- Work Scheduling and Management
- Data Entry, Verification, and Approval
- Report Generation
- Invoicing

4.2 SAMPLE CONTROL, FLOW, AND STORAGE

Presented in the following section is a description of the policies and procedures that were developed to identify, monitor, and document the flow of samples through the Laboratory. A flow chart depicting this process is presented in Figure 4-3.

4.2.1 Project Initiation

When samples are received at TriMatrix, the necessary information that will direct the analytical scheme has already been developed and implemented within the project initiation/project management process. This process starts with the award of a contract or proposal, a client request, or a pre-scheduled sampling event. The basic steps and supporting documentation involved in the project initiation process begins with the gathering of project information, communications with all affected laboratory areas, and the input of required project related data into the LIMS system. All requests for analytical work are reviewed by the project chemist, and when necessary, applicable management staff to verify the laboratory has the capability to perform the requested tests and meet the requested turnaround times. Requests for changes to in-progress projects must be made with the appropriate project chemist. Changes in methodology will typically require client approval. The project chemist will be responsible for coordinating all requests for changes with the impacted laboratory areas. All approved changes will be formally made via the laboratory's LIMS system, thus continuing the normal paperwork flow.

TriMatrix uses test methods that meet the needs of the client and are appropriate for the tests undertaken. Methods published in international, regional, or national standards are used. TriMatrix uses the latest valid edition of a method unless it is not appropriate or possible to do so. All analytical procedures are documented in SOPs supplemented with additional details to ensure consistent application.

When not specified by the client, TriMatrix will select appropriate methods published either in international, regional, or national standards, by reputable technical organizations, in relevant scientific journals, or as specified by the equipment manufacturer. Laboratory developed methods (or methods adopted by the laboratory) are also used when appropriate for the intended use, and have been validated following the various initial demonstration of capability procedures. When specified by the client, TriMatrix will inform the client if the specified method is considered inappropriate, or out of date.

Routine projects include samples matrixes and analyses that are continuously processed by TriMatrix. Non-routine projects are those that require special analyses, include parameters not routinely run by the laboratory, posses unique holding times, or require expedited turnaround. Non-routine projects will require approval from all affected laboratory areas. This approval process is communicated in several different ways, including everything from the signing of a quality assurance project plan (QAPP) to the transmission and receipt of an electronic mail message.

Occasionally, a portion of a project may involve an analytical methodology not currently possible at TriMatrix. When requested by the client, samples for analyses outside the analytical scope of TriMatrix can be subcontracted to another laboratory. It is preferred that the client specify the subcontract laboratory. When the subcontract lab is not specified by the client TriMatrix will only subcontract to laboratories that are NELAP accredited, or ISO-17025 certified, for the specific method of interest. Client specific program requirements will take precedence over this rule. A registry of subcontract

laboratories used by TriMatrix will be maintained, documenting their NELAP accreditation or ISO-17025 certification.

The development of a project within the laboratory also involves the preparation and shipment of sample collection materials and containers. The processes involved in the procurement, preparation, and shipment of sample collection materials and containers are presented in the sections below.

4.2.1.1 Sample Containers and Materials Procurement

TriMatrix utilizes only virgin bottle ware for all sample collection kits. All containers are purchased pre-cleaned and come with Certificate's of Analyses.

Specific projects or programs may require the laboratory to verify the cleanliness of the containers. When this is required specific lots will be sequestered from the container vendor. Each lot will be tested to verify the containers meet the project or program requirements. Only containers whose cleanliness has been verified will be used for the project.

4.2.1.2 Preparation of Containers

All sample containers utilized for the collection and preservation of environmental samples are prepared by the bottle prep group. The staff members of this group focus their activities exclusively in the area of sample container procurement, preparation, and shipping. Project sample container kits are requested using the Container Packing List, presented in Appendix N.

4.2.1.3 Sample Container Shipment

When all containers have been assembled as requested on the Master Bottle Packing List, the bottles are packaged and placed into one or more shipping coolers. 40 mL glass vials are packed in

small bubble pack bags. An attempt is made to organize each sample cooler to help minimize time spent in the field. When possible this is accomplished by packing bottles together by sample point. When complete, each shipping container will be inspected by a project chemist to verify its accuracy. Documentation of this inspection is made on the bottle packing list. A copy of the bottle packing list is placed in each cooler.

Also provided in each cooler is a set of instructions or comments about the containers, material safety data sheets for all chemical preservatives present, a return address label, an external COC form, and if required, TriMatrix sample bottle custody seals. All materials are packaged in a waterproof zip-lock bag. Examples of these additional materials are presented in Appendix AC.

Packing is now added to the cooler and the shipping container is sealed. When requested, signed TriMatrix custody seals can also be applied to the outgoing cooler.

4.2.1.4 Sample Receipt

The receipt of all sample shipping coolers (empty or full) will be documented in the Sample Receipt Record logbook (Appendix O). This logbook documents the delivery method, date, and time, the number of coolers received, the client, and the name of the TriMatrix employee who received the cooler. This information is entered into the logbook immediately after drop-off.

As soon as possible after the shipping cooler is received and all available information entered into the Sample Receipt Record, cooler inspection and sample temperature determination occurs. The observations associated with this step by step process are recorded on the “Sample Receiving/Log-in Checklist” (Appendix P). This Checklist must be completed for all samples for a given project received on a given day. A supplemental “Sample

Receiving/Log-in Checklist Additional Cooler Information" form is available when receiving projects consisting of more than four coolers (Appendix Q).

IMPORTANT: When initiating each Checklist, make sure the Receipt Log Page/Line number from the Sample Receipt Record logbook is recorded at the top of each Checklist. This ties the receipt of the sample coolers in with the samples themselves.

Record the cooler number of the first cooler and the current time. Observe and record the type of coolant used. When possible, the sample temperature of three random samples (locations representative of the coolant present in the cooler) will be taken. If a temperature blank was received, measure and record this temperature as well.

Sample temperatures are recorded using a calibrated infrared thermometer. Because this type of thermometer is actually measuring the temperature of the container, it is critical that the temperature is taken as the sample is removed from the cooler. The container warms up quickly and any other method will result in an incorrect reading. Do not dry the container prior to measuring the temperature. Containers wet from melt water are preferred to dry containers. Record the temperature values on the Checklist. Report all temperatures to the nearest 0.1° C. If a correction factor is necessary, record the correction factor and the corrected temperature on the Checklist. If any temperature exceeds 4° C, average the three sample results and also report the average. If the average temperature of the three samples, or the temperature of the temperature blank exceeds the 6° C required by most regulatory bodies, it must be noted on the Checklist.

If sample receipt and temperature determination occurs outside of normal business hours, place received coolers in the walk-in for

storage. Assemble all the paperwork, and place it in the after-hours basket. The remainder of the receiving process will be performed by a log-in technician during the next business day.

4.2.1.5 Sample Examination

Samples received at TriMatrix are required to be accompanied by a TriMatrix Laboratory Chain-of-Custody (COC) form (Appendix AD). For samples received without this form, the log-in technician will initiate the COC process. Should a submittal or delivery group be identified as an internal COC project, the log-in technician will initiate the procedures outlined in section 4.2.2 B.

The remainder of page 1 of the Checklist is now filled in. Observations are made on the accuracy of the COC and the condition of the sample containers. Many of the aqueous samples received have been subjected to some form of chemical preservation. Verification of the preservation is required; however, depending on the analysis this verification may not occur during the log-in process. The “Sample Preservation Verification Form” (Appendix R) specifies what container types will have their preservation verified during log-in. The form also specifies what container types can have an incorrect preservation adjusted. Preservation verification is performed via a pH check using calibrated pH strips. Determine the correct reading against the color chart on the pH strip container. Document the pH found on the Sample Preservation Verification Form. Use only the pH strips located in the log-in area whose calibration has been verified and recorded in the pH Strip Calibration Logbook (Appendix AE).

Should a) the result of any preservation check indicate that the sample has not been properly preserved in the field (or the buffering capacity of the sample has resulted in an unacceptable sample pH at receipt) or b) there is insufficient evidence indicating that other needed preservation reagents (e.g., Zinc Acetate for

Sulfides) have been added, then a Sample Receiving Non-Conformance Report (Appendix S) is to be initiated and the project chemist contacted as soon as possible. In some instances, the holding time of such samples may be shortened. No preservation adjustment may be made without approval from a project chemist.

IMPORTANT: Shaded boxes on the Checklist indicate an out-of-control situation. The selection of any shaded box during the completion of this form also requires the initiation of the Sample Receiving Non-Conformance Report.

Collect all paperwork and deliver to the appropriate project chemist for review. Any issues that require contact with the client for resolution will be made in a timely manner. The project chemist will create a submittal and return the paperwork. Once the project chemist returns the paperwork, page 2 of the Checklist can be completed, and the samples logged into the LIMS system.

4.2.1.6 Sample Log-In

All samples received by TriMatrix are logged into the LIMS system. The log-in procedure assigns a unique TriMatrix sample number to each sample, allowing samples to be tracked, data stored, and quality control associated for any sequence of events during a particular analytical period. The primary steps involved in the sample log-in process are presented below.

4.2.1.7 Sample Splitting

In the event that TriMatrix is unable to provide sample bottles, or circumstances prevent the splitting of samples in the field, the log-in technician can provide sample splitting services; however, sample splitting will typically be performed by a laboratory area chemist. These services include taking the sample as received and

sub-sampling it into the appropriate bottle with the preservative requirements as set forward in Appendix AF – Sample Collection Guidelines Bottle and Preservative Requirements. Sample splitting will only be performed when instructed by a laboratory project chemist with client approval.

A. Sample Splitting-Water Samples

Laboratory area managers will be consulted in order to insure that sufficient volume will be available to all areas of the lab after splitting. In the event that sufficient volume does not exist, the Project Chemist will be immediately notified for resolution.

When a bulk sample arrives for both organic and inorganic analysis, and sufficient sample exists, the organic aliquots will be removed first. The remainder of the sample will be transferred to properly preserved containers for each inorganic analyses.

B. Sample Splitting-Solid Samples

When solid samples, such as sediment or soil, are to be received at TriMatrix, every attempt will be made by the Project Chemist and field sampling personnel to insure that two samples are provided as replicates for the appropriate tests. One of these samples will be assigned to the organic area and the other to the inorganic area. If only one sample is received and if organic analyses are required, the organic aliquots will be removed first. Prior to sub-sampling, solid samples will be made homogeneous by either one or all of the following manners:

- Stirring
- Grinding
- Particle separation (sieving)

The laboratory area manager is responsible for deciding how a solid sample will be split. Problems or concerns that may arise on splitting a solid sample will be addressed by the Project Chemist and Laboratory Area Manager. After the organic portions have been removed or split, the remaining sample will be provided to the inorganic facilities for any further splitting.

4.2.1.8 Sample Distribution

All samples received at TriMatrix are labeled by the log-in technician. These labels include both the necessary information for proper identification, and information on any potential for flammability, reactivity, contact, or health based risks.

After completing the log-in process of all the various samples connected with a particular project, the log-in technician will store the samples in the correct Controlled Temperature Unit (CTU).

- Routine Water and Solid Samples: Samples that require refrigeration will be stored in the CTU designated for all routine water and soil samples.

- Routine Volatile Water and Solid Samples: All volatile samples are stored in designated VOA CTUs. Volatile water and soil samples are segregated and stored separately. No other sample types are stored in the VOA CTUs.

  All CTUs used for VOA sample storage will also contain a storage blank. The storage blank is a preserved 40 mL VOA vial filled with deionized/distilled water. The storage blank is replaced and analyzed on a weekly basis. If positive results are observed for any target analyte above the laboratory's minimum reporting limit, all samples stored concurrently in the CTU must be evaluated for possible contamination. All sample results

within 5 times the level quantitated in the storage blank must be qualified as estimated.

- Odoriferous and Hazardous Samples: Stored separately in a special vented facility. If volatile analyses are to be performed, samples are stored under refrigeration. Samples are identified to the laboratory by means of a narrative within the LIMS System.

All samples that are involved as physical evidence in a legal procedure or simply identified as Chain-of-Custody will be handled under COC procedural safeguards.

4.2.2 Chain-of-Custody (COC)

All samples received by the laboratory require some form of chain-of-custody (COC). TriMatrix practices two levels of COC, external and internal. The degree of custody tracking and documentation is driven by the final deposition of the laboratory data. Generally, if samples and their analytical results are subject to involvement as physical evidence or in a legal procedure, both external and internal custody procedures will be followed. If samples or results are not subject to legal procedures, only external COC procedures will be followed. A description of these two custody scenarios is presented as follows:

A. External COC

Samples only requiring external COC will have their custody tracked from sample collection to delivery at the laboratory. This process involves the completion of a TriMatrix external COC form, as presented in Appendix AD. This form accompanies the sample containers prepared by TriMatrix to the sample collection site. Any sample or submittal received at the laboratory without a TriMatrix external COC form will initiate a process where the log-in technician will complete the necessary external COC forms for carrier sign-off.

For document control purposes, all external COC forms have a unique identification number.

B. Internal COC

Samples requiring strict COC will initiate the process by which all events or periods of sample handling will require a traceable document protocol.

The internal COC process involves the completion of a TriMatrix internal COC form for all phases of the analytical process. This includes sample extractions, distillations, digestions, analyses, and disposal. An example of the TriMatrix internal COC form is presented in Appendix AG. All internal COC forms are maintained in a series of submittal or delivery group folders.

C. Sample Security

All samples, whether under external or internal COC protocols, are maintained in a limited access secured area. This level of security is applied to all phases of the analytical process from sample log-in to final sample disposal.

D. Sample Disposal

All samples received are subject to disposal as waste once tested and discarded. Three general categories discarded samples fall into are the following:

1. A sample may be returned to the client (specifically, if highly contaminated).

2. Too contaminated for municipal disposal and must be disposed of as waste through a hazardous waste facility.

3. Inert, uncontaminated, and nontoxic samples in accordance with municipal waste regulations may be disposed of in the municipal dumpster and/or the laboratory waste room sink leading to the city sewer.

4.2.3 General Laboratory Security

Access to the laboratory is handled in a secure fashion, with access restricted to authorized personnel only. All laboratory areas including sample storage, sample container preparation, analytical laboratories, sample preparation, sample disposal, analytical documents, and data files are restricted. Non-authorized personnel may enter these areas only when escorted by a laboratory staff member.

It is the responsibility of all laboratory staff members to insure that the rules of restricted access are followed and maintained at all times.

4.3 CALIBRATION AND CALIBRATION VERIFICATION

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment used in conducting laboratory analyses. Calibration of the instruments and equipment is performed prior to each use or on a scheduled periodic basis.

Calibration of laboratory instruments and equipment is performed to verify that the analysis portion of the testing process is functioning properly and at the required sensitivity. A calibration section included in each analytical SOP covers the frequency, stability, and specific calibration steps, based on analytical method requirements and instrument or equipment manufacturer's recommendations.

Initial calibration is performed using standards of certified value to establish the linear range of the analysis for the analytes of interest. Each calibration curve is verified using a Second Source Calibration Verification Standard (SCV) prepared from a source dissimilar to that used in the preparation of the calibration standards. The calibration is also verified at the beginning and during the analytical sequence, using a standard prepared from the same source as that used in the initial calibration.

Calibration activities are divided into three categories:

Field Equipment (section 4.3.1)

Laboratory Instrumentation (section 4.3.2)
Laboratory Equipment (section 4.3.3)

4.3.1 Field Equipment

Perform daily calibration checks on field equipment prior to the commencement of any field analyses. Follow the written calibration procedure for each individual piece of field equipment. The equipment is held out of service until repairs and successful recalibration occurs. A summary table of all calibration procedures and frequencies is included (Table 3).

4.3.2 Laboratory Instrumentation

Calibration of laboratory instruments is based on approved SOPs. Records of calibration, repairs, or replacement are filed and maintained by the designated laboratory analyst. These records are filed at the location where the work is performed and are subject to QA audit. For all instruments, the laboratory maintains in-house spare parts or service contracts with vendors. A summary table of all calibration procedures and frequencies is included (Table 4). Flag any instrument that does not pass daily requirements. Hold the instrument out of service until repair or successful recalibration occurs.

4.3.2.1 Inorganic/Classical Chemistries

Inorganics analysis utilizes a wide variety of wet-chemical procedures and instruments. Calibration steps may vary depending on the specific analytical method being utilized. However, certain general principles of calibration apply to all inorganics testing. Every analytical method requires calibration or calibration verification prior to sample analysis. Using a group of certified standards, the linear range is defined. The calibration is checked on a continuing basis to be certain that the method is within the required test parameters. All inorganic calibrations must meet the specific requirements described below unless required otherwise by the method or manufacturer.

The instrumentation used to conduct these analyses is calibrated using calibration standards prepared by dilution of stock solutions. One standard is prepared at the reporting limit of the analyte of interest while the other standards bracket the concentration range of the samples. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve.

An SCV originating from a dissimilar stock solution than that used for preparation of the calibration standards is prepared and analyzed. Continuing Calibration Verification blanks and standards (same source as that used in the initial calibration curve) are run at the beginning, and periodically, throughout the analytical sequence, typically after every 10 analyses. The value of the continuing calibration standard concentration must agree within the method specified criteria; generally ±15 percent of the initial value or the appropriate corrective action is taken. Corrective action may include recalibrating the instrument and must include reanalyzing the previous 10 samples.

4.3.2.2 AAS/ICP/MS Emission Systems

The atomic absorption spectrophotometer (AAS), inductively coupled plasma emission spectrophotometer (ICP), and inductively coupled plasma mass spectrometer (ICP/MS) instruments are calibrated by the use of a minimum of three calibration standards (6 for ICP/MS) prepared by dilution of certified stock solutions. One standard is prepared at the reporting limit of the analyte of interest while the other standards bracket the concentration range of the samples. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration

standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. Calibration standards contain acids at the same concentration as the digestates. A continuing calibration standard is analyzed after every 10 samples. The value of the continuing calibration standard concentration must agree within method specified criteria, generally ±10 percent of the initial value or the appropriate corrective action is taken. Corrective action may include recalibrating the instrument and must include reanalyzing the previous ten samples.

4.3.2.3 Gas/Liquid Chromatography

Analysis performed by gas chromatography follows USEPA protocols. The instrument is calibrated using three or five point calibration curves (depending on method requirements) for both volatile and semi-volatile compounds. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. Continuing calibrations are performed after every ten samples. The value of the continuing calibration standard must agree within ±15 or 20 percent (depending on method requirements) of the initial value or the appropriate corrective action is taken, which may include recalibrating the instrument and must include reanalyzing the previous ten samples.

4.3.2.4 Gas Chromatography/Mass Spectrometry (GC/MS)

Prior to calibration, the instruments used for GC/MS analyses are tuned by analysis of p-bromofluorobenzene (BFB) for volatile analyses and decafluorotriphenylphosphine (DFTPP) for semi-

volatile analyses. Once the tuning criteria for these reference compounds are met, the instrument is initially calibrated using a three or five point calibration curve (depending on method requirements). The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must still be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. The instrument tune will be verified each 12 or 24 hours of operation (depending on method requirements). Continuing calibration is verified as specified in the method. The calibration standards are commercially available certified standards containing the target analytes, surrogate spikes, and internal standards.

4.3.3 Laboratory Equipment

Personnel performing calibration should also be alert for any condition that renders a piece of equipment inoperable or unfit for use; for example, inspect thermometers to ensure that mercury or alcohol columns are not separated. If an equipment malfunction is noted during calibration, the equipment must be tagged and removed from service. The equipment is held out of service until repairs and successful recalibration occurs. Record all malfunctions, repairs, and re-calibrations in the appropriate logbook.

Maintain records for each piece of equipment requiring calibration, showing equipment description and identification number, calibration frequency and acceptable tolerances, personnel performance calibration, date, reference material used, calibration results including acceptance or failure, removal from service, repairs, and date and authorization for return to service.

4.3.3.1 Balances

An annual third party maintenance and calibration is performed on all balances. Daily calibration is performed by TriMatrix on all

balances using class S or higher NIST traceable weights. Provided daily calibration is successful the weights themselves are indirectly calibrated on a daily basis via the third party's calibration; therefore, re-certification or replacement of the weights is not required every five years.

4.3.3.2 Thermometers

Thermometer calibration is performed annually, using a NIST certified thermometer. The NIST thermometer must be re-certified or purchased new every five years. Written records are maintained of all annual calibrations.

4.4 DATA REDUCTION, VALIDATION, AND REPORTING

Data reduction is the process by which raw analytical data is tabulated and calculated. Data validation is the review of the data generation and reduction process. Data reporting is the compilation of all sample results for distribution to the client. All analytical data generated by TriMatrix Laboratories is subjected to the reduction, validation, and reporting process as described below.

4.4.1 Laboratory Data

4.4.1.1 Data Reduction

Initial results for most analyses are calculated using a computer directly interfaced to the instument. Data reduction is accomplished using software that has been validated for its intended purpose. The initial result is exported to the LIMS system. Data such as initial volume, final volume, and percent solids, are used by the LIMS system to calculate a final result. When manual data reduction is required, it is performed according to the written standard operating procedure for that analysis.

4.4.1.2 Manual Integrations

Manual integration is defined as any post acquisition adjustment to the automated software peak integration. Manual integrations are often times legitimately required to correct for baseline drift, noisy baselines, poorly resolved peaks, closely eluting or missed peaks, peak tailing, or peak splitting. Manual integration may never be used for the sole purpose of correcting failing quality control parameters (i.e. shaving or enhancing peak areas or heights to make failed calibrations, surrogates, or internal standards pass), or as a substitute for poor or ineffective sample cleanup. Manual integration must be used cautiously due to the increased scrutiny inherent with adjusted data. Particular attention will be paid to manual integrations performed on standards and blanks since these samples are typically free of interferences.

Before and after documentation must be provided with all manual integrations. This documentation must clearly show the original integration “before”, and the manual integration “after” baseline. Clear identification of manual integrations must be included in the case narrative for all samples analyzed under Federal Facilities work requirements. All quantitation reports must clearly identify manual integrations by flagging the peak with a designator that cannot be removed by the analyst. Additional documentation requirements include:

- Date of the manual integration
- Reason for the manual integration
- The integration area or height before manual integration
- The integration area or height after manual integration
- A signature/date by both the analyst and the reviewer.

Any questions concerning manual integration must be resolved with the area manager or the quality assurance officer before final results are approved and released to the Project Chemist. The

complete laboratory manual integration requirements are detailed in the TriMatrix manual integration SOP GR-10-115.

4.4.1.3 Four Levels of Data Validation

First Level Review

Data validation begins with the analyst. It is the basic responsibility of the analyst to produce data that is complete, correct, and conforms to all applicable methods and standard operating procedures. If results are not acceptable, it is the duty of the analyst to perform the appropriate corrective action and to thoroughly document that action. The analyst will verify the following before updating the analysis status to "Analyzed":

- Applicable standard operating procedures were followed
- Proper analytical sequence was followed
- Sample preparation information was correct
- Calibration has been performed properly
- Analytical results are complete
- Holding times have been met
- Method criteria were met
- Any special sample preparation or analytical requirements have been achieved
- All analytical abnormalities have been noted
- Corrective actions are thoroughly described
- Good record keeping practices have been followed
- Any problems are communicated to area manager
- Data was correctly transferred to Element
- Calculations were performed properly
- Quality control samples are within established limits
- Documentation is complete
- Raw data, including chromatograms and instrument printouts are complete
- Case narrative or qualifier pages are complete

Second Level Review

A laboratory area peer or designated validator, in essence, performs the same validation steps performed by the analyst. Particular attention should be paid to:

- Dilution factors were entered correctly and detection limits elevated accordingly
- Analysis dates are correct
- Quality control and analytical batch information is correct
- Quality control results and spike amounts are correct and in control
- Project specific limits are correct
- Run a draft copy of the report, specific to the laboratory area, to verify all results have been adjusted correctly
- Any required qualifiers or narratives have been entered

Any problems must be resolved with the analyst, and when appropriate the quality assurance manager, prior to updating the status to "Reviewed."

Third Level Review

Once all analyses associated with a work order have been entered into the LIMS system and approved, the project chemist will perform the Third Level Review. This review will verify that:

- The requirements of the client have been met
- All required narratives and qualifiers have been included
- All quality control parameters required are in the report
- Results of complimentary tests make sense
- The data is accurately presented
- Holding times have been met
- Calibration checks are sufficient

- Documentation is complete

Once this review is complete the project chemist will approve the data and generate a final report. It is during this time that any data package deliverables are collected and reviewed. When printed the work order status updates to "Reported."

Fourth Level Review

The project chemist will perform a final review of the data package hard copy to ensure that:

- All required data package components are complete and accounted for
- Quantitative results are correct
- The overall presentation of data to the client is in an understandable format

In addition to the formal data validation guidelines listed above for the analyst, area manager, and project chemist, there are many practical questions that all of these persons need to keep in mind when reviewing data and finished client reports. Among these "common-sense" evaluations of laboratory data are the following important considerations:

- Data makes good, sound, practical sense
- Multiple runs of the same samples relate, match, or are within acceptable range
- Data from complimentary analyses compares, i.e. COD>BOD>CBOD
- Total cyanide ≥ amenable and free cyanide
- Total solids ≥ suspended and dissolved solids
- TKN ≥ organic N + ammonia N
- Inorganic N = ammonia N + nitrate N + nitrite N
- TOC < BOD or COD

- Total phosphorus ≥ ortho phosphorus
- Calculated total dissolved solids/conductivity = 0.55 – 0.7
- Analytical run looks good; proper decisions were made
- Peaks from chromatogram or instrument printout look normal
- Computer identifications are correct
- Are qualitative/quantitative results real, especially low level
- Know and be sensitive to common laboratory contaminants
- Know area/analytical method pitfalls-be extra cautious
- All practices are sound and are supported by documentation-no appearance of random decisions

When complete the report will be signed. Data packages with deliverables will be scanned and archived. Work order status will be updated to "Completed".

4.4.2 Field Data

All data reduction, validation, and reporting for field activities must meet the same requirements as those required in the laboratory. Many of the field instruments, such as those measuring pH, dissolved oxygen, turbidity, temperature, and specific conductance, require a manual data printout from a computer interface. The analyst is responsible for immediate tabulation and calculation of raw data in the field. The field section manager must perform a prompt, on-site validation of field data before the opportunity is lost to perform any necessary field re-tests.

4.4.3 Subcontracted Data

Analytical results from subcontracted samples will be reported as an attachment to the TriMatrix data package. The attachment will contain the entire subcontracted data package as received by TriMatrix. To eliminate the impression that the subcontracted analyses were performed by TriMatrix, subcontracted results will never be incorporated into the TriMatrix generated report.

4.5 VERIFICATION PRACTICES - EXTERNAL/INTERNAL QUALITY CONTROL

4.5.1 Standard Reference Materials

A crucial step in the generation of quality data is the purity and traceability of reference materials used in the analyses. Reference materials may be physical standards (such as certified thermometers and weights used to calibrate laboratory thermometers and balances) or chemical standards (used to establish and check operational calibration of analytical methods). Physical standards should be traceable to the National Institute of Standards and Technology (NIST). Physical standards must be recalibrated (by an external vendor certified to perform the calibration), or purchased new every five years. Chemical reference materials of high quality can usually be obtained from reliable commercial vendors. For a given analysis, standard reference materials must be kept on hand from more than one vendor source. During the testing operation, standard reference materials from different vendor sources are crosschecked with each other.

4.5.2 Internal Quality Control Programs

TriMatrix routinely adds samples to the sample stream to demonstrate the total testing process is operating within prescribed limits for accuracy and precision. With the exception of Blanks, the concentration of these quality control samples is known prior to the analysis. Types of Quality Control Samples are presented in Table 5. Duplicates and spiked duplicates are selected at random, and when not specified are rotated among clients.

4.5.3 External Quality Control Samples-Proficiency Testing

TriMatrix Laboratories receive Performance Testing (PT) samples on a scheduled basis from state and federal regulatory agencies as well as certain client organizations. A summary of these PE samples is given below:

PT Program	Sample Type	Source	Frequency

WS	Drinking Water	ERA	Semi-Annual
WP	Waste/Ground Water	APG	Semi-Annual
Soil	Soil	ERA	Semi-Annual
Varies	Environmental	State/Federal Programs	Varies
Varies	Environmental	Client	Varies

TriMatrix receives written reports from sponsoring agencies grading not only their performance, but also a comparison to other laboratories participating in the study. This provides feedback to laboratory personnel regarding the satisfactory use of analytical methods and equipment. Additionally, results from all single and double blind PT samples are used as part of the laboratories fraud prevention and detection program.

4.6 DATA ASSESSMENT PROCEDURES

4.6.1 Precision

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

$$\%\text{RPD} = \left(\frac{S - D}{\frac{S + D}{2}} \right) \times 100$$

where:

S = first sample value (original of MS value)

D = second sample value (duplicate or MSD value)

4.6.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria using the analytical results of method blanks, reagent/preparation blank, matrix spike/matrix spike duplicate samples, equipment blank, and trip blanks. The percent recovery (%R) of matrix spikes will be calculated using the equation below:

$$\%R = \left(\frac{A - B}{C} \right) \times 100$$

where:

A = the analyte concentration determined experimentally from the spiked sample;

B = the background level determined by a separate analysis of the un-spiked sample

C = the amount of the spike added

4.6.3 Control Limits

Unless fixed in the analytical method, all quality control acceptance limits in use at TriMatrix are derived from historical data, for each method, matrix, and QC type combination. Precision and accuracy control limits are calculated at a 99% confidence level (+/- three standard deviations); warning limits are calculated at a 95% confidence level, (+/- two standard deviations). Accuracy windows are calculated using the mean of the percent recoveries. Precision windows are calculated as specified in SW-846, using the relative percent difference of the amounts found, not the percent recoveries.

4.6.4 Uncertainty

In addition to the precision and accuracy of a result, a value relating to confidence is available in the form of a measurement uncertainty estimate. The measurement uncertainty value is estimated using the QC-based nested approach and is calculated at the 95% confidence level. Uncertainty estimates are reported as "percent relative uncertainty."

4.6.5 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 follows:

$$\text{Completeness} = \left(\frac{\text{valid data obtained}}{\text{total data planned}} \right) \times 100$$

4.7 PROCEDURES FOR CORRECTIVE ACTION

When a non-conforming event or process deviation has occurred, corrective action is required. A written standard operating procedure (plan for corrective action) provides the steps for dealing with an out-of-control testing situation. The assessment of whether the process is out-of-control is based on predetermined limits for laboratory operations. Non-conformances based on statistical analysis or quality control samples are readily apparent and easy to identify. A process deviation, which does not have a directly observable impact on data quality, is more difficult to discern. Examples of the latter, subtler types of non-conformances include volatile samples not properly stored; oily layers in certain types of samples that may interfere with analysis; or a water-soaked sample label whose information is barely legible. Discovery of a non-conforming event or process deviation can result from the observations of a staff member, a review of laboratory data at any level, the result of an audit, or a client complaint. A corrective action investigation will be initiated within one week of the discovery of any non-conformance. The time frame required to resolve a specific deficiency and implement the corrective action is dependant on the magnitude of the problem and the defensibility and use of the data. Most non-conformances should be resolved within 60 days from the initiation date. Non-conformances that specifically impact sample results should be resolved within 14 days.

NOTE: The client must be contacted within 48 hours (2 business days) upon the discovery of any event that may cast doubt on the validity of a sample result.

The overall scheme of a corrective action plan can be outlined as follows:

1. Define the problem and evaluate the significance of the non-conformance;

2. Assign responsibility for evaluating the problem and determine if the client should be notified and/or work recalled;
3. Determine thorough investigation of all the pertinent facts what the probable cause of the problem is;
4. Select and implement the action(s) most likely to eliminate the problem and prevent recurrence;
5. Assign responsibility for carrying out the corrective steps and implement the action;
6. Follow-up to ensure that the problem has been eliminated and when necessary authorize the resumption of work.

Specific responsibility for implementing corrective action is as follows:

It is the responsibility of the analyst or other employee who observes a non-conforming event to:

- Identify and define the problem.
- Fill out a Non-Conformance Investigation Report (refer to Appendix AH).
  When applicable, investigate and attempt to determine the cause of the problem. Report the problem promptly to the area manager. When applicable, accept responsibility for implementing the corrective action approved by the area manager.
- When applicable, evaluate the effectiveness of the corrective action.
- When applicable, verify that the corrective action has eliminated the problem.

It is the responsibility of the laboratory area manager to:

- Review the problem and the proposed corrective action.
- If the reporting person does not have a remedy, work together with the person to determine a satisfactory solution.
- Assign the final corrective action steps to be performed.

It is the responsibility of the QA Department to:

- Follow-up to ensure that the problem has been eliminated and when necessary authorize the resumption of work.
- Review, sign, and categorize every Non-Conformance Investigation Report.
- Randomly review corrective action documentation in laboratory through internal audits to ensure that adequate records are being kept.

The ultimate goal of every non-conformance investigation is to resolve the error through identification of the error's root cause. Ideally, once the source of error is found, change can be implemented to prevent reoccurrence of the same error thereby providing a system of continuous quality improvement.

Non-conformances can originate from anyone in the laboratory. Provide the QA department with a copy of the initial report at the time of its distribution, followed by a copy of the completed report. The final report will be distributed to all necessary personnel. Initiation of non-conformance reports associated with out-of-control PT samples will commence with the QA department. The initial non-conformance will be typed up and may include attachments such as a graph charting the history of PT results for that analyte. The history of results for that analyte in PT studies will also be reviewed through the database, looking at additional items such as method, matrix, analyst, vendor, and study type (WP, WS, etc.).

NOTE: Non-conformances associated with PT samples must be completed and distributed to state, federal, and other applicable regulatory agencies within the time frame established by that agency.

Returned non-conformance reports will be typed and the final report may include copies of raw data, information concerning traceability, graphs charting historical data, graphs charting trends in analysis, calibration graphs, or any other information relevant to the investigation.

When investigating a failing PT sample, a questionable analytical result, or a client complaint, the following systematic approach for error analysis should be followed until the primary source of error is located and resolved. Progress through them in the order they are presented below (easy to determine transcription error through difficult to determine analytical/procedural failure).

1. Consolidate all necessary raw information, run data and associated calibration and quality control data for both the reported and any non-reported analyses of that sample.
2. Confirm that the intended result was the reported result (transcription error).
3. Verify that the sample was prepped correctly.

4. Verify the correct analytical and pre-treatment method was used.
5. Double check all manual calculations, looking for incorrectly calculated results, missing dilution factor, wrong initial and final volumes, etc. Where possible manually calculate the result and compare with the reported result.
6. Compare the age of the calibration to the PT analysis date.
7. Review data associated with all quality control samples for biases. Also evaluate all QC solutions with respect to age, source, storage, and handling.
8. Determine the reasonableness of the data. Verify that all QC parameters were in control. Compare results to established limits to the data quality objectives of the study (i.e. tighter QC required for WS studies).
9. Review standard laboratory techniques used on the sample and all associated QC analyses. Were measurements used in quantitation made volumetrically? Were pipets and volumetric flasks used, or were less stringent techniques employed? Were serial dilutions made during the preparation of the curve?
10. Review analytical conditions, integration, background corrections, analyte resolution, and any confirmation runs.
11. Review calibration ranges. Are they too large for the analysis? An over extended calibration range will appear S-shaped. Check the population of curve points in the area of the analyte concentration.
12. Review calibration type (linear, average, response factor, polynomial non-linear, etc.). Reprocess multi-level curve data through a best fit program and if linear, perform a residuals analysis to identify outlier calibration points. If the result was quantitated using an average response factor, compare with the best-fit information and confirm justification for use of the average response factor quantitation.

In general, there are three major areas where corrective action is required. These categories are described below. Non-Conformance Reports are required on indications flagged with a *. Other indications may require a Non-Conformance Report based on the circumstances.

4.7.1 Quality Control Failures

These are usually handled within the laboratory by the analyst.

Indications of Non-Conformance

- Blanks, laboratory control, or spiked samples contain contamination greater than acceptable levels.
- Suspicious trends in spike recoveries or relative percent differences (RPD) between duplicates.
- Initial instrument blank, initial calibration standards, QC check standards, continuing calibration standard spikes, or method blanks are outside acceptance criteria.
- The method blank or instrument blank analysis exceeds the detection limit for the analyte.

Recommended Corrective Action

- Prepare another instrument blank. If the response is still greater than the reporting limit, look for sources of contamination in reagents, the laboratory working environment, and the instrument.
- Reanalyze standard. If results are still unacceptable, prepare new standards. If necessary obtain new primary standards.
- Reanalyze continuing calibration standard. If necessary, recalibrate and reanalyze samples since last successful continuing calibration.
- Evaluate preparation of spikes, spiking techniques, spiking equipment and materials.

4.7.2 Procedural Failures

These are usually handled by the laboratory area manager and the quality assurance department.

Indications of Non-Conformance

- There are unusual changes in detection limits.
- Statistical quality control data is demonstrating unacceptable trends or is outside the warning or acceptance limits.
- Deficiencies are evidenced on performance evaluation samples or internal or external audits.
- Clients express concern about the quality of their data.

Recommended Corrective Action

- Review the method with the analyst.
- Reanalyze the samples and evaluate the results.
- Recalibrate the instrument or analysis method with freshly prepared standards and reanalyze the samples.
- Re-extract and reanalyze the samples per the method.
- Evaluate the data and sample behavior and investigate any possible chemical interferences.
- Re-run the samples using the method of standard additions.
- Check the instrument for possible maintenance deficiencies.
- Seek additional help from other analysts or provide additional training for personnel involved.
- Perform a system audit to evaluate corrective action measures.

4.7.3 Test Specification Failures

These are usually handled by the analyst, laboratory area manager, and the quality assurance department.

Indications of Non-Conformance

- Quality control check standard data is outside the acceptance limits defined for that analyte.

Recommended Corrective Action

- Review the method with the analyst.
- Reanalyze the check standard and evaluate the results.
- Prepare fresh check standard or new primary standard.
- Recalibrate the instrument or analysis method.
- Switch to a different standard vendor.
- Investigate possible chemical interferences.
- Check the instrument for possible maintenance deficiencies.
- Retrain the analyst.

4.7.4 Customer Complaints

The Quality Assurance Department coordinates with the client services staff to receive quality feedback from clients. It is the responsibility of the QA department to communicate any customer complaints to the laboratory operating areas and to follow-up on corrective action taken to prevent a recurrence.

4.8 PROCEDURES FOR PREVENTIVE ACTION

Changes and enhancements to existing policies and procedures are not always made based on the result of failing analytical performance or other non-conformances. Borderline performance, equipment changes/modernization, or outdated internal procedures are all areas that may require modification or enhancement. Employees are encouraged to analyze internal procedures of all kinds, and offer suggestions for improvement. A Preventive Action Investigation form exists for this purpose (Appendix AI). The form is used to record a description of the existing procedure and a proposed solution, an action plan and systematic implementation schedule, and a follow-up section to monitor the effectiveness of any resulting changes.

All Preventive Action Investigations are loaded into a database similar to that used to track non-conformances

4.9 DEPARTURE FROM DOCUMENTED PROCEDURES

4.9.1 Management Policies

Any departure from a laboratory written standard operating procedure not directly involving sample analysis or processing must be approved by the area manager. The area manager must file a Non-Conformance Investigation Report. The Non-Conformance Investigation Report must be included as part of the data package.

Any departure from a SOP involving sample processing or sample analysis must be justified in writing by the analyst and laboratory area manager. The prior written approval of the laboratory president must be received before performing the analysis. The laboratory president must also file a Non-

Conformance Investigation Report. This Non-Conformance Investigation Report must be included as part of the data package (the exception to this requirement is those items in the analytical methods where a written justification for technical and scientific reasons has been determined by the analyst and approved by the Laboratory President as a deviation from the analytical method).

4.9.2 Method Modification and Variances

Modification of, and variances in, analytical methods, except for the deviations justified in writing and approved per section 4.9.1, are strictly prohibited.

4.10 PERFORMANCE AND SYSTEM AUDITS

4.10.1 Internal Audits

Annually the laboratory will be audited by the quality assurance department to verify compliance to ISO-17025 and various State and Federal requirements. Additionally, quarterly internal audits will be conducted by the quality assurance department. Together these audits will encompass all elements of the quality system. A formal written follow-up will be conducted after every internal audit to verify that any deficiencies cited have been corrected, and that the corrective actions have been successful. The following areas will be included in the required internal audits.

4.10.1.1 System Audits

System audits are used to determine that each component within a laboratory system is functioning properly and adheres to the appropriate standard operating procedures, analytical methods, and requirements of the Quality Assurance Manual. Systems to be audited include:

A). Sample Handling and Control
B). Sample Analysis

C). Records Processing and Control
D). Support Systems (such as air handling, DI water, analytical balances, raw materials, etc.)

If during the course of an internal audit, problems were uncovered that may have impacted the laboratories ability to generate quality data, written notification must be provided to all impacted clients. Impacted clients include all those clients who received results from samples analyzed during the time frame the problem occurred. This is accomplished by a letter explaining the problem, and includes revised copies of the report that, if necessary, include any required data qualifiers.

4.10.1.2 Documentation Audits

The Quality Assurance department also performs audits of the laboratory documentation (laboratory notebooks, benchsheets, instrument run logs, client file folders, etc.) to assess the thoroughness and completeness of the documents.

4.10.1.3 Surveillance Audits

The Quality Assurance department, Area Manager, or their designate observes an analyst in detail as a test is being performed. Attention is given to general laboratory demeanor (orderliness, cleanliness, good laboratory practices in measuring, documentation, etc.) as well as to adherence to analytical methods and standard operating procedures.

4.10.1.4 Quality Assurance Reports to Management

The Quality Assurance Manager provides the Laboratory President with a copy of all external audit reports. The report details any deficiencies identified as well as recommended corrective actions.

4.10.2 External Audits

4.10.2.1 On-Site Audits

Audits of the laboratory conducted by regulating agencies and client organizations are to be perceived by the laboratory staff as learning experiences and opportunities to hear suggestions from knowledgeable persons on how operations might be improved. Consequently, the laboratory staff is to be open and cooperative with external auditors. Formal follow-up using written summaries of external audits is to be carried out to ensure that any suggested improvements are thoroughly evaluated.

Figure 4-1
Documentation System Structure

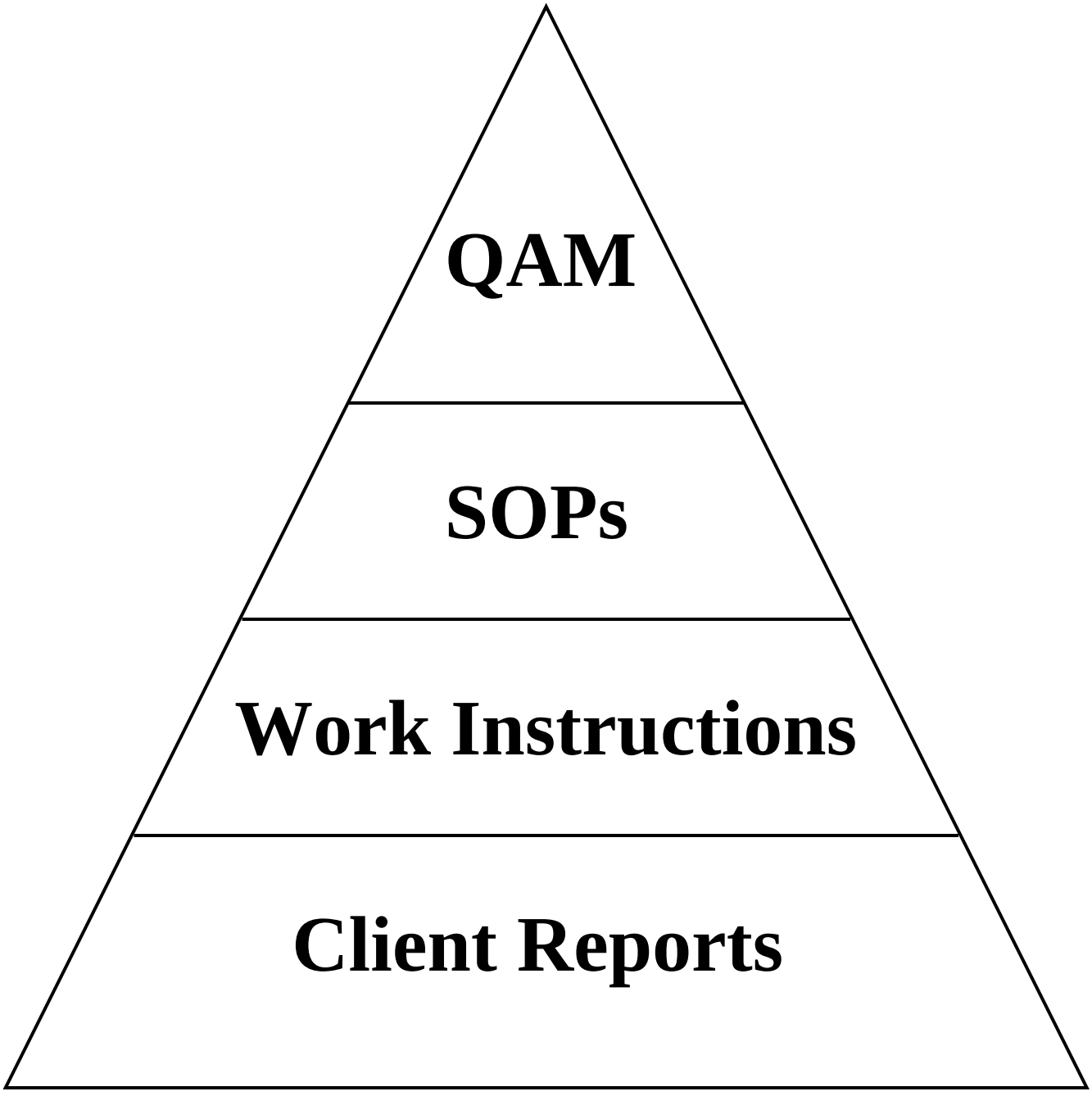


Figure 4-2
Document – Benchsheets/Client Report
Flow Diagram

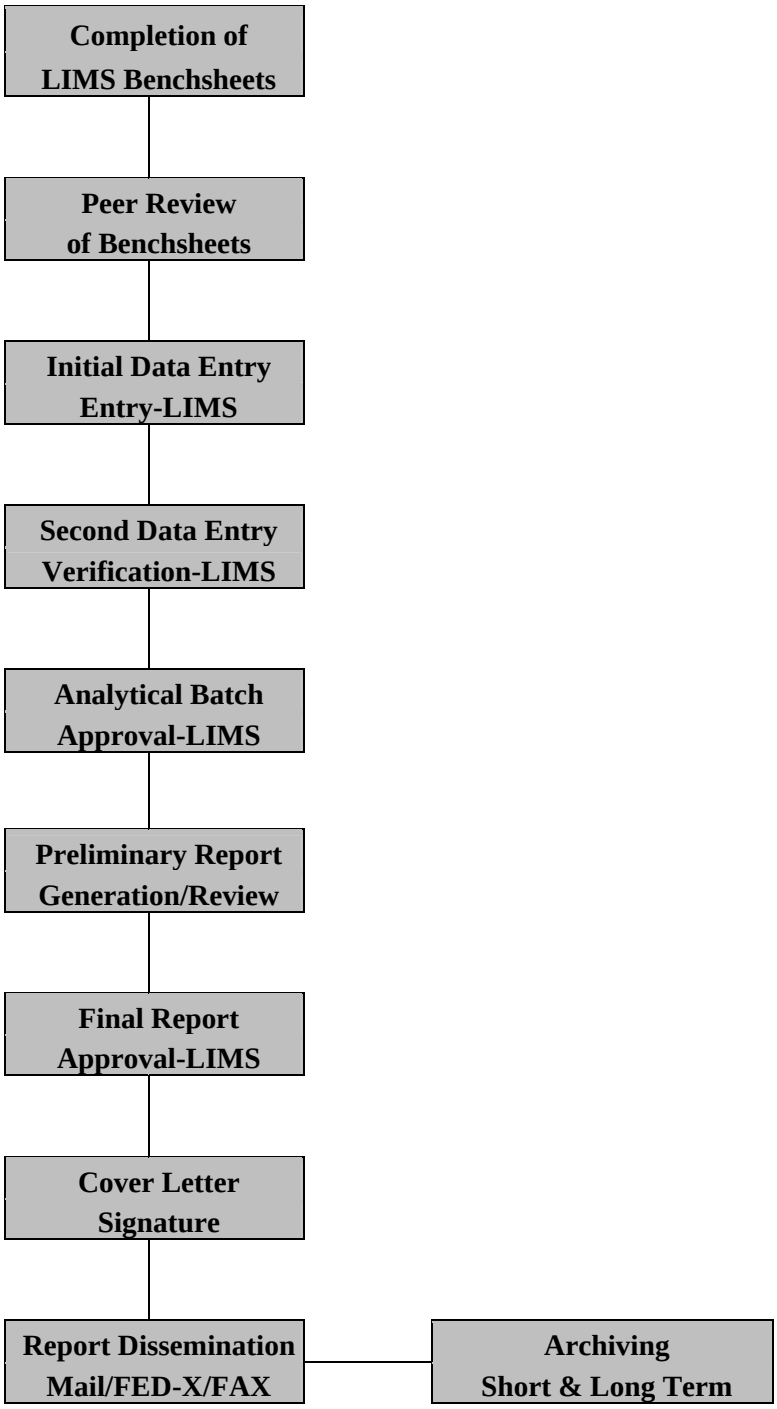
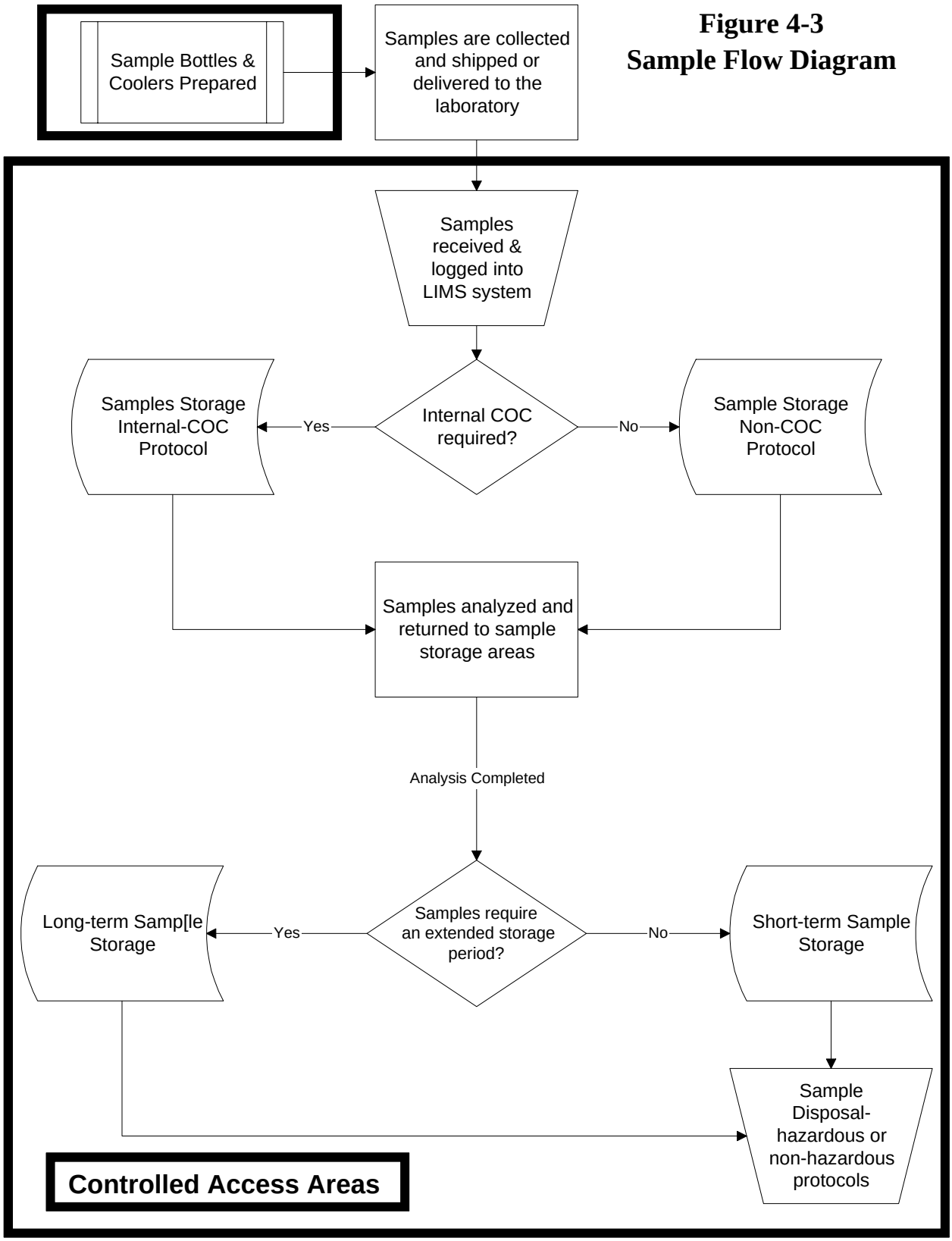


Figure 4-3
Sample Flow Diagram



5.0 REFERENCES

- Methods for Chemical Analysis of Water and Wastes; EPA-600/4-79-020 most current revision.
- Standard Methods for the Evaluation of Water and Wastewater; Current Edition, APHA, AWWA, WPCF.
- Handbook for Analytical Quality Assurance in Water and Wastewater Laboratories; EPA 600/4-79-019, most current revision.
- Physical and Chemical Methods for the Evaluation of Solid Waste; EPA-SW-846, most current revision.
- Guidelines Establishing Text Procedures for the Analysis of Pollutants; 40 CFR; Parts 100 to 149, Current Edition.
- Good Automated Laboratory Practices; USEPA Office of Administration and Resource Management, most current revision.

TABLE 1
Default Data Archiving Systems

Document Archives

Document Description	Storage Location	Storage Duration
Laboratory benchsheets	on-site	1 year
Laboratory benchsheets	off-site	6 years
Instrument Print-Outs (raw data)	on-site	1 year
Instrument Print-Outs (raw data)	off-site	6 years
Laboratory Logs (run, maintenance, analyst)	on-site	1 year
Laboratory Logs (run, maintenance, analyst)	off-site	6 years
Client Files (reports, correspondence, invoices)	on-site	1 year
Client Files (reports, correspondence, invoices)	off-site	6 years
Proposal Files	on-site	5 years
Purchase Agreements	on-site	5 years
SOPs	on-site	5 years

Electronic Archives

File Description	Storage Location	Storage Duration	Storage Media
Instrument Data Files-GC/MS	on-site	1 year	Compact Disk
Instrument Data Files-GC/MS (copy)	off-site	10 years	Compact Disk
Instrument Data files-GC (Turbochrom)	on-site	1 year	Compact Disk
Instrument Data files-GC (Turbochrom) (copy)	off-site	10 years	Compact Disk
Instrument Data files-AA, ICP, ICP/MS	on-site	1 year	Compact Disk
Instrument Data files-AA, ICP, ICP/MS (copy)	off-site	10 years	Compact Disk
Instrument Data files-Auto Analyzer	on-site	1 year	Compact Disk
Instrument Data files-Auto Analyzer (copy)	off-site	10 years	Compact Disk
LIMS daily backup	on-site fire-safe	30 day rotation	DAT-Tape
SOPs	on-site	indefinitely	Compact Disk

TABLE 2
Laboratory SOP Categories

Trace Metals
Gas Chromatograph
Spectrophotometric Procedures
Gravimetric Procedures
Extractions-Organic
Sales and Customer Service
Laboratory Computer Operations
Sample Receiving, Storage, & Disposal
Bottle Prep
Microbiology
Waste Characterization
Instrumental-General
Gas Chromatography/Mass Spectroscopy
Titrimetric Procedures
Electrochemical/Potentiometric Procedures
Quality Assurance
Business and Accounting
Laboratory Safety and Security
Miscellaneous
Inorganic-General

TABLE 3
Field Equipment Calibration

Equipment	Method Reference	Minimum # Standards Initial Calibration	Type of Curve	Frequency of Calibration	Acceptance/ Rejection Criteria Initial Calibration	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
Conductivity Meter	SW-846 Method 9050	2	---	Initial	± 5% of Value	Daily	---
Dissolved Oxygen Meter	Standard Method 4500-O G.	---	---	Initial	± 5% of Value	Daily	---
Temperature Probes	Standard Method 2550 B.	---	---	Initial	± 5% of Value	Daily	---
pH Meter	SW-846 Method 9040	3	Linearity	Initial	Adjust slope to within ±0.05 pH units accuracy	Daily	---

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
Mercury Cold Vapor AA	SW-846 7470/7471 EPA 245.1	5	Correlation coefficient must be ≥0.995	Daily, at the beginning of every analytical batch, and when CCV fails acceptance criteria	Every calibration	90-110% recovery 95-105% recovery	Every 10 samples	90-110% recovery 90-110% recovery
ICP	SW-846 6010 EPA 200.7	3	same as above	same as above	same as above	95-105% recovery	same as above	90-110% recovery
ICP/MS	SW-846 6020 EPA 200.8	6	same as above	same as above	same as above	90-110% recovery	same as above	90-110% recovery
Ion Chromatograph	SW-846 9056 EPA 300.1	6	Correlation coefficient must be ≥0.995	Every 6 months or when CCV fails	Every calibration	90-110% recovery	Every 10 samples	90-110% recovery
Konelab: Sulfate Chloride	EPA 600/4-79-020 Method 375.2 Method 325.2	 10 8	same as above	Every batch	same as above	85-115% recovery	Every 10 samples	85-115% recovery
Phenolics (Total)	SW-846 9065 EPA 420.1	5-7	same as above	same as above	same as above	85-115% recovery	Every 10 samples	85-115% recovery
Cyanide Total and Amenable	SW-846 9012, 9014 EPA 335.1, 335.3, 335.4	7	same as above	same as above	same as above	90-110% recovery	Every 10 samples	90-110% recovery

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
TOC Analyzer-TOC	EPA 415.1	5	same as above	same as above	same as above	85-115% recovery	Every 10 samples	85-115% recovery
GC-PID/ ELCD	SW-846 8021	5 for linear 6 for quadratic	≤20% RSD use average RF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration; ±20% for compounds that boil below 30° C (Bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, and vinyl chloride
	EPA 601/602	3	<10% RSD use average RF or regression, ≥10% must use regression	As needed when CCV fails method Table 2 criteria				Method Table 2 criteria
GC-FID	SW-846 8015	5 for linear 6 for quadratic	≤20% RSD use average CF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
GC-ECD	SW-846 8081 SW-846 8151 SW-846 8082 SW-846 8121 EPA 608 EPA 612	5 for linear 6 for quadratic 3	≤20% RSD use average CF or regression, >20% must use regression <10% RSD use average CF or regression, ≥10% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration; breakdown criteria: DDT <15% Endrin <15%, <20% total
GC-HPLC	SW-846 8310	5 for linear 6 for quadratic	≤20% RSD use average CF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
GC/MS-Volatiles	SW-846 8260	5 for linear 6 for quadratic	CCCs – %RSD ≤30% 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene ethyl benzene, vinyl chloride, all other target analytes ≤15% use average RF for quantitation, otherwise regression SPCCs – average RF ≥ 0.10 for chloromethane, 1,1-dichloroethane and bromoform; ≥ 0.30 for 1,1,2,2-tetrachloroethene and chlorobenzene	As needed, when CCV fails	As needed, with analysis of each curve	80-120% recovery	12 hours	8260: CCCs – % Difference or drift ≤20%, all other target analytes within 20% expected value, high recovery acceptable when analyte not present in sample; SPCCs same criteria as initial calibration
	EPA 624	3	<35% RSD for all compounds use average RF, otherwise use regression				24 hours	Recovery of all analytes must meet recoveries specified in Table 5

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
GC/MS-Semi-volatiles	SW-846-8270	5 for linear 6 for quadratic	CCCs – %RSD ≤30% acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-diphenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol, 2,4,6-trichlorophenol, all other target analytes ≤15% use average RF for quantitation, otherwise regression SPCCs – average RF ≥0.05 N-nitrosodi-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, 4-nitrophenol	As needed, when CCV fails	As needed, with analysis of each curve	80-120% recovery	12 hours	8270: CCCs % Difference or drift ≤20%; all other target analytes within 20% expected value, high recovery acceptable when analyte not present in sample; SPCCs same criteria as initial calibration
	EPA 625	3	<35% RSD for all compounds use average RF, otherwise use regression				24 hours	80-120% recovery

TABLE 5
Quality Control Sample Types

Blank Type	Abbreviation	Description	Frequency of Use
Method Preparation Blank	MPB	This blank has been carried through the entire analytical process including any pretreatment procedures. The MPB will monitor any contaminants that may affect the sample results. General acceptance limits for the MPB are less than the test reporting Limit. If contamination is detected in the MPB above the reporting limit, all samples with analyte concentrations within 10x that found in the MPB must be flagged for re-extraction or digestion. If it is not possible to re-prep the samples then all analyses for that batch must be qualified.	One per analytical batch

TABLE 5
Quality Control Sample Types

Blank Type	Abbreviation	Description	Frequency of Use
Continuing Calibration Blank	CCB	The continuing calibration blank is a reagent blank that is analyzed as a sample, generally after 10 samples have been tested. The CCB must be run prior to re-zeroing an instrument, unless this practice was performed for each previous sample. The CCB will verify whether significant instrument drift has occurred during the analytical run near the test method detection limit. General acceptance limits are ± the test reporting limit. If the CCB falls outside the acceptance limits, the instrument must be recalibrated and the previous 10 samples reanalyzed. For automated tests where run data is generated after all analyses are completed, 10 samples before and after the unacceptable CCB must be reanalyzed, i.e., all sample results must be encased in acceptable CCB. The reanalysis must also include the ICB and ICV QC samples.	Every ten samples/or as specified in the analytical method.

TABLE 5
Quality Control Sample Types

Blank Type	Abbreviation	Description	Frequency of Use
Field Trip Blank	FTB	These are used with VOA vials where there is the possibility that organic contaminants may diffuse through the PTFE-faced silicone rubber septum of the sample vial. A field trip blank vial filled with organic-free water accompanies the sample containers to and from a client location, at the discretion of the client, may be analyzed along with the samples.	One per sample shipping container
Storage Blank	STB	Reagent-grade water (40 mL aliquot) is stored with samples in a client set. Per the discretion of the client, it may be analyzed after all samples in that set are analyzed. The purpose is to determine the level of contamination acquired during storage.	One per sample storage refrigerator or client sample set (if required)

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Laboratory Fortified Blank or Blank Spike	LFB or BS	This is a fortified method preparation blank in which an aliquot of de-ionized water has been spiked with a known amount of a stock reference standard or spiking solution. A blank spike is required for each digestion or distillation batch. The purpose of the blank spike is to verify the analyst's spiking procedure and assure that any matrix interference shown by the spike and spike duplicate is really matrix induced.	One per analytical batch or as specified in the analytical method

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Second-Source Calibration Verification	SCV	The SCV is identical to the CCV with the exception it must be made from a source dissimilar to that used to prepare the initial calibration curve. The purpose of the SCV is to validate the accuracy both the calibration standards, and the initial calibration curve. Unless otherwise specified by the method, recovery limits for this QC type are typically 80-120%. Sample analysis may not begin prior to the analysis of a successful SCV.	One with every initial calibration

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Continuing Calibration Verification	CCV	The continuing calibration verification standard is generally the standard used as the midpoint of the initial calibration curve. The standard is analyzed and quantitated in the in the same manner as a sample. The CCV will reveal any significant instrument drift. Acceptance limits for this QC type are ± 10%, or as stated in the method. If the CCV falls outside the acceptance window, the instrument must be recalibrated and the previous 10 samples reanalyzed. For automated tests where run data is generated after all analysis is complete, all samples run after the last acceptable CCV must be reanalyzed, i.e. all samples must be bracketed by an acceptable CCV.	Every 10 samples or as specified in the analytical method

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Detection Limit	CRDL	A standard which contains the minimum level of detection acceptable under a contract Statement of Work must be analyzed for particular contract sample sets to demonstrate that detection limit can be met.	One per analytical batch for certain contract sample sets and methods only.
Sample Duplicate	DUP	The sample duplicate is a replicate analysis of a particular sample that has been analyzed previously during the sample analytical batch. The purpose of the duplicate is to monitor precision within the analytical process.	Every 10 samples for each matrix type

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Sample Matrix Spike	SPK	The sample matrix spike is an aliquot of a sample that has been spiked with a known amount of a stock reference standard or spiking solution. A the purpose of the SPK is to monitor sample matrix effects on the test. Acceptance limits for this QC type are based on the 95% confidence limits established for a test and matrix.	Every 10 samples for each matrix type, or as specified in the analytical method

TABLE 5
Quality Control Sample Types

Matrix QC Type	Abbreviation	Description	Frequency of Use
Matrix Spike Duplicate	MSD	A matrix spike duplicate is an aliquot of the same sample used for the matrix spike (SPK). A spike duplicate is required for each matrix type within a digestion or distillation batch. A spike duplicate analysis may be required on a non-distilled or non-digested sample if the spike has indicated a matrix interference. The purpose of this duplicate spike is to confirm any matrix effects on the test. Acceptance limits for this QC type are based on the 95% confidence limits established for a test and matrix.	Every 10 samples for each matrix type or as specified in the analytical method

TABLE 5
Quality Control Sample Types

Matrix QC Type	Abbreviation	Description	Frequency of Use
Field Duplicate	FDUP	This may be required to evaluate the uniformity of samples and sampling techniques at a field location. Acceptance limits for this QC type are based on established confidence limits, with generally two levels or ranges. The first range extends from the test reporting limit to 10x the test reporting limit. The second range encompasses any values higher than 10x the MDL.	As required on a project basis
Post-Digestion Spike	PDS	The post-digestion spike may be required, on a project basis, when a matrix precludes the use of pre-digestion spike.	One per analytical batch when required by project

TABLE 5
Quality Control Sample Types

Matrix QC Type	Abbreviation	Description	Frequency of Use
Surrogate Spike	SUR	For almost all organic analyses, the analytical method requires surrogate compounds to be added to every blank, sample, matrix spike, matrix spike duplicate, and standard. Surrogate compounds are used to measure analytical efficiency by measuring percent recovery from the known value. They are generally brominated, fluorinated, or isotopically labeled compounds not typically detected in environmental samples.	Every QC and per batch for semi-volatile, volatile, pesticide, PCB analysis
Internal Standard	IST	These are compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), at a known concentration, prior to analysis. Internal standards are used as the basis of quantitation of the target compounds.	Every QC and client sample per batch for volatiles and semi-volatiles

Appendix A

TriMatrix Laboratories, Inc.

CHEMIST I

General Description

Under direct supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties.

Educational/Background Requirements

- Associates degree and 3 or more years of experience in an environmental or related laboratory setting; or

- BS degree in Chemistry or a related field of science.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist I.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).

- Responsible for the daily operation and routine maintenance of instruments and equipment.

- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.

- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.

- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.

- Follow all laboratory safety procedures.

- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

CHEMIST II

General Description

Under ***general*** supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties.

Educational/Background Requirements

- Associates degree and ***5*** or more years of experience in an ***applicable discipline***; or
- BS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline***; or
- ***MS degree in Chemistry or a related field of science***.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist II.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- ***Assist other chemists and technicians with their professional development***.

- ***Act as company advocate by setting a positive example in work habits and attitude to other staff members***.
- ***Demonstrate ability to work independently with minimal errors***.
- ***Capable of conducting peer review on routine data packages***.
- ***Possess the minimum level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements***.
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

CHEMIST III

General Description

Under ***minimal*** supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties. ***Eligible for consideration of group leader status.***

Educational/Background Requirements

- Associates degree and ***7 or more years of experience in an applicable discipline***; or
- BS degree in Chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist III.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine/***non-routine*** maintenance ***and troubleshooting*** of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Assist other chemists and technicians with their professional development.

- Act as company advocate by setting a positive example in work habits and attitude to other staff members.

- Demonstrate ***increased*** ability to work independently with minimal errors.

- Capable of conducting peer review on routine ***and non-routine*** data packages. ***Has demonstrated knowledge to perform final data review and approval on LIMS.***

- Possess ***an above average*** level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- ***Assist in the development and maintenance of laboratory SOPs.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

CHEMIST IV

General Description

Under minimal supervision of the area manager and/***or the technical director***, conducts ***complex*** analyses on samples to determine their chemical and/or physical properties. Eligible for consideration of group leader status.

Educational/Background Requirements

- Associates degree and ***10*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***7*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- ***Ph.D. in Chemistry or a related field of science and experience in an environmental or related laboratory setting***.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist IV.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation ***of, and assisting other chemists in***, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.

- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other chemists and technicians with their professional development ***and in the integration of new methods and technologies***.

- Act as company advocate by setting a positive example in work habits and attitude to other staff members, ***prospective employees, existing and perspective clientele, and the general public***.

- Demonstrate ***superior*** ability to work independently with minimal errors.

- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.

- Possess ***a superior*** level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- ***Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.***

- ***When appropriate, work with the technical director to develop new methods and technologies***.

- ***Develop, review, and update laboratory SOPs as necessary***.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

CHEMIST V

General Description

Under minimal supervision of the area manager and/or the technical director, conducts complex analyses on samples to determine their chemical and/or physical properties. Eligible for consideration of group leader status. ***May work directly with the technical director to develop new methods and technologies for the laboratory***.

Educational/Background Requirements

- Associates degree and ***13*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and ***6*** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and ***2 or more years of*** experience in an environmental or related laboratory setting.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist V.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, assisting other chemists in, ***and serving as the primary reference for***, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.

- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other chemists and technicians with their professional development and in the integration of new methods and technologies.

- Act as company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.

- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.

- ***Responsible for the study and implementation of*** new methods and technologies.

- Develop, review, and update existing laboratory SOPs as necessary, ***write new SOPs as required to reflect advancements in methods and technologies***.

- ***Work with management team to plan for future equipment acquisitions***.

- ***Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions***.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

SENIOR CHEMIST

General Description

Working independently or under minimal supervision of, ***an*** area manager, technical director, ***or the laboratory president***, conducts ***or supervises analysis of complex non-routine projects*** to determine their chemical and/or physical properties. Eligible for consideration of group leader status.

Educational/Background Requirements

- BS degree in Chemistry or a related field of science and ***15*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and ***7*** or more years of experience in an environmental or related laboratory setting.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Senior Chemist.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, assisting other chemists in, and serving as the primary reference for, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other chemists and technicians with their professional development and in the integration of new methods and technologies.

- Act as company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.

- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.

- Responsible for the study and implementation of new methods and technologies.

- Develop, review, and update existing laboratory SOPs as necessary, write new SOPs as required to reflect advancements in methods and technologies.

- Work with management team to plan for future equipment acquisitions.

- Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST I

General Description

Under direct supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs.

Educational/Background Requirements

- Associates degree and 3 or more years of experience in an environmental or related laboratory setting; or

- BS degree in Chemistry or a related field of science.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist I.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).

- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.

- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.

- Review all final reports for accuracy and completeness.

- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".

- Follow all laboratory safety procedures.

- Prepare proposal outlines for existing clients.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST II

General Description

Under ***general*** supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs.

Educational/Background Requirements

- Associates degree and ***5*** or more years of experience in an ***applicable discipline;*** or
- BS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline;*** or
- ***MS degree in Chemistry or a related field of science.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist II.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness.
- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.
- Prepare proposal outlines for existing ***and new*** clients.
- ***Assist other project chemists and technicians with their professional development.***

- ***Act as a company advocate by setting a positive example in work habits and attitude to other staff members.***
- ***Demonstrate ability to work independently with minimal errors.***
- ***Posses the minimum level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.***
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST III

General Description

Under ***minimal*** supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs. ***Eligible for consideration of group leader status.***

Educational/Background Requirements

- Associates degree and ***7*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist III.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. ***Assist with the preparation, archiving, and delivery of a CLP or "CLP Like" deliverables package.***
- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.

- ***Prepare and/or coordinate the preparation of proposals for existing and new clients under direct supervision of the client services manager, sales manager, or laboratory president.***
- Assist other project chemists and technicians with their professional development.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members.
- Demonstrate ***increased*** ability to work independently with minimal errors.
- Posses ***an above average*** level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.
- ***Demonstrate ability to improve productivity as shown by an increase in project workload and throughput.***
- ***Provide data interpretation services to clients.***
- ***Assist in the development and maintenance of laboratory SOPs.***
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST IV

General Description

Under minimal supervision of the client services manager and/***or the sales manager,*** acts as the primary interface with the client to assure laboratory services are meeting client needs. ***May work directly with the sales manager to develop increased business from existing clients.*** Eligible for consideration of group leader status.

Educational/Background Requirements

- Associates degree and ***10*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***7*** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- ***Ph.D. in Chemistry or a related field of science and experience in an environmental or related laboratory setting.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist IV.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, ***and assist other project chemists with,*** incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. ***Coordinate*** the preparation, archiving, and delivery of CLP or "CLP Like" deliverables packages.

- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".

- Follow all laboratory safety procedures.

- Prepare and/or coordinate the preparation of proposals for existing and new clients under ***minimum*** supervision of the client services manager, sales manager, or laboratory president.

- Assist other project chemists and technicians with their professional development ***and in the integration of new methods and technologies.***

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members***, prospective employees, existing and perspective clientele, and the general public.***

- Demonstrate ***superior*** ability to work independently with minimal errors.

- Posses ***a superior*** level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput ***as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or "CLP Like" deliverables package and/or managing projects to specifications outlines in QAPPs.***

- Provide data interpretation services to clients.

- ***Develop, review, and update laboratory SOPs as necessary.***

- ***When appropriate, work with sales manager to develop additional business from existing clients.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST V

General Description

Under minimal supervision of the client services manager and/or the sales manager, acts as the primary interface with the client to assure laboratory services are meeting client needs. ***Works*** directly with the sales manager to ***establish relationships with new clients as well as increase*** business from existing clients. Eligible for consideration of group leader status.

Educational/Background Requirements

- Associates degree and ***13*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and ***6*** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and ***2 or more years of*** experience in an environmental or related laboratory setting.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist V.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, and assist other project chemists with, incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. Coordinate the preparation, archiving, and delivery of CLP or “CLP Like” deliverables packages.

- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".

- Follow all laboratory safety procedures.

- Prepare and/or coordinate the preparation of proposals for existing and new clients under minimum supervision of the client services manager, sales manager, or laboratory president. ***Take an active and substantial role on the marketing team in the development and coordination of large technical and cost proposals, qualifications packages, and marketing literature.***

- Assist other project chemists and technicians with their professional development and ***serve as the primary reference for*** the integration of new methods and technologies.

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Posses a superior level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or "CLP Like" deliverables package and/or managing projects to specifications outlines in QAPPs. ***Improve the productivity of others through training, assistance and the development and implementation of new, more efficient procedures.***

- Provide data interpretation services to clients. ***Assist clients in developing work plans or QAPPs by providing technical and administrative laboratory documentation and/or writing the laboratory portion of QAPPs.***

- Develop, review, and update laboratory SOPs as necessary. ***Write new SOPs as required to reflect advancements in procedures or technologies.***

- ***Routinely*** work with sales manager to develop additional business from existing clients ***and new clients.***

- ***Responsible for the study and implementation of new procedures and technologies.***

- ***Work with management team to plan for future equipment and software acquisitions.***

- ***Provide input to client services manager, sales manager, and/or laboratory president on personnel issues including performance reviews and staff additions / reductions.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

SENIOR PROJECT CHEMIST

General Description

Working independently or under minimal supervision of the client services manager and/or the sales manager***, or laboratory president,*** acts as the primary interface with the client to assure laboratory services are meeting client needs. Works directly with the sales manager to establish relationships with new clients as well as increase business from existing clients. ***Works directly with the laboratory president to develop the laboratory portion of QAPPs, work plans, and other technical documents.*** Eligible for consideration of group leader status.

Educational/Background Requirements

- BS degree in Chemistry or a related field of science and ***15*** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and ***7*** or more years of experience in an environmental or related laboratory setting.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Senior Project Chemist.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, and assist other project chemists with, incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. Coordinate the preparation, archiving, and delivery of CLP or “CLP Like” deliverables packages.

- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".

- Follow all laboratory safety procedures.

- Prepare and/or coordinate the preparation of proposals for existing and new clients under minimum supervision of the client services manager, sales manager, or laboratory president. Take an active and substantial role on the marketing team in the development and coordination of large technical and cost proposals, qualifications packages, and marketing literature.

- Assist other project chemists and technicians with their professional development and serve as the primary reference for the integration of new methods and technologies.

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Posses a superior level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or “CLP Like” deliverables package and/or managing projects to specifications outlines in QAPPs. Improve the productivity of others through training, assistance and the development and implementation of new, more efficient procedures.

- Provide data interpretation services to clients. Assist clients in developing work plans or QAPPs by providing technical and administrative laboratory documentation and/or writing the laboratory portion of QAPPs.

- Develop, review, and update laboratory SOPs as necessary. Write new SOPs as required to reflect advancements in procedures or technologies.

- Routinely work with sales manager to develop additional business from existing clients and new clients.

- Responsible for the study and implementation of new procedures and technologies.

- Work with management team to plan for future equipment and software acquisitions.

- Provide input to client services manager, sales manager, and/or laboratory president on personnel issues including performance reviews and staff additions / reductions.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN I

General Description

Under direct supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory.

Educational/Background Requirements

- High school diploma or equivalent.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician I.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN II

General Description

Under ***general*** supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory.

Educational/Background Requirements

- High school diploma or equivalent ***and 2 or more years of experience in an applicable discipline;*** or
- ***Associates degree and 1 or more years of experience in an applicable discipline;*** or
- ***BS degree in Chemistry or a related field of science***.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician II.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- ***Assist other technicians with their professional development.***
- ***Act as a company advocate by setting a positive example in work habits and attitude to other staff members.***

- ***Demonstrate ability to work independently with minimal errors.***
- ***Possess the minimum level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.***
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN III

General Description

Under ***minimal*** supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory. ***Eligible for consideration of group leader status.***

Educational/Background Requirements

- High school diploma or equivalent and ***4*** or more years of experience in an applicable discipline; or
- Associates degree and ***3*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***
- ***MS degree in Chemistry or a related field of science.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician III.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine/***non-routine*** maintenance ***and troubleshooting*** of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other technicians with their professional development.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members.
- Demonstrate ***increased*** ability to work independently with minimal errors.
- Possess ***an above average*** level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- ***Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput.***
- ***Assist in the development and maintenance of laboratory SOPs.***
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN IV

General Description

Under minimal supervision of the area manager and***/or the technical director***, performs ***complex*** tasks necessary for efficient operation of the laboratory. Eligible for consideration of group leader status.

Educational/Background Requirements

- High school diploma or equivalent and ***7*** or more years of experience in an applicable discipline; or
- Associates degree and ***5*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician IV.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation ***of, and assisting other technicians in,*** routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other technicians with their professional development ***and in the integration of new procedures and technologies.***

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, ***prospective employees, existing and prospective clientele, and the general public***.

- Demonstrate ***superior*** ability to work independently with minimal errors.

- Possess ***a superior*** level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput***, addition of new procedures/technologies and/or operation of additional equipment/instruments.***

- ***When appropriate, work with the technical director, laboratory president, or sales manager to develop new procedures and technologies.***

- ***Develop, review, and update laboratory SOPs as necessary.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN V

General Description

Under minimal supervision of the area manager and/or the technical director, performs complex tasks necessary for efficient operation of the laboratory. Eligible for consideration of group leader status. ***May work directly with the technical director, laboratory president, or sales manager to develop methods, procedures, and technologies for the laboratory.***

Educational/Background Requirements

- High school diploma or equivalent and ***10*** or more years of experience in an applicable discipline; or
- Associates degree and ***8*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***6*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or related field of science and ***4*** or more years of experience in an applicable discipline.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician V.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, and assisting other technicians in, ***and serving as the primary reference for,*** routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.

- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other technicians with their professional development and in the integration of new procedures and technologies.

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and prospective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput, addition of new procedures/technologies and/or operation of additional equipment/instruments.

- ***Responsible for the study and implementation of*** new procedures and technologies.

- Develop, review, and update laboratory SOPs as necessary***, write new SOPs as required to reflect advancement in procedures and technologies.***

- ***Work with management team to plan for future equipment acquisitions.***

- ***Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

SENIOR TECHNICIAN

General Description

Working independently or under minimal supervision of***, an*** area manager, technical director, ***or the laboratory president,*** performs ***or supervises tasks related to complex non-routine projects*** necessary for efficient operation of the laboratory. Eligible for consideration of group leader status.

Educational/Background Requirements

- High school diploma or equivalent and ***15*** or more years of experience in an applicable discipline; or
- Associates degree and ***13*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or related field of science and ***7*** or more years of experience in an applicable discipline.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Senior Technician.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, and assisting other technicians in, and serving as the primary reference for, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other technicians with their professional development and in the integration of new procedures and technologies.

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and prospective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput, addition of new procedures/technologies and/or operation of additional equipment/instruments.

- Responsible for the study and implementation of new procedures and technologies.

- Develop, review, and update laboratory SOPs as necessary, write new SOPs as required to reflect advancement in procedures and technologies.

- Work with management team to plan for future equipment acquisitions.

- Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

GROUP LEADER

General Description

In addition to the duties associated with the current chemist level, a group leader also takes on administrative responsibilities involved with the operation of the laboratory area.

Educational/Background Requirements

- Minimum of those specified with a Chemist III.

Minimum Required Skills and Responsibilities

Consistent with current Chemist Level, with additional or increased emphasis on the following requirements.

- Act as the area manager when the area manager is absent, filling such duties as supervision of employees and review and approval of data.
- Act as an additional source of information for management and others regarding laboratory area analysis capabilities.
- Responsible for the scheduling of work and the monitoring of workload for such items as hold times and due dates.
- Provide leadership, guidance, and training to other laboratory personnel on methods, equipment, and quality control.
- Develop, review and update laboratory SOPs as necessary.
- Assure that new methods, policies, and procedures are integrated into the laboratory area.
- Assume a primary responsibility for verifying that sample analyses are adhering to all method and laboratory specified quality assurance parameters.

Appendix B

TriMatrix Laboratories, Inc.

Inorganic Analyses

Parameter	Reference Citation
ACIDITY AS $CaCO_3$	SDM 2310 B
ALKALINITY, BICARBONATE	SDM 2320 B
ALKALINITY, CARBONATE	SDM 2320 B
ALKALINITY, HYDROXIDE	SDM 2320 B
ALKALINITY, PHENOLPHTHALEIN	SDM 2320 B
ALKALINITY, TOTAL	SDM 2320 B
BOD, (5-DAY)	SDM 5210 B
BOD, (5-DAY), DISSOLVED	SDM 5210 B
BOD, CARBONACEOUS (5-DAY)	SDM 5210 B
BROMIDE	USEPA 9056, ASTM D1246-88
CARBON DIOXIDE	SDM 4500-CO_2 C
CARBON, DISSOLVED ORGANIC	USEPA 9060, SDM 5310 D
CARBON, PURGEABLE ORGANIC	USEPA 9060
CARBON, TOTAL INORGANIC	USEPA 9060
CARBON, TOTAL ORGANIC	USEPA 9060, MSA 29.3.5.2, SDM 5310 D
CARBON,ORGANIC(NON-PURGE)	USEPA 9060
CATION EXCHANGE CAPACITY	USEPA-9081
CHEMICAL OXYGEN DEMAND	SDM 5220 D
CHLORIDE	SDM 4500-Cl B, USEPA 300.0/9056
CHLORINE, TOTAL RESIDUAL	HACH-8167
CHROMIUM, HEXAVALENT	SDM 3500-Cr D/USEPA 7196A
COLIFORM, FECAL	SDM 9222 D
COLIFORM, TOTAL	SDM 9223 B
COLOR (APPARENT)	SDM 2120 B
CONDUCTIVITY @ 25*C	USEPA-120.1/9050A, SDM 2510 B
CORROSION TOWARD STEEL	USEPA-1110
CYANIDE, AMENABLE	USEPA-9012A, SDM 4500-CN G
CYANIDE, FREE	USEPA-9014
CYANIDE, WEAK ACID DIS.	SDM-4500-CN I
CYANIDE,TOTAL	USEPA-335.4/9012A
DENSITY	SDM 2710 F
EXTRACTABLE ORGANIC HALIDES-EOX	USEPA-9023
FLUORIDE	USEPA-300.0/9056, SDM 4500-F C
FORMALDEHYDE	USEPA-8315A
GROUNDWATER DEPTH	USGS
GROUNDWATER LEVEL	USGS
HARDNESS, TOTAL	SDM 2340 C
HEM; OIL & GREASE	USEPA-1664/9070A/9071B
HETEROTROPHIC PLATE COUNT	SDM 9215 B
IGNITABILITY, SETAFLASH CLOSED-CUP	USEPA-1020A
IRON, FERRIC BY CALCULATION	SDM 3500-Fe D
IRON, FERROUS	SDM 3500-Fe D
NITROCELLULOSE	USARMY BR&D Lab
NITROGEN, AMMONIA	SDM 4500-NH_3 G
NITROGEN, INORGANIC (NH4)	SDM 4500-NH_3 G
NITROGEN, INORGANIC (NO3+NO2)	USEPA-353.2, SDM 4500-NO_3 F
NITROGEN, INORGANIC	USEPA-350.1 + 353.2
NITROGEN, NITRATE	USEPA-300.0/353.2/9056, SDM 4500-NO_3 F

TriMatrix Laboratories, Inc.

Inorganic Analyses

Parameter	Reference Citation
NITROGEN, NITRATE+NITRITE	USEPA-353.2, SDM 4500-NO_3 F
NITROGEN, NITRITE	USEPA-300.0/353.2/9056, SDM 4500-NO_2 B
NITROGEN, ORG. (NH4)	USEPA-350.1
NITROGEN, ORGANIC	USEPA-351.2
NITROGEN, TOTAL KJELDAHL	USEPA-351.2
ODOR	SDM 2150 B
OXYGEN, DISSOLVED	SDM 4500-O G
PAINT FILTER LIQUIDS TEST	USEPA-9095
PERCENT ASH	USEPA-160.4
PERCENT MOISTURE	SDM 2540 B
PERCENT SOLIDS	SDM 2540 B
PERCENT VOLATILE SOLIDS	USEPA-160.4, SDM 2540 G
PH	USEPA-150.1/9040B/9045C
PHENOLICS, TOTAL	USEPA-420.1/B17420.2/9066
PHOSPHORUS, ORTHO	SDM 4500-P E
PHOSPHORUS, TOTAL	USEPA-365.1, SDM 4500-P F
PHOSPHORUS, TOTAL-SOLUBLE	USEPA-365.1, SDM 4500-P F
RESIDUE, DISSOLVED @ 180C	SDM 2540 C
RESIDUE, DISSOLVED-VOL.	USEPA-160.4
RESIDUE, SUSPENDED	SDM 2540 D
RESIDUE, SUSPENDED-VOL.	USEPA-160.4
RESIDUE, TOTAL	SDM 2540 B
RESIDUE, TOTAL-VOLATILE	USEPA-160.4, SDM 2540 G
SGT-HEM; NON-POLAR MATERIAL	USEPA-1664/9070A/9071B
SILICA, DISSOLVED	SDM 4500-$Si0_2$ D
SODIUM HEXAMETAPHOSPHATE	USEPA-365.1
SPECIFIC GRAVITY	ASTM-D 1429-79, SDM 2710 F
STATIC WATER LEVEL	USGS
SULFATE	USEPA-300.0/375.2/9056/9038, SDM 4500-$S0_4$ F
SULFIDE	USEPA-9034, SDM 4500-S_2 F
SULFIDES, ACID VOLATILE	ET&C VOL 12
SULFITE	SDM 4500-SO_3 B
SURFACTANTS, MBAS	SDM 5540 C
TEMPERATURE	SDM 2550 B
THIOCYANATE	SDM 4500-CN M
TOTAL ORGANIC HALIDES	USEPA-9020B/9023
TURBIDITY	SDM 2130 B

TriMatrix Laboratories, Inc.

Metals Analyses

Parameter	Reference Citation
ALUMINUM, ICP	USEPA-200.7/6010B
ANTIMONY, ICP	USEPA-200.7/6010B
ANTIMONY, MS	USEPA-200.8/6020
ARSENIC, ICP	USEPA-200.7/6010B
ARSENIC, MS	USEPA-200.8/6020
BARIUM, ICP	USEPA-200.7/6010B
BARIUM, MS	USEPA-200.8/6020
BERYLLIUM, ICP	USEPA-200.7/6010B
BERYLLIUM, MS	USEPA-200.8/6020
BORON, ICP	USEPA-200.7/6010B
BORON, MS	USEPA-200.8/6020
CADMIUM, ICP	USEPA-200.7/6010B
CADMIUM, MS	USEPA-200.8/6020
CALCIUM AS $CaCO_3$	USEPA-200.7/6010B
CALCIUM, ICP	USEPA-200.7/6010B
CHROMIUM, ICP	USEPA-200.7/6010B
CHROMIUM, MS	USEPA-200.8/6020
COBALT, ICP	USEPA-200.7/6010B
COBALT, MS	USEPA-200.8/6020
COPPER, ICP	USEPA-200.7/6010B
COPPER, MS	USEPA-200.8/6020
HARDNESS BY CALCULATION, ICP	USEPA-200.7/6010B
IRON, ICP	USEPA-200.7/6010B
LEAD, ICP	USEPA-200.7/6010B
LEAD, MS	USEPA-200.8/6020
LITHIUM, ICP	USEPA-200.7/6010B
MAGNESIUM AS $CaCO_3$, ICP	USEPA-200.7/6010B
MAGNESIUM, ICP	USEPA-200.7/6010B
MANGANESE, ICP	USEPA-200.7/6010B
MANGANESE, MS	USEPA-200.8/6020
MERCURY, COLD VAPOR	USEPA-245.1/7470A/7471A
MOLYBDENUM, ICP	USEPA-200.7/6010B
MOLYBDENUM, MS	USEPA-200.8/6020
NICKEL, ICP	USEPA-200.7/6010B
NICKEL, MS	USEPA-200.8/6020
PHOSPHORUS, ICP	USEPA-200.7/6010B
POTASSIUM, ICP	USEPA-200.7/6010B
SELENIUM, ICP	USEPA-200.7/6010B
SELENIUM, MS	USEPA-200.8/6020
SILICON, ICP	USEPA-200.7/6010B
SILVER, ICP	USEPA-200.7/6010B
SILVER, MS	USEPA-200.8/6020
SODIUM, ICP	USEPA-200.7/6010B
STRONTIUM, TOTAL	USEPA-200.7/6010B
THALLIUM, ICP	USEPA-200.7/6010B
THALLIUM, MS	USEPA-200.8/6020
TIN, ICP	USEPA-200.7/6010B
TIN, MS	USEPA-200.8/6020
TITANIUM, ICP	USEPA-200.7/6010B

TriMatrix Laboratories, Inc.

Metals Analyses

Parameter	Reference Citation
VANADIUM, ICP	USEPA-200.7/6010B
VANADIUM, MS	USEPA-200.8/6020
ZINC, ICP	USEPA-200.7/6010B
ZINC, MS	USEPA-200.8/6020

TriMatrix Laboratories, Inc.

Semi-Volatile Organic Analyses

Parameter	Reference Citation
HPLC ACRYLAMIDE	EPA-8316
GC ORGANOCHLORINE PESTICIDES	USEPA-608/8081A
GC METHOXYCHLOR	USEPA-608.2
HPLC POLYNUCLEAR AROMATIC HYDROCARBONS	USEPA-610/8310
GC/MS BASE/NEUTRAL/ACIDS	USEPA-625/8270C
GC ANALYSIS OF 1,2-DIBROMOMETHANE/ 1,2-DIBROMO-3-CHLOROPROPANE/ 1,2,3-TRICHLOROPROPANE BY MICROEXTRACTION	USEPA-8011
GC DIESEL RANGE ORGANICS	USEPA-8015B, CALIFORNIA LUFT METHOD, WISCONSIN METHOD PUBL-SW-141
GC GLYCOLS	USEPA-8015B
GC POLYCHLORINATED BIPHENYLS	USEPA-8082
GC CHLORINATED HYDROCARBONS	USEPA-8121
GC HERBICIDES	USEPA-8151A
HPLC ALDEHYDES	USEPA-8315A
HPLC NITROAROMATICS AND NITRAMINES	USEPA-8330
HPLC NITROGLYCERINE	USEPA-8332

TriMatrix Laboratories, Inc.

Volatile Organic Analyses

Parameter	Reference Citation
GC GASOLINE RANGE ORGANICS	USEPA-8015B, CALIFORNIA DHS LUFT, IOWA-PA1, WISCONSIN METHOD PUBL-SW-140
GC AIR ANALYSIS	40CFR METHOD 18
GC DISSOLVED HEADSPACE ANALYSIS OF METHANE/ETHANE/ETHYLENE	RSK-175
GC ALCOHOLS	USEPA-8015B
GC VOLATILE ORGANICS	USEPA-601/602/8021B
GC/MS VOLATILE ORGANICS	USEPA-524.2/624/8260B

Appendix C

TriMatrix Laboratories, Inc.

Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
190	Administration	Sonicator - Fisher	F550,# F1520			
200	Administration	Sonicator - Fisher	F550,# F1309			
209	Administration	Mettler PC4400 Toploading Balance				
212	Administration	Mettler AB204 Analytical Balance				
215	Administration	Denver Instrument P-4002 Toploading Balance				
225	Administration	Dionex Accelerated Solvent Extractor	Model ASE 300	8/2003	3070313	New
300	Administration	Fisher Ultrasonic Cleaner	FS110			
301	Administration	Fisher Heated Ultrasonic Cleaner	FS21H			
307	Administration	Denver Instruments Top Loading Balance	Model P-2002	1/2007	P2K2126010	New
317	Client Services	YSI Multi-Parameter Meter	Model 556MPS	2005	05G1614AM	New
318	Client Services	YSI Multi-Parameter Meter	Model 556MPS	2004	04C2296AE	New
319	Client Services	HACH Turbidimeter	Model 2100P		30300030404	New
320	Client Services	Fisher Accumet Multi-Parameter Meter	Model AP84		274436	New
100	Wet Chemistry	Orion pH/ISE Meter	8102			
120	Wet Chemistry	Ultraviolet Spectrophotometer Shimadzu	1601			
161	Wet Chemistry	Auto-analyzer Lachat Quick Chem				
164	Wet Chemistry	pH/ISE meter Orion	710A			
165	Wet Chemistry	Expandable Ion Analyzer Orion Research	EA920			
167	Wet Chemistry	Spectrophotometer (UV-VIS) Shimadzu	1201			
171	Wet Chemistry	Polarograph EG&G Princeton Applied Research	384B			
176	Wet Chemistry	Field Meter				
177	Wet Chemistry	Mettler AE200 Analytical Balance				
178	Wet Chemistry	Turbidimeter Hach	2100N			
186	Wet Chemistry	Koehler Rapid Tester Flashpoint Tester	RT-1			
187	Wet Chemistry	Mettler DL12 Auto-Titrator				
188	Wet Chemistry	YSI Conductivity Meter	3200			
189	Wet Chemistry	Lachat	FIA-8000			
194	Wet Chemistry	Total Organic Halogen: ThermoGlass	1200			
196	Wet Chemistry	Lachat IC	8000			
198	Wet Chemistry	Total Organic Carbon Analyzer, OI Analytical	1010			
205	Wet Chemistry	OHAUS TP4KD Toploading Balance				
206	Wet Chemistry	Mettler BB600 Toploading Balance				
207	Wet Chemistry	Denver Instrument A-250 Analytical Balance				
208	Wet Chemistry	Mettler AE163 Analytical Balance				

TriMatrix Laboratories, Inc.

Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
210	Wet Chemistry	Mettler AE163 Analytical Balance				
298	Wet Chemistry	Konelab Automated Ultraviolet Spectrophotometer	Model Aqua 20			
299	Wet Chemistry	OIC Available Cyanide Analyzer				
303	Wet Chemistry	Konelab Automated Ultraviolet Spectrophotometer	Model 20	1/2006	24618583	Refurb
305	Wet Chemistry	Orion 3-Star Benchtop DO Meter	Model 1113000	2/2006	7383	New
306	Wet Chemistry	Dionex Ion Chromatograph	Model ICS-2000	3/2006	6020239	New
309	Wet Chemistry	pH Meter, Fisher Accumet Basic	Model AB15	3/28/2007	AB92325491	New
310	Wet Chemistry	Market Forge Autoclave	Model STM-E	3/30/2007	226071	New
313	Wet Chemistry	HACH Turbidimeter	Model 2100N	8/1/2008	07060C022389	New
314	Wet Chemistry	VWR Forced Air Oven	Model 1370FM	10/3/2007	4104307	New
315	Wet Chemistry	Thermo Scientific TOX Analyzer	Model ECS 1200	10/18/2007	2003.481	New
321	Wet Chemistry	BW Technologies	Gas Alert Micro		Propoerty of Kent Couty DPW	
322	Wet Chemistry	Chemetrics VVR	Photometer		5121	
324	Wet Chemistry	OI Analytical TOC Analyzer	Aurora Model 1030	2/2008	E750730372E	New
326	Wet Chemistry	OI Analytical Automated Chemistry Analyzer	Flow Solution 3100 (322689/323898)	9/2008	821831887/826833549	New
198T	Wet Chemistry					
305a	Wet Chemistry	Orion DO Probe	Model 081010MD	2/2006	Lot Number RJS16	New
312a	Wet Chemistry	HACH Portable Multi-meter (pH/Cond/Sal/TDS/LDO)	Model HQ40d	7/1/2007	70700010664	New
312b	Wet Chemistry	HACH Portable Multi-meter (pH/Cond/Sal/TDS/LDO)	Model HQ40d	7/1/2007	70700010664	New
324T	Wet Chemistry	OI Analytical TOC Analyzer	Aurora Model 1030	6/17/2008		Loaner
101	Metals	ICP Spectrophotometer PE	Optima 3000			
106	Metals	Atomic Absorption Spectrophotometer Furnace	3			
114	Metals	ICP Mass Spectrometer	ELAN 6000			
116	Metals	Perkin Elmer Optima Trace ICP	3300 DV			
201	Metals	ICP Mass Spectrometer	ELAN 6100			
202	Metals	PSA Low-Level Mercury Analyzer	Millenium System			
203	Metals	A&D FX-2000 Toploading Balance				
211	Metals	Mettler PB1502 Toploading Balance				
216	Metals	PSA Cold Vapor AA Mercury Analyzer	Millenium System			
217	Metals	Env. Express Hotblock, CS154		2000	424CEC0564	
218	Metals	Env. Express Hotblock, CS154		2000	944CEC1008	
219	Metals	Env. Express Hotblock, CS154		2002	1423CEC1147	
220	Metals	Env. Express Hotblock, CS154		2002	1423CEC1113	
311	Metals	Perkin Elmer Optima ICP-OES	Model 5300DV	5/7/2007	077C7032601	New

TriMatrix Laboratories, Inc.

Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
316	Metals	Mettler Analytical Balance	Model XS204	12/21/2007	1128261601	New
144	Semivolatiles GC	Gas Chromatograph (Dual ECD)	HP-5890A			
151	Semivolatiles GC	Liquid Chromatograph Perkin Elmer	PDA 235/240 HPLC			
157	Semivolatiles GC	Gas Chromatograph HP	5890A (FID)			
158	Semivolatiles GC	Gas Chromatograph HP	5890A (ECD)			
159	Semivolatiles GC	Gas Chromatograph Varian	3400 (FID)-SV			
174	Semivolatiles GC	Gas Chromatograph HP	5890 (ECD/FID)			
199	Semivolatiles GC	Gas Chromatograph HP-6890 Dual ECD				
221	Semivolatiles GC	Perkin-Elmer 200 LC Plus HPLC				
222	Semivolatiles GC	Agilent 6890 Dual ECD				
325	Semivolatiles GC	Clarus Gas Chromatograph	Model 500	7/2008	5564	New
133	Semivolatiles MS	GC/MS Varian Ion Trap	Saturn II			
138	Semivolatiles MS	GC/MS Varian Ion Trap	Saturn II			
195	Semivolatiles MS	HP 5973 Quadrupole Mass Spectrometer				
304	Semivolatiles MS	MS = Agilent MSD	Model 5975B	9/7/2006	MS = US60522528 GC = HP 6890N SN US10626080	New
308	Semivolatiles MS	MS = Agilent MSD	Model 5975B	2/2007	US65125179 GC = HP 6890N SN CN10703067	New
117	Volatiles GC	Agilent PID/ELCD GC	6890			
140	Volatiles GC	HP GC	5890 Series II			
142	Volatiles GC	HP GC	5890 Series II			
132	Volatiles MS	GC/MS Varian Ion Trap	Saturn II			
139	Volatiles MS	HP Quadrupole GC/MS	5971			
145	Volatiles MS	HP Quadrupole GC/MS	GC-5890/MS-5971			
197	Volatiles MS	HP 5973 Quadrupole GC/MS				
204	Volatiles MS	Mettler BB2440 Toploading Balance				
224	Volatiles MS	Agilent 5973 Inert MSD				
302	Volatiles MS	Branson Heated Ultrasonic Cleaner	3210R-MTH			
323	Volatiles MS	Agilent GC/MS	Model 6890/5973 Inert	2/21/2008	CN10426060/US35120404	New

Appendix D

TriMatrix Laboratories, Inc.

New Employee Orientation Checklist

Name: _____ Employee #: _____ Date of Hire: ____/____/____

I. Personnel Information Review (Human Resources Manager)

Reviewed (☑)	Item
	Employee Information Sheet Completed
	I-9 Employment Eligibility Verification Form Completed
	W-4 Forms Completed
	Employee Benefits Reviewed
	Direct Deposit Forms Initiated
	Details of Compensation Reviewed
	Key Fob to the Facility Provided (Number _____)
	Employee Handbook Distributed
	Code of Ethics / Data Integrity Policy Agreement Form Signed and Collected. Violation of Ethics Policy Explained.

Signatures below attest that all the information or items described above have been discussed/provided:

____/____/____ _____ ____/____/____ _____

Human Resources Manager Signature Employee Signature

TriMatrix Laboratories, Inc.

New Employee Orientation Checklist

II. Quality Assurance Training (Quality Assurance Officer)

Reviewed (☑)	Item
	Initial and Continuing Demonstration of Capability Requirements Reviewed
	Corrective Action (Non-Conformance) Investigation Procedure Reviewed
	Error Correction Policy Reviewed
	Code of Ethics/Data Integrity Policies Explained
	Initials Added to the Initials Logbook
	Training Forms Initiated for the Following Documents: QA Manual Corrective Action SOP, GR-10-106 or GR-03-101 or GR-03-124 Manual Integration SOP, GR-10-115 General Guidelines for Data Validation and Reporting, GR-10-103 Internal Chain-of-Custody, GR-10-104 Data Confidentiality, GR-10-118

Signatures below attest that all the information or items described above have been discussed/provided:

____/____/____ _____ ____/____/____ _____

Quality Assurance Officer Signature Employee Signature

TriMatrix Laboratories, Inc.

New Employee Orientation Checklist

III. Safety Training (Health and Safety Officer)

Reviewed (☑)	Item
	MSDS Location Discussed
	Safety Walk/Safety Equipment Review, First-Aid Cabinet Locations Identified
	Safety Exam Explained-First two of thirteen videos completed (others to be completed on own during normal working hours)
	Training Forms Initiated for the Following Documents: Chemical Hygiene Plan Safety Manual Copy Emergency Action Plan Copy
	Safety Glasses Ordered or Distributed

Signatures below attest that all the information or items described above have been discussed/provided:

____/____/____ _____ ____/____/____

Appendix E

TriMatrix Laboratories, Inc.

CODE OF ETHICS / DATA INTEGRITY AGREEMENT

All full time, part time and contracted employees working for TriMatrix Laboratories, Inc. are required to make every effort to conduct quality work with data integrity, ethical practices and professionalism. To ensure strength in the individual, in the laboratory organization and in client relationships, each employee must be aware of the following company policies:

I. Each TriMatrix employee is responsible for the propriety and consequences of his or her actions when representing the laboratory through sample analysis, data review, adherence to policies and procedures, client /vender relationships, other employees and/or visitors.

II. All aspects of company business must be conducted in an ethical, legal and professional manner, and in compliance with all applicable federal, state and local laws and regulations.

III. Under no circumstances must client confidentiality be compromised or any information regarding the client be revealed to another agency without the client's prior written permission.

IV. Gratuities, gifts and/or rewards provided by clients or vendors are laboratory property and may not be kept for personal use without written approval.

V. Reporting of data integrity issues is encouraged. Reporting shall be kept confidential when anonymity is requested and/or required.

Additionally, violations of the data integrity/code of ethics policy may result in immediate termination of employment with TriMatrix Laboratories, Inc. Such violations include the following:

A. Intentionally misrepresenting laboratory data in any manner.

B. Intentionally misapplying any date and/or time.

C. Intentional representation of another employee without written approval.

D. Intentional omission of any information, fact or datum.

E. Intentional deviation from or shortcut through a procedure without written approval.

A highly ethical approach to laboratory analysis/reporting is a key component of the TriMatrix laboratory objective. This approach is backed by management in providing the facilities, equipment and time necessary minimize undue pressures to make compromises, whether such pressures be internal or external.

AGREEMENT STATEMENT

I have read and understood the Code of Ethics/Data Integrity Agreement, and agree to abide by all policies stated. I understand that violation of these policies may result in severe consequences up to and including termination of my employment with TriMatrix Laboratories, Inc.

_____	_____	_____
Employee (print name)	Signature	Date

Appendix F

TriMatrix Laboratories, Inc.

New Instrument Accuracy Study

Instrument Number: ______________ **Method Reference:** ______________________ **Analyst:** ______________ **Date Analyzed:** ______________

Compound	Analysis #1		Analysis #2		Analysis #3		Analysis #4		Analysis #5		Analysis #6		Analysis #7	
	Conc.:		Conc.:		Conc.:		Conc.:		Conc.:		Conc.:		Conc.:	
	Units:		Units:		Units:		Units:		Units:		Units:		Units:	
	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery

TriMatrix Laboratories, Inc.

New Instrument Accuracy Study

Instrument Number: _____ **Method Reference:** _____ **Analyst:** _____ **Date Analyzed:** _____

Compound	Standard Deviation of Percent Recoveries	Standard Deviation Window	Pass/ Fail Standard Deviation	Average Percent Recovery	Percent Recovery Window	Pass/ Fail Percent Recovery	Overall Pass/ Fail

Appendix G

TriMatrix Laboratories, Inc.

New Instrument Information and Initial Demonstration of Capability

Item:		**Serial Number:**	
Manufacturer:		**Date Received:**	
Model:		**Location:**	

Initial Demonstration of Capability Passed: Yes / No / NA

Date Initial Demonstration of Capability Completed:

Initial Demonstration of Capability Data Attached: Yes / No / NA

Adequate Sensitivity Achieved (LFB or MDL Completed): Yes / No / NA

LFB or MDL Documentation Attached: Yes / No / NA

Date LFB or MDL Completed:

Linear Range Developed and Demonstrated Yes / No / NA

Linear Range Development Information Attached: Yes / No / NA

Notes:

Approvals and Assigned Instrument Number

Quality Assurance Manager	**Laboratory Area Manager**

TriMatrix Instrument Number: **Date In Service:**

Appendix H

TriMatrix Laboratories, Inc.

********** LABORATORY
DEMONSTRATION OF CAPABILITY FOR **********

Parameter	Date Analyzed	Method	Inst. Number	Units	Amount Spiked	Cert. #1 Amount Found	Cert. #2 Amount Found	Cert. #3 Amount Found	Cert. #4 Amount Found	Average Percent Recovery	Percent Recovery Window	Pass/Fail Percent Recovery	Percent RSD	Percent RSD Window	Percent RSD Pass/Fail	Overall Pass/ Fail

Appendix I

TriMatrix Laboratories, Inc.

INORGANIC LABORATORY
DEMONSTRATION OF CAPABILITY

Parameter: Percent Solids

Trainer: John Doe

Method: SW-846 3550B/GR-07-115

Trainee: John Smith

Analyst	Date	Run #1	Run #2	Run #3	Run #4	Units	Inst. #	Standard Deviation	Average	Degrees of Freedom D	Experimental Student's t Value	Tabular Student's t Value	Are the Two Sets of Results Statistically the Same AND RSDs<20?
John Doe	12/31/02	48.3	55.6	44.2	47.5	%	117	4.81	48.9	5.48	0.227	4.032	YES(PASS)
John Smith	12/31/02	45.9	50.2	52.1	44.7	%	117	3.50	48.2				

Appendix J

TriMatrix Laboratories, Inc.

NELAC Demonstration of Capability Certification Statement

Employee Name:_____ **Date:**_____

Method Name(s), Number(s), and Revision(s):

_____

Matrix:_____ **Analyte(s) or Parameter(s):**_____

SOP Number:_____ **Revision Number:**_____

We, the undersigned, CERTIFY that:

Yes / NA	
☐☐	1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.
☐☐	2. The test method(s) was performed by the analyst identified on this certification.
☐☐	3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.
☐☐	4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

With *true* meaning consistent with supporting data; *accurate* meaning based on good laboratory practices consistent with sound scientific principles/practices; *complete* meaning includes the results of all supporting performance testing; and *self explanatory* meaning data properly labeled and stored so that the results are clear and require no additional explanation.

☐☐	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 assessors.

This certification form must be completed each time an Initial Demonstration of Capability study is performed, or when a Continuing Demonstration of Capability study is performed in conjunction with a revised SOP.

_____ _____

Area Supervisor
Heather L. Brady — Date

_____ _____

Quality Assurance Department
Tom C. Boocher — Date

Appendix K

TriMatrix Laboratories, Inc.

LABORATORY TRAINING CHECKLIST

Employee Name: _____

Instructor Name: _____

Method Number(s) and Revision(s): _____

SOP Name, Number, and Revision: _____

Applicable Matrices: _____

n/a	Trainer/Trainee Initials		CheckPoint Item
❑			1) The employee has read the method and the standard operating procedure.
❑			2) The instructor has reviewed the method and the procedure with the employee.
❑			3) The instructor has performed a manual demonstration of the procedure.
❑			4) The employee has correctly performed the procedure under direct supervision.
❑			5) The employee has correctly performed the procedure without direct supervision.
❑			6) The employee has successfully and exclusively completed an Initial Demonstration of Capability (IDC).
❑			7) The DoC spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
☒	-------	-------	8) If applicable, or a MDL study does not yet exist, the employee has successfully completed a MDL study for all applicable matrixes.
☒	-------	-------	9) The MDL study spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
❑			10) The employee has been instructed in the QA/QC requirements of this procedure.
❑			11) The employee has been instructed in the proper procedure governing paperflow, benchsheet completion, and other relevant documentation requirements.
❑			12) NELAC Demonstration of Capability Certification Statement is Attached.

The required CheckPoints have been successfully completed, and in my opinion this employee has been adequately trained to correctly perform this procedure.

Instructor: _____ **Date:** _____

I have read and understand the SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.

Employee: _____ **Date:** _____

Appendix L

TriMatrix Laboratories, Inc.

INORGANIC/METALS/SEMI-VOLATILE/VOLATILE LABORATORY
2008 WATER/SOIL
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Instrument Number	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1 Amount Found	Rep. #2 Amount Found	Rep. #3 Amount Found	Rep. #4 Amount Found	Rep. #5 Amount Found	Rep. #6 Amount Found	Rep. #7 Amount Found	Average Amount Found	Average % Recovery	Standard Deviation	MDL

This Page For Lab Use Only

TriMatrix Laboratories, Inc.

This Page For Lab Use Only

INORGANIC/METALS/SEMI-VOLATILE/VOLATILE LABORATORY
2008 WATER/SOIL
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Average Amount Found	Average % Recovery	Standard Deviation	MDL	Amount Spiked	MDL Window	Pass / Fail	Average % Recovery Check	Minimum Report Limit
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
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							Missing Parameter / Compound		
							Missing Parameter / Compound		

TriMatrix Laboratories, Inc.

INORGANIC/METALS/SEMI-VOLATILE/VOLATILE LABORATORY
2008 WATER/SOIL
METHOD DETECTION LIMIT VERIFICATION STUDY

Parameter / Compound	Instrument Number	Reference Citation	MDL Result	Units	Date	Analyst	Verification Concentration	MDL/MDL Verification Concentration Difference	MPB Result	MDL Verification Result	MPB/MDL Verification Response Increase	Acceptable (≥3x MPB Response)?

file: MDL 2008 version 10.XLS

revision: 2008.10

Appendix M

TriMatrix Laboratories, Inc.

SOP MAJOR REVISION
LABORATORY TRAINING CHECKLIST

Employee Name: _____

Method Number(s) and SOP Name, Number, and Revision: _____

Applicable Matrices: _____

n/a	Employee Initials	CheckPoint Item
❑		1) I have read the updated method and/or the revised Standard Operating Procedure.
❑		2) I have successfully completed an Initial Demonstration of Capability (IDC).
❑		3) The DoC spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
❑		4) If applicable, or a MDL study does not yet exist, I have successfully completed a MDL study for all applicable matrixes.
❑		5) The MDL study spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
❑		6) I have been instructed in any new QA/QC requirements of this procedure.
❑		7) NELAC Demonstration of Capability Certification Statement is Attached.

The required CheckPoints have been successfully completed.

Date: _____ **Quality Assurance:** _____

I have read and understand the revised SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.

Date: _____ **Employee Signature:** _____

TriMatrix Laboratories, Inc.

SOP MINOR REVISION
LABORATORY TRAINING CHECKLIST

Employee Name: _____

Method Number(s) and Revision(s): _____

SOP Name, Number, and Revision: _____

Applicable Matrices: _____

n/a	Employee Initials	CheckPoint Item
❑		1) I have read and understood the updated method and/or the revised Standard Operating Procedure.
❑		2) I have read and understood any new QA/QC requirements of this procedure.
❑		3) NELAC Demonstration of Capability Certification Statement is Attached.

I have read and understand the revised SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.

Date:_____ **Employee Signature:**_____

The SOP revision has been successfully implemented.

Date:_____ **QA/QC Signature:**_____

Appendix N

TriMatrix Laboratories, Inc.

Container Packing List

For any questions regarding these containers, contact a Project Chemist at (616) 975-4500.

Client: Project:

#	Sets	Sample Locations	Sample Container Types and Quantities Requested																									
			0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26
1																													
2																													
3																													
4																													
5																													
6																													
7																													
8																													
9																													
10																													
11																													
12																													
13																													
14																													
15																													
16																													
17																													
18																													
19																													
20																													
Total Containers																													

This container type requires field-filtering

MATRIX	#	TEST	SIZE (mL) / TYPE CONTAINER	OPTIONS	PRESERVATIVE	TAG COLOR
WATER	0	Unpreserved Purgeable Organics	40 mL Clear Glass Vial	40	Cool to 4° C	Yellow & Black Stripe
	1	Preserved Purgeable Organics	40 mL Clear Glass Vial (pre-preserved)	40	HCl; Cool to 4° C	Yellow
	2	Non-Purgeable Organics	1000 mL Amber Glass	1000	Cool to 4° C	Salmon
	3	General Short Hold	Plastic	125, 250, 500, 1000	Cool to 4° C	Green
	4	Nutrients	Plastic	125, 250, 500, 1000	pH <2 w/ H_2SO_4	Dark Blue
	5	Cyanides	500 mL Amber Plastic	500	pH >12 w/ NaOH	Light Blue
	6	Total Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red
	7	Oil & Grease/TPH	Clear Glass	1000WM, 1000NM	pH <2 w/ H_2SO_4	Dark Blue
	8	Bacteria	125 mL Plastic (pre-preserved)	125	$Na_2S_2O_3$; Cool to 4° C	Pre-Labeled (White)
	9	Sulfide	500 mL Amber Glass + NaOH ampule	500	Zinc Acetate at Lab; NaOH in Field	Light Green
	10	TOX	250 mL Amber Glass **w/ Septa Lid**	250	pH <2 w/ H_2SO_4	Lilac
	11	TOC	40 mL Amber Vial	40	pH <2 w/ H_2SO_4	Pink
	12	DRO	1000 mL Amber Glass	1000	pH <2 w/ HCl	Gray
	13	Phenols	500 mL Amber Glass	500	pH <2 w/ H_2SO_4	Brown
	14	Formaldehyde	250 mL Amber Glass	250	Cool to 4° C	Orange
	15	Dissolved Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red & White Stripe
SOIL	16	Inorganics/Metals	WM Plastic	125, 250, 500, 1000	Cool to 4° C	White
	17	Non Purgeable Organics	WM Clear Glass	125, 250, 500, 1000	Cool to 4° C	Manila
	18	Purgeable Organics - Bulk	60 mL WM Clear Glass	60	Cool to 4° C	Light Yellow
	19	TCLP Volatiles	125 mL Clear Glass Vial	125	Cool to 4° C	Yellow & Black Stripe
	20	% Solids	125 mL WM Plastic	125	Cool to 4° C	Yellow & White Stripe
	21	Purgeable Organics	Encore Sampler	5g, 25g	Cool to 4° C	Label on Bag
	22	Purgeable Organics - PrePres.	40 mL Pre-Tared Clear Glass Vial + 10 mL MeOH ampule	40	MeOH in field; Cool to 4° C	Pre-Labeled (Light Yellow added at Lab)
MISC	23					
	24					
	25	Pesticide WWs by Method 608	1000 mL Amber Glass	1000	pH 5-9; Cool to 4° C	Yellow & White Stripe
	26	Drinking Water Volatiles	40 mL Clear Glass Vial	40	Ascorbic Acid at Lab; HCl in Field	Yellow

Notes:

DI Water for Equipment Blanks	Container Type and Size	Qty.
VOC Free		
Millipore		
ASTM Metals Free		

TriMatrix Laboratories, Inc.

Project Chemist Initials	Added to Calendar & Folders (initials/date)	Revision:	Revised By/Date:

Client: _____ Project Manager: _____

Project: _____ Contact: _____

TriMatrix Project No: _____ Date of Request: _____

Type of Order: ○ One-Time ⇨ Due to Client: _____ ○ AM ◉ PM

or

◉ Calendar ⇨ Frequency: ○ Weekly ○ Semi-Annually ○ Monthly ○ Annually ○ Quarterly ◉ Daily

Prepare Containers For:

Months	☐ Jan	☐ Feb	☐ Mar	☐ Apr	☐ May	☐ Jun
	☐ Jul	☐ Aug	☐ Sep	☐ Oct	☐ Nov	☐ Dec
Weeks	☐ 1	☐ 2	☐ 3	☐ 4	☐ 5	
Days	☐ M	☐ T	☐ W	☐ TH	☐ F	

Containers will be ◉ Picked Up or ○ Shipped via: ○ First Overnight ◉ Standard Overnight

Pick up/Ship Date: _____ ○ Priority Overnight ○ Express Saver

Ship Containers to: ○ 2-Day ○ Ground

_____ ○ Saturday Delivery ○ TriMatrix Courier

_____ ○ Other: _____

_____

_____ ☐ Shipment to be billed to FedEx Account No.:

Telephone No: _____

Shipment to include: ☐ COCs (Qty) _____ ☐ Custody Seals ☐ Temperature Blanks

☐ MSDS Sheets for all preservatives used ☐ WB TM#? ○ Y ◉ N

Comments: ☐ Cooler Banding Required

_____

_____

_____

Assembled by/Date:	Checked by/Date:	Shipped by/Date:

Cooler Number(s) Used:	Coolers Sealed With Tape	Coolers Sealed With Banding Strap	Tracking Number Label(s):
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	

Appendix O

Sample Receipt Record

TriMatrix Laboratories, Inc.

Date: _____

Delivery Method A: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method B: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method C: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method D: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

TriMatrix Courier (TC): _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Page/ Line Number	Client	Quantity of Coolers OR TriMatrix Cooler Number	Arrived in Laboratory					Submittal Number (Project Chemist)	Folder Prepared (Log-In ✓)
			Time	AM	PM	Received By	Delivery Method Letter		
1-1									
1-2									
1-3									
1-4									
1-5									
1-6									
1-7									
1-8									
1-9									
1-10									
1-11									
1-12									
1-13									
1-14									
1-15									
1-16									
1-17									
1-18									
1-19									
1-20									
1-21									

Appendix P

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST

Client	Project-Submittal No. new / add to
Receipt Record Page/Line No.	Project Chemist / Sample Nos.

Coolers Received

Recorded by (initials/date)	☐ Cooler ☐ Box ☐ Other_____	Qty Received	Thermometer Used: ☐ IR Gun (#202) ☐ Digital Thermometer (#54) ☐ Other (#_____)	☐ See Additional Cooler Information Form

Cooler No.	Time	Cooler No.	Time	Cooler No.	Time	Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location: Dispersed / Top / Middle / Bottom

Coolant/Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

If any shaded areas checked, complete Sample Receiving Non-Conformance Form

Paperwork Received

☐ **No COC received**

N/A	Yes	No	
	☐	☐	**Chain of Custody Record(s)?**
	☐		If **No**, COC initiated by _____
	☐		Rec'd for Lab signed/date/time?
☐	☐		Shipping Document?
☐	☐		Other _____

COC ID Nos.

☐ TriMatrix

☐ Other (name or ID#)_____

Check COC for Accuracy

☐ **No analysis requested**

Yes	No	
☐	☐	Sample ID matches COC?
☐	☐	Sample date and time matches COC?
☐		Container type completed on COC?
☐	☐	All container types indicated are received?

Sample Condition Summary

☐ **Non-TriMatrix containers, see Notes**

N/A	Yes	No	
	☐	☐	Broken containers/lids?
	☐	☐	Missing or incomplete labels?
	☐	☐	Illegible information on labels?
	☐	☐	Low volume received?
	☐	☐	Inappropriate containers received?
☐	☐	☐	VOC vials / TOX containers have headspace?
☐	☐	☐	Extra sample locations / containers not listed on COC?

Check Sample Preservation

N/A	Yes	No	
	☐	☐	Average sample temperature ≤6° C?
☐	☐		Completed Sample Preservation Verification Form?
☐	☐	☐	Samples preserved correctly?
☐	☐		If "No", added orange tag?
☐	☐		Received pre-preserved VOC soils?

☐ MeOH ☐ Na_2SO_4

Check for Short Hold-Time Prep/Analyses

- ☐ Bacteriological
- ☐ Air Bags
- ☐ EnCores / Methanol Pre-Preserved
- ☐ Formaldehyde/Aldehyde
- ☐ Green-tagged Containers
- ☐ Yellow/White-tagged 1L Ambers (SV Prep-Lab)

AFTER HOURS ONLY:
COPIES OF COC TO LAB AREA(S)
- ☐ NONE RECEIVED
- ☐ RECEIVED, COCs TO LAB(S)

Notes

- ☐ Trip blank received ☐ Trip blank not listed on COC
- ☐ No COC received, Proj. Chemist reviewed (init./date) _____
- ☐ No analysis requested, Proj. Chemist completed (init./date) _____

Cooler Received (Date/Time)	Paperwork Delivered (Date/Time)	≤1 Hour Goal Met?
		Yes / No

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST - page 2

Project Chemist Use

Notify Laboratory Personnel of Short Hold-Times and/or Rush Work ☐ **NONE**

(Lab personnel notified/date)

- ☐ Inorganics _____
- ☐ Microbiology (bacteria) _____
- ☐ Metals Prep _____
- ☐ Metals _____
- ☐ GC-Volatiles _____
- ☐ MS-Volatiles _____
- ☐ Semi-Vol. Prep _____
- ☐ GC-Semi-Volatiles _____
- ☐ MS-Semi-Volatiles _____

Log-In Priority ☐ RUSH ☐ Standard

Project Chemist Notes to Log-In Personnel

Trip Blank: ☐ Log-in ☐ Do not log-in

☐ Prep Storage Blank for Client (VOCs)

☐ Sub-Contracting required ☐ Coolant required

☐ Non-TriMatrix or non-standard container type(s) received
Check pH of container type _____
Expected pH:_____ ☐ Adjust if needed

☐ Adjust pH of orange-tagged containers

☐ Lab-filter samples and document on Preservation Form

Sample Narratives to be added at Log-in

Log-In Use

Log Samples into LIMS Sample Nos.

N/A	Yes	
	☐	Receive samples in LIMS
	☐	Date/Time received entered in LIMS match COC
	☐	Read project and submittal narratives
☐	☐	Enter VOC rack/tray number into submittal narrative
	☐	Enter sample information into LIMS
☐	☐	Add any sample narratives
☐	☐	If non-conformance issues, add sample qualifiers
	☐	Print sample number labels

Log-in Analyst (initials/date/time)

Label Sample containers

N/A	Yes	No	
	☐	☐	LIMS label matches tag?
☐	☐		**DISCREPANCIES CORRECTED IN LIMS**
			Initials/Date: _____
☐	☐		Applicable stickers applied to labels?
			☐ MS/MSD sample
			☐ Composite before analysis
☐	☐		Applicable stickers applied to containers?
			☐ Waste sample
			☐ PT sample
			☐ USDA regulated
	☐	☐	Orange-tagged containers present?
☐	☐	☐	Adjust pH per Project Chemist
☐	☐		Initials and Date/Time Adjusted on orange tag?
☐	☐		Initials and Date/Time Adjusted on Preservation Form?

Verify Label Accuracy

	☐	Second analyst checked labels for accuracy?
☐	☐	Verify that Orange-tagged containers adjusted/initialed?

Labeled by (initials/date)	Verified by (initials/date)

Sample Storage Check all that apply

bacteria……	☐ bacteria refrigerator
non-volatiles……	☐ walk-in cooler
volatiles……..….	☐ volatile lab refrigerator
waste……...…	☐ waste cabinet
waste VOCs....…	☐ log-in hood refrigerator
low-level Hg ….	☐ metals lab - DO NOT STORE IN WALK-IN

Paperwork

N/A	Yes	
	☐	original COC (white)
	☐	copy of COC (yellow)
	☐	receiving/log-in checklist
☐	☐	additional cooler information form
☐	☐	sample preservation verification
☐	☐	sample receiving non-conformance form
☐	☐	shipping documents
☐	☐	custody seals
	☐	arrival log
☐	☐	other (note)

Appendix Q

TriMatrix
Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST
ADDITIONAL COOLER INFORMATION

Recorded by (initials/date)	Client		Project-Submittal No.
	Receipt Log No.	Sample Nos.	Project Chemist

Cooler No.	Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact
Coolant Location:
Dispersed / Top / Middle / Bottom
Coolant/Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers
Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact
Coolant Location:
Dispersed / Top / Middle / Bottom
Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers
Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact
Coolant Location:
Dispersed / Top / Middle / Bottom
Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers
Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact
Coolant Location:
Dispersed / Top / Middle / Bottom
Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers
Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact
Coolant Location:
Dispersed / Top / Middle / Bottom
Coolant/Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers
Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact
Coolant Location:
Dispersed / Top / Middle / Bottom
Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers
Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact
Coolant Location:
Dispersed / Top / Middle / Bottom
Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers
Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact
Coolant Location:
Dispersed / Top / Middle / Bottom
Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers
Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Comments

Appendix R

TriMatrix Laboratories, Inc.

SAMPLE PRESERVATION VERIFICATION FORM

page ___ of ____

Client		Project-Submittal No.
Receipt Log No.	Completed By (initials/date)	Project Chemist

COC ID No.			Adjusted by:_____ Date:_____		DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
Container Type	5	4	13		3	6	15	
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe	
Preservative	NaOH	H_2SO_4	H_2SO_4		None	HNO_3	HNO_3	
Expected pH	**>12**	**<2**	**<2**		**~7**	**<2**	**<2**	
COC Line No. 1								
COC Line No. 2								
COC Line No. 3								
COC Line No. 4								
COC Line No. 5								
COC Line No. 6								
COC Line No. 7								
COC Line No. 8								
COC Line No. 9								
COC Line No. 10								

Comments

COC ID No.			Adjusted by:_____ Date:_____		DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
Container Type	5	4	13		3	6	15	
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe	
Preservative	NaOH	H_2SO_4	H_2SO_4		None	HNO_3	HNO_3	
Expected pH	**>12**	**<2**	**<2**		**~7**	**<2**	**<2**	
COC Line No. 1								
COC Line No. 2								
COC Line No. 3								
COC Line No. 4								
COC Line No. 5								
COC Line No. 6								
COC Line No. 7								
COC Line No. 8								
COC Line No. 9								
COC Line No. 10								

Comments

pH strip lot No.

☐ **HC896537**

☐ _____

Aqueous Samples: For each sample and container type, check the box if pH is acceptable. **If pH is not acceptable for any sample container, record pH in box, and note on Sample Receiving Checklist and on Sample Receiving Non-Conformance Form.** If approved by Project Chemist, add acid or base to the sample to achieve the correct pH. Add up to, but do not exceed 2x the volume initially added at container prep (see table below for initial volumes used). **Add orange pH tag to sample container and record information requested. Record adjusted pH on this form. Do not adjust pH for container types 3, 6, and 15.**

Container Size (mL)	Original Vol. of Preservative (mL)
Container Type 5:	NaOH
500	2.5
1000	5.0
Container Type 4:	H_2SO_4
125	0.5
250	1.0
500	2.0
1000	4.0
Container Type 13	H_2SO_4
500	2.5

Appendix S

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING NON-CONFORMANCE REPORT

Client		Project-Submittal No.
Receipt Log No.	Completed By (initials/date)	Project Chemist

List non-conformance issues associated with this submittal in the chart below/left. Identify discrepancies between the COC and sample tags in the chart below/right. Add comments as needed. Give to Project Chemist for immediate action.

		Type of Problem									
COC ID No.	Line No.	Discrepancy	Missing Container	Broken Container	Label Missing / Incomplete	Label Illegible	Low Volume	Inappropriate Container	Headspace	Not Listed on COC	Preservation

COC					Sample Tag					Line Item Comments
Sample Field ID	Date Sampled	Time Sampled	Container Type	Qty	Sample Field ID	Date Sampled	Time Sampled	Container Type	Qty	

General Comments:

Project Chemist (initials/date)

Appendix T

Client:
Project: **TCLP Semi-Volatiles**
Work Order: **TCLP October 2008**

Project Manager: **Rick D. Wilburn**
Project Number: **[none]**
SDG:

Report To:
R
T
2
L
Phone:
Fax:

Invoice To:
TriMatrix Laboratories
Mr. Rick D. Wilburn
5560 Corporate Exchange Court SE
Grand Rapids, MI 49512-5503
Phone: 616-975-4500 x4
Fax: 616-942-7463

Client Due Date: **Nov-24-08 23:00 (21 day TAT)**
Date Received: Oct-24-08 12:00
Date Logged In: Oct-27-08 08:15
W.O. Comments: *QC is 3MD.*

Report Level: **3MD**
Received By: Rick D. Wilburn
Logged In By: William D. Cole

Analysis	Lab Due Date	TAT	Expires	Analysis Comments
0810557-01 TCLP Semi-Volatiles [Soil] ***Sampled Oct-23-08 08:00 Eastern by***				
8270C TCLP Herbs	Nov-24-08 17:00	10	Oct-30-08 08:00	
8270C TCLP SVOC/Pest	Nov-24-08 17:00	10	Oct-30-08 08:00	
TCLP Organics Extraction	Nov-24-08 17:00	10	Nov-06-08 08:00	
0810557-02 TCLP Analytes in Soil [Soil] ***Sampled Oct-23-08 08:00 Eastern by***				
8151A Herbicides CLP [dual-col]	Nov-24-08 17:00	10	Nov-06-08 08:00	
8270C Standard SVOCs	Nov-24-08 17:00	10	Nov-06-08 08:00	
Solids, Total 3550B (%)	Nov-24-08 17:00	10	Nov-06-08 08:00	

Client:	Project Manager: **Rick D. Wilburn**
Project: **TCLP Semi-Volatiles**	Project Number: **[none]**
Work Order: **TCLP October 2008**	SDG:

Inorganic - Wet Chemistry Analysis Detail

* indicates custom

Matrix	Analysis	Unit	MDL	RL
Soil	Solids, Total 3550B (%)	%	0.1	0.1

Semivolatiles GC Analysis Detail

* indicates custom

	Analyte	CLrept?	QCrept?	MDL	RL
Soil	**8151A Herbicides CLP [dual-col]**	**mg/kg**			
	2,4-D	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex)	Y	Y	0.0051	0.05
	2,4-D [2C]	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex) [2C]	Y	Y	0.0051	0.05

Semivolatiles MS Analysis Detail

* indicates custom

	Analyte	CLrept?	QCrept?	MDL	RL
Soil	**TCLP Organics Extraction**				
Soil	**8270C Standard SVOCs**	**mg/kg**			
	1,4-Dichlorobenzene	Y	Y	0.000906	0.0167
	2,4-Dinitrotoluene	Y	Y	0.00406	0.0167
	Hexachlorobenzene	Y	Y	0.00337	0.0167
	Hexachlorobutadiene	Y	Y	0.00107	0.0167
	Hexachloroethane	Y	Y	0.000778	0.0167
	3+4-Methylphenol	Y	Y	0.00129	0.0167
	2-Methylphenol	Y	Y	0.00223	0.0167
	Nitrobenzene	Y	Y	0.00199	0.0167
	Pentachlorophenol	Y	Y	0.00368	0.0167
	Pyridine	Y	Y	0.00566	0.0167
	2,4,5-Trichlorophenol	Y	Y	0.00329	0.0167
	2,4,6-Trichlorophenol	Y	Y	0.00132	0.0167
Soil	**8270C TCLP Herbs**	**mg/L**			
	2,4-D	Y	Y	0.00409	0.1
	2,4,5-TP (Silvex)	Y	Y	0.00381	0.1
Soil	**8270C TCLP SVOC/Pest**	**mg/L**			
	1,4-Dichlorobenzene	Y	Y	0.0000148	0.005
	2,4-Dinitrotoluene	Y	Y	0.000214	0.005
	Hexachlorobenzene	Y	Y	0.0000117	0.005
	Hexachlorobutadiene	Y	Y	0.000125	0.005
	Hexachloroethane	Y	Y	0.0000378	0.005
	Nitrobenzene	Y	Y	0.0000257	0.005
	Pyridine	Y	Y	0.000385	0.05
	Pentachlorophenol	Y	Y	0.000187	0.005
	2,4,6-Trichlorophenol	Y	Y	0.0000267	0.005
	2,4,5-Trichlorophenol	Y	Y	0.000109	0.005

Client:	Project Manager: **Rick D. Wilburn**
Project: **TCLP Semi-Volatiles**	Project Number: **[none]**
Work Order: **TCLP October 2008**	SDG:

Semivolatiles MS Analysis Detail

Analyte	CLrept?	QCrept?	* indicates custom MDL	RL
2-Methylphenol	Y	Y	0.000144	0.005
3-Methylphenol	Y	Y	0.0000157	0.005
4-Methylphenol	Y	Y	0.0000157	0.005
gamma-BHC (Lindane)	Y	Y	0.0000566	0.005
Endrin	Y	Y	0.000284	0.005
Methoxychlor	Y	Y	0.0000723	0.005
Technical Chlordane	Y	Y	0.000124	0.005
Heptachlor	Y	Y	0.0000907	0.005
Heptachlor Epoxide	Y	Y	0.0000758	0.005
Toxaphene	Y	Y	0.000293	0.5

Appendix U

Semivolatiles GC Sample Receipt Notice

Client: **C**
Project: **TCLP Semi-Volatiles**
Client Due Date: **Nov-24-08 23:00 (21 day TAT)**
W.O. Comments: *QC is 3MD.*

Project Manager: **Rick D. Wilburn**
Project Number: **[none]**
Report Level: **3MD**

Lab Number	Sample Name / Analysis	Matrix	Sampled Date / TAT	Expire Date	Sample Comments / Lab Due Date	Comments
0810557-02	**TCLP Analytes in Soil**	**Soil**	**Oct-23-08 08:00 Eastern**			
	8151A Herbicides CLP [dual-col]		10	Nov-06-08 08:00	Nov-24-08 17:00	

Semivolatiles GC Analysis Detail

* indicates custom

	Analyte	CLrept?	QCrept?	MDL	RL
Soil	**8151A Herbicides CLP [dual-col]**	**mg/kg**			
	2,4-D	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex)	Y	Y	0.0051	0.05
	2,4-D [2C]	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex) [2C]	Y	Y	0.0051	0.05

Semivolatiles MS Sample Receipt Notice

Client: **T**
Project: **TCLP Semi-Volatiles**
Client Due Date: **Nov-24-08 23:00 (21 day TAT)**
W.O. Comments: *QC is 3MD.*

Project Manager: **Rick D. Wilburn**
Project Number: **[none]**
Report Level: **3MD**

Lab Number	Sample Name / Analysis	Matrix	Sampled Date / TAT	Expire Date	Sample Comments / Lab Due Date	Comments
0810557-01	**TCLP Semi-Volatiles**	**Soil**	**Oct-23-08 08:00 Eastern**			
	8270C TCLP Herbs		10	Oct-30-08 08:00	Nov-24-08 17:00	
	8270C TCLP SVOC/Pest		10	Oct-30-08 08:00	Nov-24-08 17:00	
	TCLP Organics Extraction		10	Nov-06-08 08:00	Nov-24-08 17:00	
0810557-02	**TCLP Analytes in Soil**	**Soil**	**Oct-23-08 08:00 Eastern**			
	8270C Standard SVOCs		10	Nov-06-08 08:00	Nov-24-08 17:00	

Semivolatiles MS Analysis Detail

	Analyte	CLrept?	QCrept?	* indicates custom MDL	RL
Soil	**TCLP Organics Extraction**				
Soil	**8270C Standard SVOCs**	**mg/kg**			
	1,4-Dichlorobenzene	Y	Y	0.000906	0.0167
	2,4-Dinitrotoluene	Y	Y	0.00406	0.0167
	Hexachlorobenzene	Y	Y	0.00337	0.0167
	Hexachlorobutadiene	Y	Y	0.00107	0.0167
	Hexachloroethane	Y	Y	0.000778	0.0167
	3+4-Methylphenol	Y	Y	0.00129	0.0167
	2-Methylphenol	Y	Y	0.00223	0.0167
	Nitrobenzene	Y	Y	0.00199	0.0167
	Pentachlorophenol	Y	Y	0.00368	0.0167
	Pyridine	Y	Y	0.00566	0.0167
	2,4,5-Trichlorophenol	Y	Y	0.00329	0.0167
	2,4,6-Trichlorophenol	Y	Y	0.00132	0.0167
Soil	**8270C TCLP Herbs**	**mg/L**			
	2,4-D	Y	Y	0.00409	0.1
	2,4,5-TP (Silvex)	Y	Y	0.00381	0.1
Soil	**8270C TCLP SVOC/Pest**	**mg/L**			
	1,4-Dichlorobenzene	Y	Y	0.0000148	0.005
	2,4-Dinitrotoluene	Y	Y	0.000214	0.005
	Hexachlorobenzene	Y	Y	0.0000117	0.005
	Hexachlorobutadiene	Y	Y	0.000125	0.005
	Hexachloroethane	Y	Y	0.0000378	0.005
	Nitrobenzene	Y	Y	0.0000257	0.005
	Pyridine	Y	Y	0.000385	0.05
	Pentachlorophenol	Y	Y	0.000187	0.005
	2,4,6-Trichlorophenol	Y	Y	0.0000267	0.005
	2,4,5-Trichlorophenol	Y	Y	0.000109	0.005
	2-Methylphenol	Y	Y	0.000144	0.005
	3-Methylphenol	Y	Y	0.0000157	0.005
	4-Methylphenol	Y	Y	0.0000157	0.005
	gamma-BHC (Lindane)	Y	Y	0.0000566	0.005
	Endrin	Y	Y	0.000284	0.005
	Methoxychlor	Y	Y	0.0000723	0.005
	Technical Chlordane	Y	Y	0.000124	0.005
	Heptachlor	Y	Y	0.0000907	0.005
	Heptachlor Epoxide	Y	Y	0.0000758	0.005
	Toxaphene	Y	Y	0.000293	0.5

Semivolatiles MS, Soil, 3550B Sonication Extraction

Surrogate #1 = 8110251 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0810557	8270C Standard SVOCs	0810557	8270C MDEQ BNA	0810557	8270C MDEQ Base/Neutrals
0810648	8270C MDEQ BNA	0810665	8270C MDEQ Base/Neutrals	0811070	8270C Standard SVOCs
0811070	8270C MDEQ BNA	0811070	8270C MDEQ Base/Neutrals	0811154	8270C MDEQ BNA

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0813151-BLK1		Nov-10-08 07:09	BJH	30	1	100				BLANK	
0813151-BLK2		Nov-10-08 07:09	JLB	30	1	100				BLANK	mdeq base/neutrals
0813151-BLK3		Nov-10-08 07:09		30	1	100				BLANK	
0813151-BLK4		Nov-10-08 07:09		30	1	100				BLANK	
0813151-DUP1		Nov-10-08 07:09	BJH	30	1	100	0810557-02			DUPLICATE	
0813151-BS1		Nov-10-08 07:09	BJH	30	1	100		8110206	100	LCS	
0813151-BS2		Nov-10-08 07:09	BJH	30	1	100		8100124	100	LCS	
0813151-BS3		Nov-10-08 07:09	JLB	30	1	100		8110206	100	LCS	mdeq base/neutrals
0813151-BS4		Nov-10-08 07:09		30	1	100		8110206	100	LCS	
0813151-BS5		Nov-10-08 07:09		30	1	100		8110206	100	LCS	
0813151-MS1		Nov-10-08 07:09	BJH	30	1	100	0811070-06	8110206	100	MATRIX SPIKE	
0813151-MSD1		Nov-10-08 07:09	BJH	30	1	100	0811070-06	8110206	100	MATRIX SPIKE DUP	
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					Added for BatchQC in: 0813151
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					Added for BatchQC in: 0813151
0810648-19	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-20	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-21	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-24	A	Nov-10-08 07:09	BJH	30	5	100					stopped at 5 mL
0810648-25	A	Nov-10-08 07:09	BJH	30	5	100					stopped at 5 mL
0810665-01	A	Nov-10-08 07:09	BJH	30	1	100					2,4-Dinitrotoluene only
0810665-02	A	Nov-10-08 07:09	BJH	30	1	100					2,4-Dinitrotoluene only

Comments:

Analyst Initials:

Semivolatiles MS, Soil, Nov-12-08

Instrument = 308, Calibration = 8K05005

Sequence Analyses:
8270C MDEQ BNA

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client / QC Type*	*Extraction Comments*
8111315-TUN1	QC		8100195	8060452	MS TUNE	
8111315-CCV1	QC		8110293	8060452	CALIBRATION CHECK	
8111315-CCV2	QC		8110085	8060452	CALIBRATION CHECK	
0813151-BLK4	QC			8060452	BLANK	
0813151-BS5	QC			8060452	LCS	
0811154-04	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-05	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-06	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-07	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-09	8270C MDEQ BNA	A 02		8060452	Engineering	

Comments:

Analyst Initials:

Lab PM (Rick D. Wilburn) Nov-11-08 - Dec-09-08

yzed,Available,Batched,Cancelled,Entered,Hold,Invoiced,Leached,Prepared,Received,Reported,Reviewed,Subcontr

Lab Number	Analysis	Matrix	RptLev	RTAT	Due	Expires	Status	Client	Project	Sample [Analysis] Comments
0810557-01	8270C TCLP Herbs	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	8270C TCLP SVOC/P	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	TCLP Organics Extrac	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8151A Herbicides CLI	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8270C Standard SVOC	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810558-01	DRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-02-08	Reported	RTC	Minnesota DRO/GRO	
0810558-01	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	GRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	

Appendix V

TriMatrix Laboratories, Inc. - Department

Work Orders Received Sep-01-08 to Sep-30-08 - Printed Dec-08-08 10:26 by TCB

Department	Samples	Analyses	Price	Surcharge	Total
Inorganic - Wet Chemistry	1622	5878	$120,471.90	$333.75	$120,805.60
Metals	997	10085	$83,255.61	$829.00	$84,084.61
Semivolatiles GC	495	638	$60,893.00	$110.00	$61,003.00
Semivolatiles MS	468	548	$78,247.00	$58.15	$78,305.15
Volatiles GC	116	117	$5,438.50	$14.40	$5,452.90
Volatiles MS	1382	1400	$135,669.00	$297.75	$135,966.80
****TOTALS****	**5080**	**18666**	**$483,975.01**	**$1,643.05**	**$485,618.06**

TriMatrix Laboratories, Inc. - % On-Time by Department [Sep-01-08 to Sep-30-08]

Printed Dec-08-08 10:36 by TCB

Department: [All]

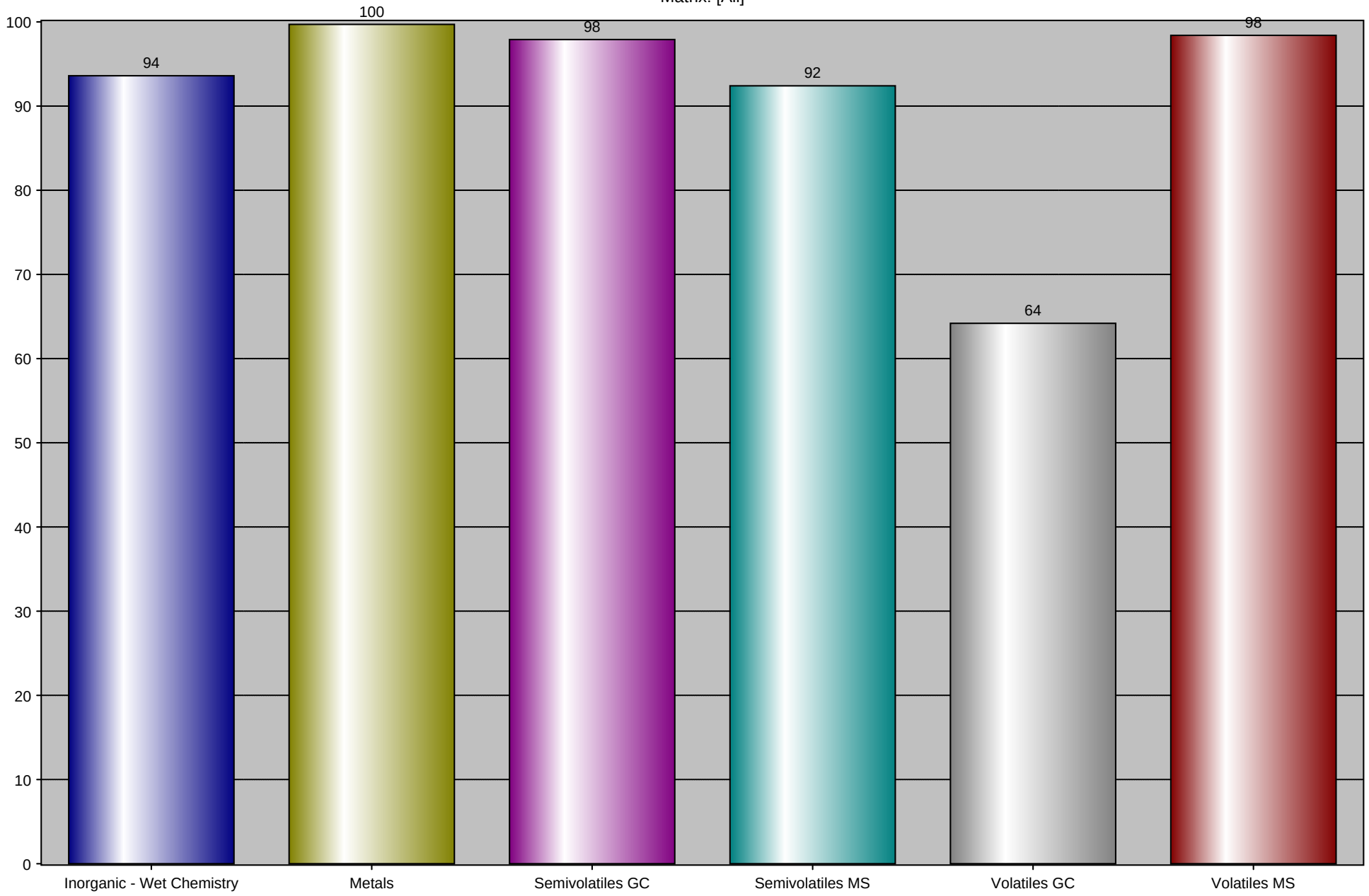
Analysis: [All]

Matrix: [All]

Department	On-Time	Total	%
Inorganic - Wet Chemistry	4117	4399	93.6
Metals	6889	6907	99.7
Semivolatiles GC	644	658	97.9
Semivolatiles MS	390	422	92.4
Volatiles GC	147	229	64.2
Volatiles MS	1415	1438	98.4

TriMatrix Laboratories, Inc. - % On-Time by Department [Sep-01-08 to Sep-30-08]

Printed Dec-08-08 10:34 by TCB
Department: [All]
Analysis: [All]
Matrix: [All]



Lab PM (Rick D. Wilburn) Jan-01-08 - Dec-09-08

Available,Cancelled,Completed,Invoiced,Preliminary,Received,Reported

Work Order	Done	RptLvl	Pending	Status	Client	Project Name (Number)	PMgr	TAT	Received	Due
0707274	64/64	3MD		Completed	Environmental Resource Associates	ERA WS PT Samples Summer (35005)	RDW	22	Jul-17-07	Aug-16-07
0801455	2/2	3MD		Completed	Environmental Resource Associates	ERA WP PT Samples (35005)	RDW	19	Jan-25-08	Feb-21-08
0801456	120/120	3FL		Completed	Environmental Resource Associates	Semi-Annual Solid PE Study (35338)	RDW	21	Jan-25-08	Feb-25-08
0801501	136/136	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	18	Jan-30-08	Feb-25-08
0802130	60/60	3MD		Completed	Environmental Resource Associates	ERA WS PT Samples Winter (35005)	RDW	16	Feb-08-08	Mar-03-08
0802188	4/4	3MD		Completed	Environmental Resource Associates	Micro Analyst Cert (34110)	RDW	10	Feb-12-08	Feb-26-08
0803447	4/4	3MD		Completed	Environmental Resource Associates	Micro Analyst Cert (34110)	RDW	10	Mar-27-08	Apr-10-08
0804113	116/116	3MD		Completed	Analytical Products Group	WP Performance Testing Program Spring (35508)	RDW	10	Apr-07-08	Apr-21-08
0805464	1/1	3MD		Completed	Analytical Products Group	WP Performance Testing Program Spring Re-Do (35508)	RDW	5	May-21-08	May-29-08
0805489	70/70	3MD		Completed	Analytical Products Group	DMRQA Testing (36330)	RDW	26	May-21-08	Jun-27-08
0806220	4/4	3MD		Completed	Analytical Products Group	WP Performance Testing Program Quick Turn (35508)	RDW	10	Jun-11-08	Jun-25-08
0806250	75/75	3MD		Completed	TriMatrix Laboratories	pH Strip Testing (36236)	RDW	10	Jun-12-08	Jun-26-08
0807484	126/126	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	18	Jul-23-08	Aug-18-08
0807485	120/120	3MD		Completed	Environmental Resource Associates	Semi-Annual Solid PE Study (35338)	RDW	22	Jul-24-08	Aug-25-08
0807486	2/2	3MD		Completed	Environmental Resource Associates	ERA WP PT Samples (35005)	RDW	17	Jul-24-08	Aug-18-08
0808052	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-04-08	Aug-18-08
0808059	2/2	3MD		Completed	Analytical Products Group	DMRQA Testing Micro (36330)	RDW	10	Aug-05-08	Aug-19-08
0808244	48/48	2RLM		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-13-08	Aug-27-08
0808571	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-28-08	Sep-12-08
0809070	10/10	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	17	Sep-04-08	Sep-29-08
0809419	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Sep-22-08	Oct-06-08
0810124	110/110	3MD		Completed	Analytical Products Group	WP Performance Testing Program Fall (35508)	RDW	18	Oct-08-08	Nov-03-08
0810557	6/6	3MD		Completed	RTC	TCLP Semi-Volatiles ([none])	RDW	21	Oct-24-08	Nov-24-08

Lab PM (Rick D. Wilburn) Jan-01-08 - Dec-09-08

Available,Cancelled,Completed,Invoiced,Preliminary,Received,Reported

Work Order	Done	RptLvl	Pending	Status	Client	Project Name (Number)	PMgr	TAT	Received	Due
0810558	4/4	3MD		Completed	RTC	Minnesota DRO/GRO ([none])	RDW	21	Oct-24-08	Nov-24-08

Lab PM (Rick D. Wilburn) Nov-01-08 - Nov-30-08

yzed,Available,Batched,Cancelled,Entered,Hold,Invoiced,Leached,Prepared,Received,Reported,Reviewed,Subcontr

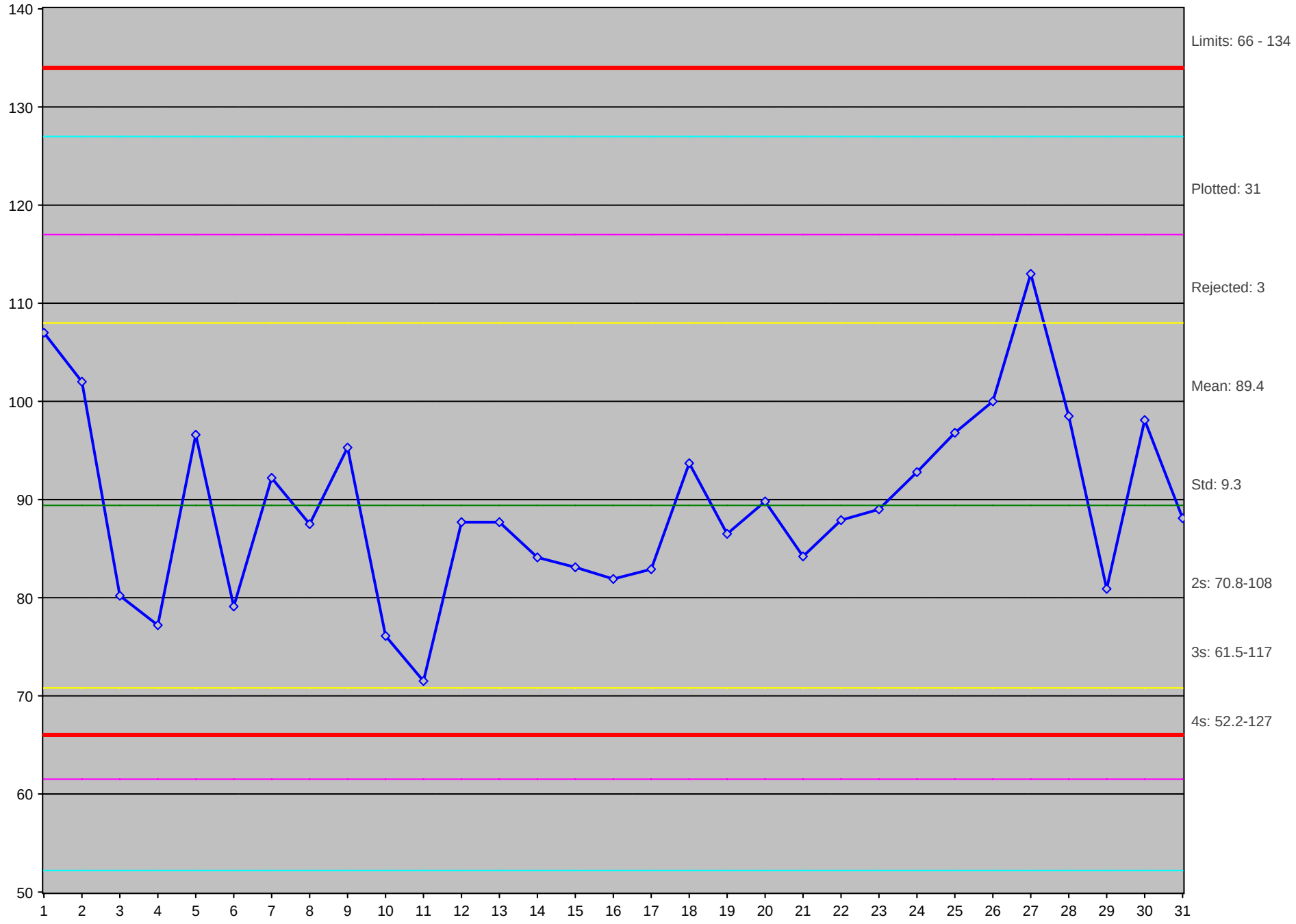
Lab Number	Analysis	Matrix	RptLev	RTAT	Due	Expires	Status	Client	Project	Sample [Analysis] Comments
0810557-01	8270C TCLP Herbs	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	8270C TCLP SVOC/P	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	TCLP Organics Extrac	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8151A Herbicides CLI	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8270C Standard SVOC	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810558-01	DRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-02-08	Reported	RTC	Minnesota DRO/GRO	
0810558-01	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	GRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	

Appendix W

TriMatrix Laboratories, Inc. - LCS %R for CHRYSENE

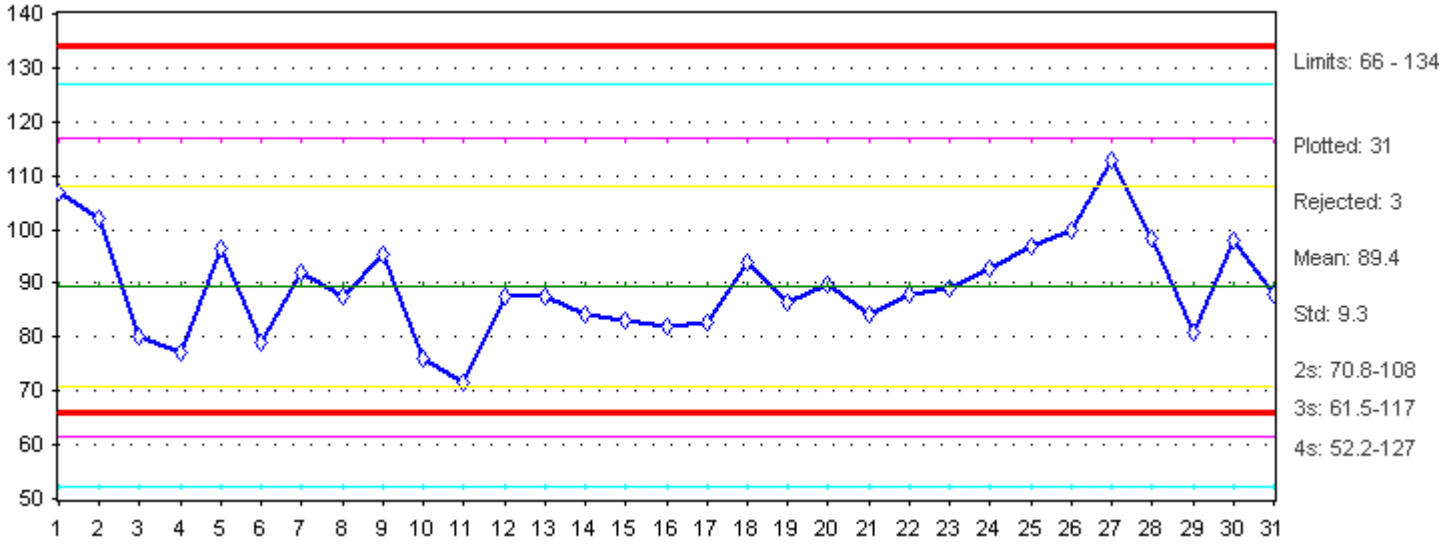
8270C Standard SVOCs IN Water Printed: Dec-08-08 11:04 by TCB

All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]



Printed:	Dec-08-08 11:09	Matrices:	Water
Client:	All Clients	Instruments:	All Instruments
Project:	All Projects	Prepared By:	All Extractionists
Analyses:	8270C Standard SVOCs	Analyzed By:	All Analysts
		Extractions:	All Extractions

Chrysene ***LCS %R***

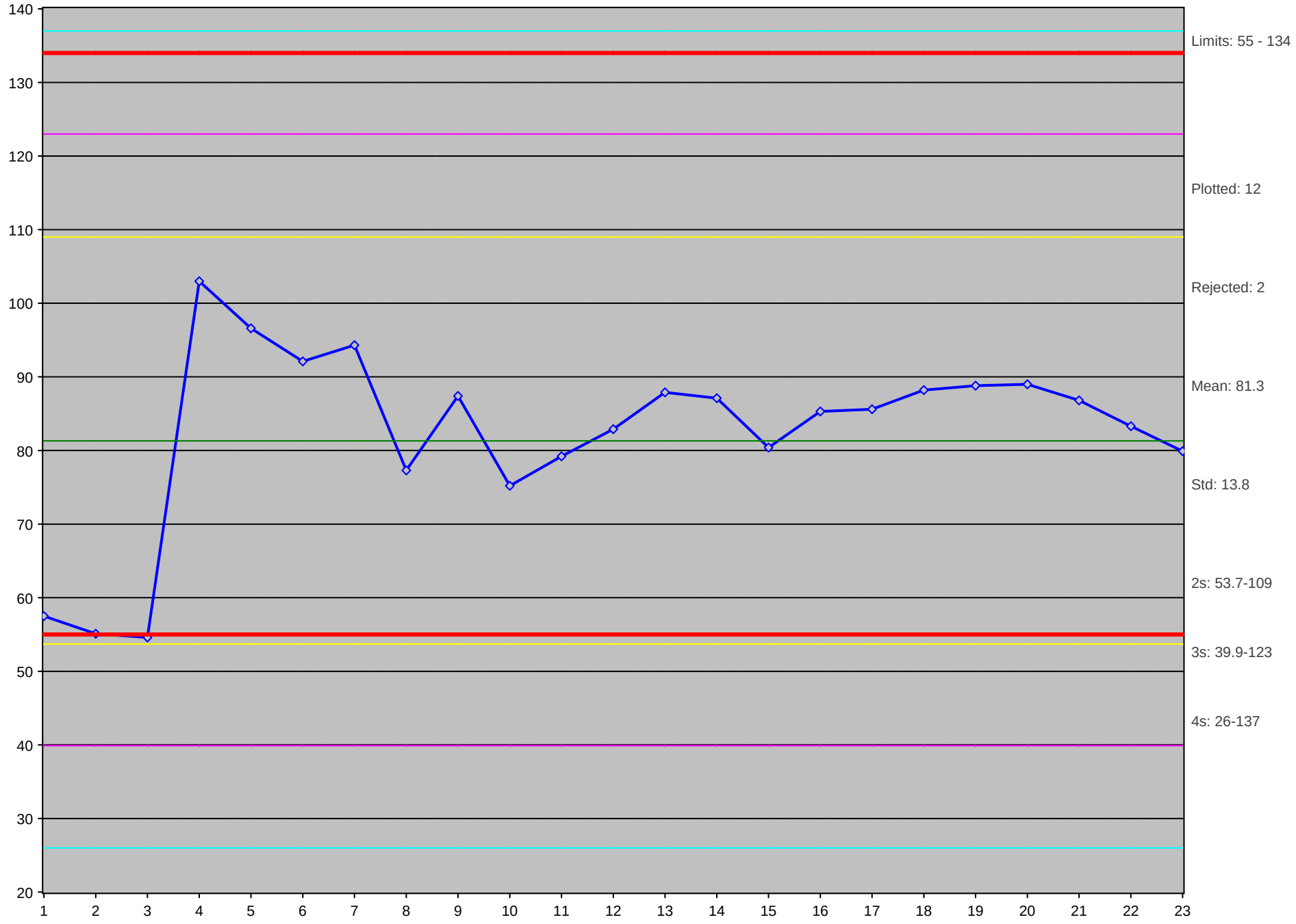


Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	0809978-BS1	8/29/08	9/2/08	9.6 ug/L	10.25	106.7708	66-134	
	0810083-BS2	9/2/08	9/3/08	9.6 ug/L	9.832	102.4167	66-134	
X	0810084-BS1	9/2/08	9/4/08	9.6 ug/L	0		66-134	
	0810561-BS1	9/12/08	9/17/08	96 ug/L	76.98	80.1875	66-134	
	0810624-BS1	9/15/08	9/16/08	9.6 ug/L	7.41	77.1875	66-134	
	0810739-BS1	9/17/08	9/18/08	96 ug/L	92.76	96.625	66-134	
X	0810740-BS1	9/17/08	9/22/08	9.6 ug/L	0		66-134	
	0810938-BS1	9/22/08	9/26/08	9.6 ug/L	7.59	79.0625	66-134	
	0810561-BS2	9/22/08	9/23/08	9.6 ug/L	8.852	92.20833	66-134	GN020
	0810967-BS1	9/23/08	9/26/08	96 ug/L	83.96	87.45833	66-134	
	0811030-BS1	9/24/08	9/27/08	96 ug/L	91.5	95.3125	66-134	
	0811107-BS1	9/25/08	9/27/08	9.6 ug/L	7.31	76.14583	66-134	
X	0811107-BS2	9/25/08	10/3/08	9.6 ug/L	0		66-134	
	0811170-BS1	9/26/08	9/27/08	9.6 ug/L	6.86	71.45833	66-134	
	0811170-BS2	9/29/08	10/2/08	9.6 ug/L	8.419	87.69791	66-134	
	0811170-BS3	9/29/08	10/2/08	9.6 ug/L	8.419	87.69791	66-134	
	0811030-BS2	10/1/08	10/1/08	9.6 ug/L	8.07	84.06249	66-134	
	0811661-BS1	10/9/08	10/10/08	9.6 ug/L	7.98	83.125	66-134	
	0811711-BS1	10/13/08	10/13/08	9.6 ug/L	7.866	81.9375	66-134	
	0811661-BS2	10/14/08	10/15/08	9.6 ug/L	7.96	82.91666	66-134	
	0811858-BS2	10/15/08	10/16/08	96 ug/L	89.94	93.6875	66-134	
	0811858-BS1	10/15/08	10/20/08	9.6 ug/L	8.3	86.45833	66-134	
	0812029-BS1	10/20/08	10/25/08	9.6 ug/L	8.62	89.79166	66-134	
	0812131-BS1	10/21/08	10/21/08	9.6 ug/L	8.08	84.16666	66-134	
	0812166-BS1	10/21/08	10/22/08	9.6 ug/L	8.44	87.91666	66-134	
	0812461-BS1	10/27/08	10/30/08	9.6 ug/L	8.54	88.95833	66-134	
	0812462-BS1	10/27/08	10/29/08	9.6 ug/L	8.91	92.81249	66-134	

TriMatrix Laboratories, Inc. - MS %R for CHRYSENE

8270C Standard SVOCs IN Water Printed: Dec-08-08 11:12 by TCB

All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]



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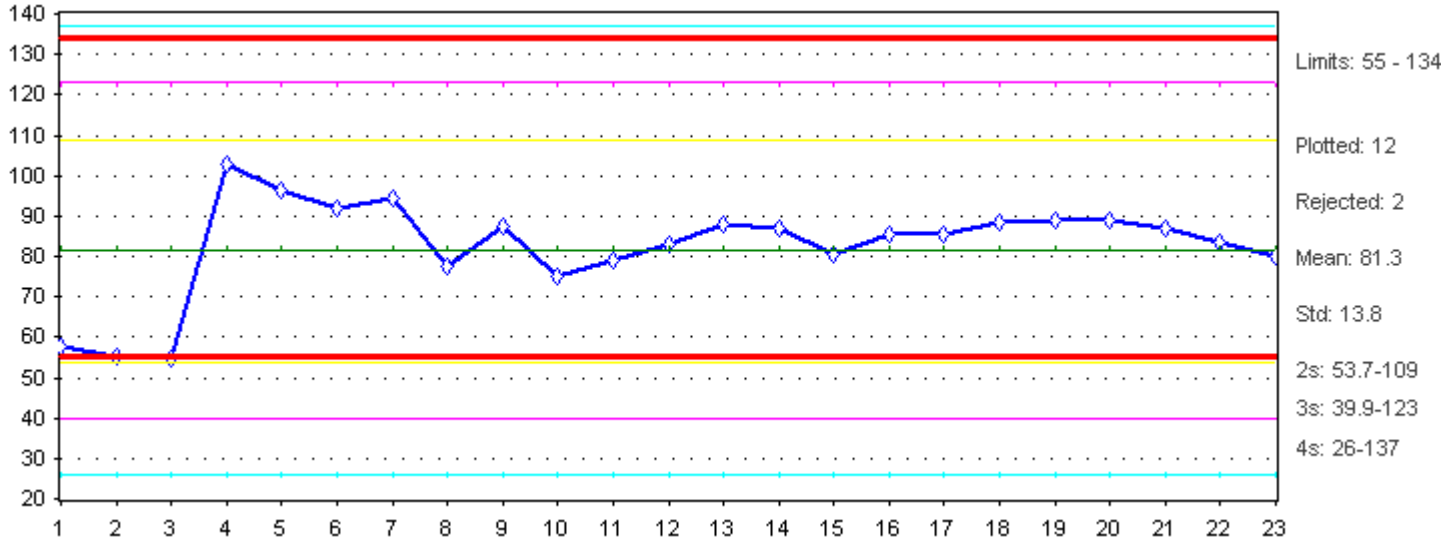
Project:	All Projects	Prepared By:	All Extractionists
Analyses:	8270C Standard SVOCs	Analyzed By:	All Analysts
		Extractions:	All Extractions

Chrysene ***MS %R***

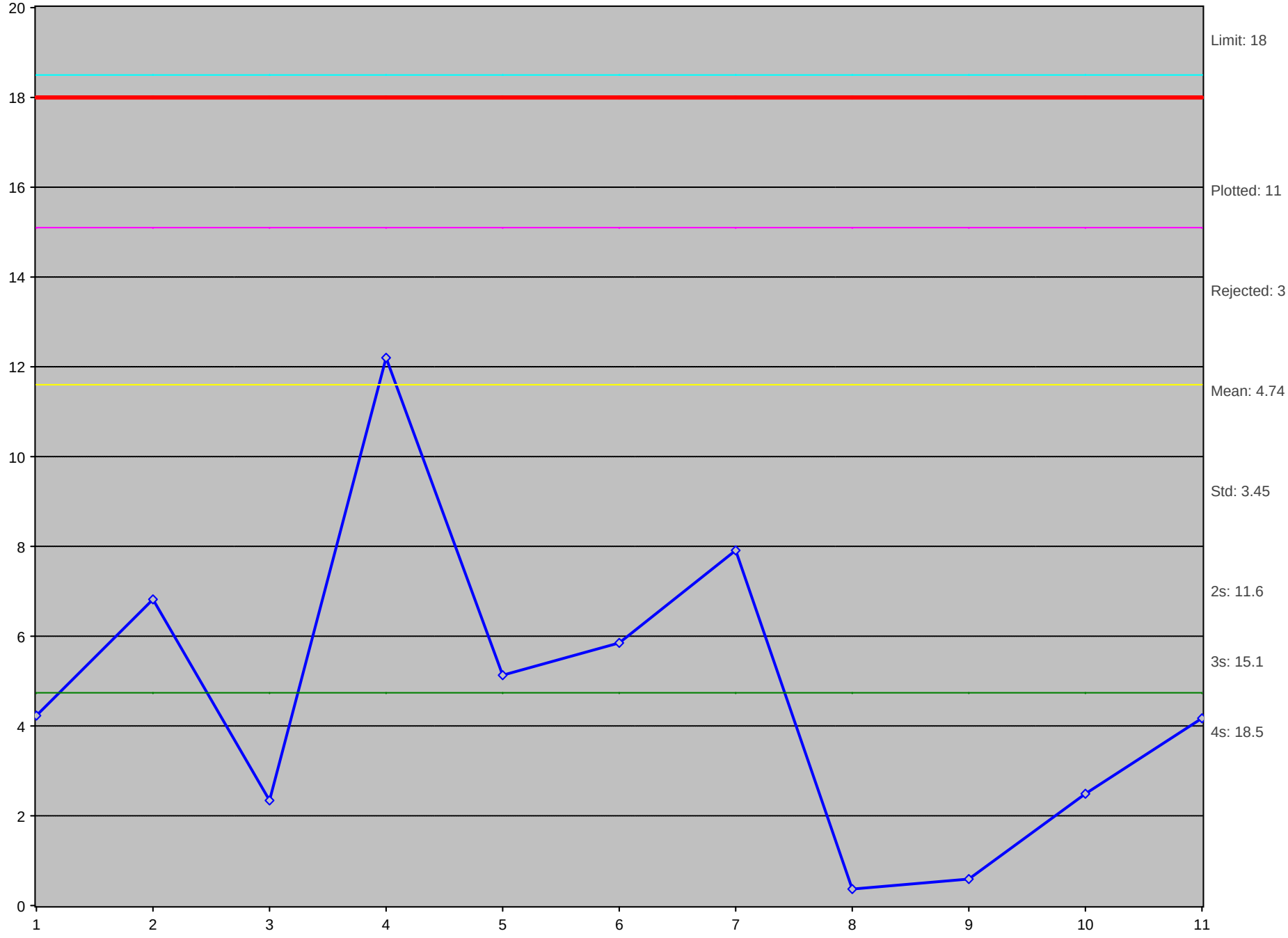


Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Source	Limits	Qualifiers
	0809978-MS1	8/29/08	9/2/08	9.69697 ug/L	5.606061	57.5	808570-08 (3.030303E-0	55-134	
	0809978-MSD1	8/29/08	9/2/08	9.69697 ug/L	5.373738	55.10417	808570-08 (3.030303E-0	55-134	
X	0810084-MSD1	9/2/08	9/4/08	9.6 ug/L	0.01	0.520833	0808570-29 (0.05)	55-134	
	0810084-MS1	9/2/08	9/5/08	9.6 ug/L	5.29	54.58333	0808570-29 (0.05)	55-134	
	0810561-MS1	9/12/08	9/18/08	96.9697 ug/L	100.2828	103.4166	0809136-03 (ND)	55-134	
	0810561-MSD1	9/12/08	9/18/08	96.9697 ug/L	93.66666	96.59373	0809136-03 (ND)	55-134	
	0810561-MS2	9/12/08	9/18/08	97.95918 ug/L	90.2347	92.11459	0809136-05 (ND)	55-134	
	0810561-MSD2	9/12/08	9/18/08	97.95918 ug/L	92.36735	94.29167	0809136-05 (ND)	55-134	
	0810967-MS1	9/23/08	9/27/08	97.95918 ug/L	75.7551	77.33333	0809311-06 (ND)	55-134	
	0810967-MSD1	9/23/08	9/27/08	97.95918 ug/L	85.59183	87.37499	0809311-06 (ND)	55-134	
	0811107-MS1	9/25/08	9/27/08	9.795918 ug/L	7.367347	75.20833	0809435-05 (ND)	55-134	
	0811107-MSD1	9/25/08	9/27/08	9.795918 ug/L	7.755102	79.16667	0809435-05 (ND)	55-134	
	0811661-MS1	10/9/08	10/10/08	10.90909 ug/L	9.045455	82.91668	0810133-04 (ND)	55-134	
	0811661-MSD1	10/9/08	10/10/08	10.90909 ug/L	9.590909	87.91667	0810133-04 (ND)	55-134	
	0811711-MS1	10/13/08	10/13/08	9.795918 ug/L	8.529592	87.07291	0810180-03	55-134	
	0811711-MSD1	10/13/08	10/14/08	9.795918 ug/L	7.880612	80.44791	0810180-03	55-134	
	0811858-MS1	10/15/08	10/16/08	9.6 ug/L	8.19	85.31249	0810220-33 (ND)	55-134	
	0811858-MSD1	10/15/08	10/16/08	9.6 ug/L	8.22	85.625	0810220-33 (ND)	55-134	
	0812029-MS1	10/20/08	10/25/08	9.69697 ug/L	8.555556	88.22917	0810321-06	55-134	
	0812029-MSD1	10/20/08	10/25/08	9.69697 ug/L	8.606061	88.75	0810321-06	55-134	
	0812461-MS1	10/27/08	10/29/08	9.795918 ug/L	8.714286	88.95833	0810435-08 (ND)	55-134	
	0812461-MSD1	10/27/08	10/29/08	9.795918 ug/L	8.5	86.77083	0810435-08 (ND)	55-134	
X	0812462-MS1	10/27/08	10/28/08	ug/L	0		0810452-01 (ND)	55-134	
X	0812462-MSD1	10/27/08	10/28/08	ug/L	0		0810452-01 (ND)	55-134	
X	0813364-MS1	11/13/08	11/17/08	ug/L	0		0811231-07	55-134	
X	0813364-MSD1	11/13/08	11/17/08	ug/L	0		0811231-07	55-134	
	0813840-MS1	11/24/08	12/2/08	9.494949 ug/L	7.909091	83.29787	0811481-04 (ND)	55-134	
	0813840-MSD1	11/24/08	12/2/08	9.494949 ug/L	7.585859	79.89362	0811481-04 (ND)	55-134	

TriMatrix Laboratories, Inc. - MS/MSD RPD for CHRYSENE

8270C Standard SVOCs IN Water Printed: Dec-08-08 11:15 by TCB

All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]



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Project:	All Projects	Prepared By:	All Extractionists
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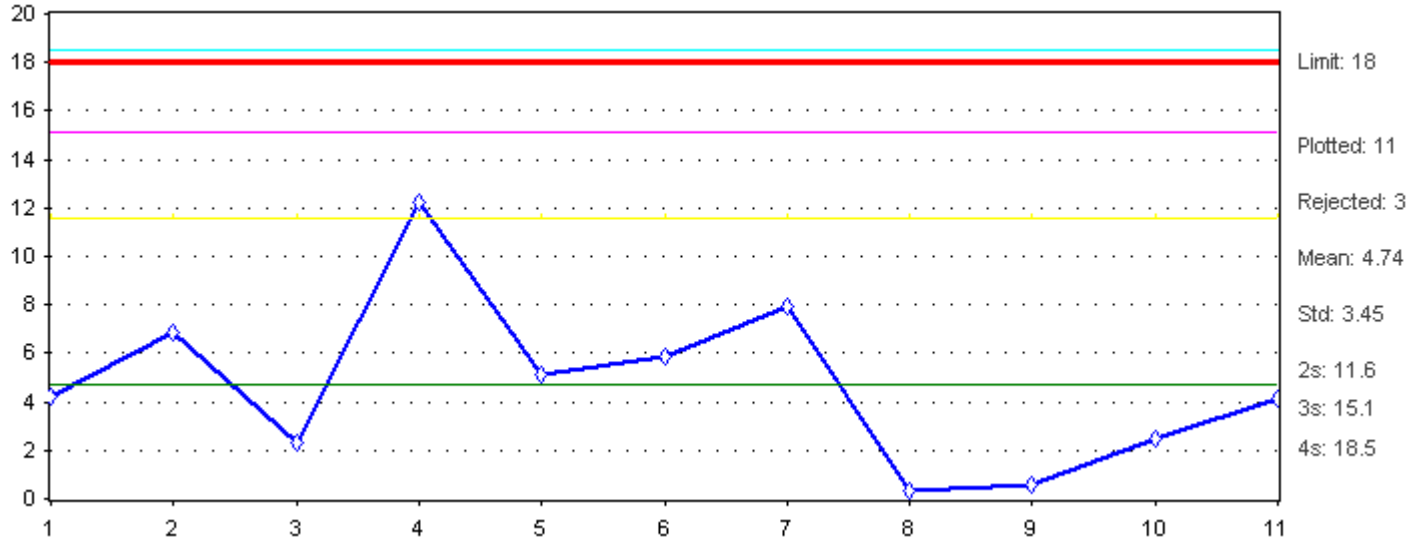
		Extractions:	All Extractions

Chrysene ***MS/MSD RPD***



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Dup	Dup %R	Source	RPD	Limit	Qualifier
	0809978-MSD1	8/29/08	9/2/08	9.69697 ug/L	5.373738	55.10417	5.606061	57.5	08570-08 (3.030303E-0	4.231829	18	
X	0810084-MSD1	9/2/08	9/4/08	9.6 ug/L	0.01	-0.5208333	5.29	54.58333	0808570-29 (0.05)		18	
	0810561-MSD1	9/12/08	9/18/08	96.9697 ug/L	93.66666	96.59373	100.2828	103.4166	0809136-03 (ND)	6.822543	18	
	0810561-MSD2	9/12/08	9/18/08	97.95918 ug/L	92.36735	94.29167	90.2347	92.11459	0809136-05 (ND)	2.335839	18	
	0810967-MSD1	9/23/08	9/27/08	97.95918 ug/L	85.59183	87.37499	75.7551	77.33333	0809311-06 (ND)	12.19327	18	
	0811107-MSD1	9/25/08	9/27/08	9.795918 ug/L	7.755102	79.16667	7.367347	75.20833	0809435-05 (ND)	5.128209	18	
	0811661-MSD1	10/9/08	10/10/08	10.90909 ug/L	9.590909	87.91667	9.045455	82.91668	0810133-04 (ND)	5.853653	18	
	0811711-MSD1	10/13/08	10/14/08	9.795918 ug/L	7.880612	80.44791	8.529592	87.07291	0810180-03	7.909465	18	
	0811858-MSD1	10/15/08	10/16/08	9.6 ug/L	8.22	85.625	8.19	85.31249	0810220-33 (ND)	.365639	18	
	0812029-MSD1	10/20/08	10/25/08	9.69697 ug/L	8.606061	88.75	8.555556	88.22917	0810321-06	.588577	18	
	0812461-MSD1	10/27/08	10/29/08	9.795918 ug/L	8.5	86.77083	8.714286	88.95833	0810435-08 (ND)	2.489628	18	
X	0812462-MSD1	10/27/08	10/28/08	ug/L	0		0		0810452-01 (ND)		18	
X	0813364-MSD1	11/13/08	11/17/08	ug/L	0		0		0811231-07		18	
	0813840-MSD1	11/24/08	12/2/08	9.494949 ug/L	7.585859	79.89362	7.909091	83.29787	0811481-04 (ND)	4.172097	18	

Appendix X

TriMatrix Laboratories, Inc.

Controlled Temperature Unit #55
Daily Log Sheet

Description: Fisher Isotemp Freezer
Model Number: 13-986-148
Serial #: 2017080504449
Location: Volatile Organic Laboratory

Purpose: Volatile Low-Level Soil Samples
Control Windows: Low: ≥ -20° C High: ≤ -7° C
Thermometer #: 184
Thermometer Serial #: 1353

Date	Initials	Time of First Reading	First Reading (°C)	Time of Second Reading	Second Reading (°C)	Weekend Minimum (°C)	Weekend Maximum (°C)	Adjustments/Observations/Comments

Appendix Y

TriMatrix Laboratories, Inc.

Balance Calibration Verification Acceptance Window Calculations

Balance ID: 204
Manufacturer: Mettler
Serial Number: J58563
Model Number: BB2440

Calibration Source: External
Calibration Weight (g): 1000
Calibration Weight Error (g): 0.00171811
Location: Volatiles Laboratory; South Bench Top

I. Calibration Weight Correction Calculations

Calibration Verification Weight Nominal Mass (g)	Calibration Verification Weight Correction Factor (g)	Calibration Verification Weight Actual Mass (g)	Linear Error of Balance (g)	Calibration Verification Weight Expected Mass (g)	Calibration Weight Combinations Used For Verification	
					Nominal	Actual
0.5000	0.00000190	0.5000	-0.00000086	0.5000	0.50	0.5000
5.0000	0.00000910	5.0000	-0.00000859	5.0000		
20.0000	0.00003540	20.0000	-0.00003436	20.0000	0.50 + 5 + 20	25.5000
100.0000	0.00006020	100.0001	-0.00017181	99.9999	0.50 + 100	100.4999

II. 20 Measurements Using Each Calibration Verification Mass

Date	Replicate Number	Mass 1 (g) 0.50	Mass 2 (g) 25.50	Mass 3 (g) 100.50			
1/29/2008	1	0.49	25.48	100.49			
1/29/2008	2	0.49	25.49	100.48			
1/29/2008	3	0.48	25.47	100.48			
1/29/2008	4	0.49	25.48	100.49			
1/29/2008	5	0.48	25.48	100.48			
1/29/2008	6	0.49	25.51	100.49			
1/29/2008	7	0.52	25.49	100.49			
1/30/2008	8	0.52	25.49	100.49			
1/30/2008	9	0.50	25.48	100.48			
1/30/2008	10	0.48	25.50	100.49			
1/30/2008	11	0.50	25.48	100.48			
1/30/2008	12	0.49	25.49	100.49			
1/30/2008	13	0.48	25.50	100.50			
1/30/2008	14	0.49	25.49	100.49			
1/31/2008	15	0.51	25.50	100.50			
1/31/2008	16	0.51	25.49	100.49			
1/31/2008	17	0.48	25.52	100.51			
1/31/2008	18	0.52	25.51	100.49			
1/31/2008	19	0.50	25.52	100.48			
1/31/2008	20	0.51	25.51	100.49			

III. Calibration Verification Acceptance Window Calculations

Standard Deviation:	0.01424411	0.01429022	0.00788069			
Random Error:	0.04273234	0.04287067	0.02364207			
Acceptance Window Low:	0.46	25.46	100.48			
Acceptance Window High:	0.54	25.54	100.52			

TriMatrix Laboratories, Inc.

Daily Balance Calibration Logbook

Balance ID: 204 **Serial #:** J58563 **Calibration Source:** External **Location:** Volatiles Laboratory;
Manufacturer: Mettler **Model #:** BB2440 **Calibration Weight (g):** 1000 South Bench Top

Date	Analyst	Calibration Verification Expected Mass 0.50	Window Pass / Fail 0.46 -0.54	Calibration Verification Expected Mass 25.50	Window Pass / Fail 25.46 -25.54	Calibration Verification Expected Mass 100.50	Window Pass / Fail 100.48 -100.52	Calibration Verification Expected Mass	Window Pass / Fail	Calibration Verification Expected Mass	Window Pass / Fail

Appendix Z

Analytical Standard Record

TriMatrix Laboratories, Inc.

7120763

Description:	8260 1 UG/L 12-28-07	Expires:	Feb-15-08
Standard Type:	Calibration Standard	Prepared:	Dec-28-07
Solvent:	WTR	Prepared By:	Diane L. VanMale
Final Volume (mls):	100	Department:	Volatiles MS
Vials:	1	Last Edit:	Dec-28-07 15:42 by DLV

Analyte	CAS Number	Concentration	Units
4-Methyl-2-pentanone (MIBK)	108-10-1	0.001	ppm
1-Chlorohexane	544-10-5	0.001	ppm
2,2-Dichloropropane	594-20-7	0.001	ppm
2-Butanone (MEK)	78-93-3	0.001	ppm
2-Chloroethyl Vinyl Ether	110-75-8	0.001	ppm
2-Chlorotoluene	95-49-8	0.001	ppm
2-Hexanone	591-78-6	0.001	ppm
2-Methylnaphthalene	91-57-6	0.001	ppm
4-Bromofluorobenzene	460-00-4	0.04	ppm
Carbon Disulfide	75-15-0	0.001	ppm
4-Isopropyltoluene	99-87-6	0.001	ppm
1,3-Dichloropropane	142-28-9	0.001	ppm
Acetone	67-64-1	0.001	ppm
Acrolein	107-02-8	0.001	ppm
Acrylonitrile	107-13-1	0.001	ppm
Benzene	71-43-2	0.001	ppm
Bromobenzene	108-86-1	0.001	ppm
Bromochloromethane	74-97-5	0.001	ppm
Bromodichloromethane	75-27-4	0.001	ppm
Bromoform	75-25-2	0.001	ppm
1,1,1,2-Tetrachloroethane	630-20-6	0.001	ppm
4-Chlorotoluene	106-43-4	0.001	ppm
1,2-Dibromo-3-chloropropane	96-12-8	0.001	ppm
1,1,1-Trichloroethane	71-55-6	0.001	ppm
1,1,2,2-Tetrachloroethane	79-34-5	0.001	ppm
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.001	ppm
1,1,2-Trichloroethane	79-00-5	0.001	ppm
1,1-Dichloroethane	75-34-3	0.001	ppm
1,1-Dichloroethene	75-35-4	0.001	ppm
1,1-Dichloropropene	563-58-6	0.001	ppm

Reviewed By Date

1,2,3-Trichlorobenzene	87-61-6	0.001	ppm
1,2,3-Trichloropropane	96-18-4	0.001	ppm
1,4-Dichlorobenzene	106-46-7	0.001	ppm
1,2,4-Trimethylbenzene	95-63-6	0.001	ppm
1,3-Dichloropropene (Total)	542-75-6	0.002	ppm
1,2-Dibromoethane	106-93-4	0.001	ppm
1,2-Dichlorobenzene	95-50-1	0.001	ppm
1,2-Dichloroethane	107-06-2	0.001	ppm
1,2-Dichloroethane-d4	17060-07-0	0.04	ppm
1,2-Dichloroethene (Total)	540-59-0	0.002	ppm
1,2-Dichloropropane	78-87-5	0.001	ppm
1,3,5-Trimethylbenzene	108-67-8	0.001	ppm
1,3-Dichlorobenzene	541-73-1	0.001	ppm
Carbon Tetrachloride	56-23-5	0.001	ppm
1,2,4-Trichlorobenzene	120-82-1	0.001	ppm
Total Trihalomethanes		0.004	ppm
n-Butylbenzene	104-51-8	0.001	ppm
n-Propylbenzene	103-65-1	0.001	ppm
Naphthalene	91-20-3	0.001	ppm
sec-Butylbenzene	135-98-8	0.001	ppm
Styrene	100-42-5	0.001	ppm
tert-Butylbenzene	98-06-6	0.001	ppm
Tetrachloroethene	127-18-4	0.001	ppm
Tetrahydrofuran	109-99-9	0.001	ppm
Bromomethane	74-83-9	0.001	ppm
Toluene-d8	2037-26-5	0.04	ppm
Methyl tert-Butyl Ether	1634-04-4	0.001	ppm
trans-1,2-Dichloroethene	156-60-5	0.001	ppm
trans-1,3-Dichloropropene	10061-02-6	0.001	ppm
trans-1,4-Dichloro-2-butene	110-57-6	0.001	ppm
Trichloroethene	79-01-6	0.001	ppm
Trichlorofluoromethane	75-69-4	0.001	ppm
Vinyl Acetate	108-05-4	0.001	ppm
Vinyl Chloride	75-01-4	0.001	ppm
Xylene (Total)	1330-20-7	0.003	ppm
Xylene, Meta + Para	136777-61-2	0.002	ppm
Toluene	108-88-3	0.001	ppm
Dichlorofluoromethane	75-43-4	0.001	ppm
Chlorobenzene	108-90-7	0.001	ppm

Reviewed By Date

Analytical Standard Record

TriMatrix Laboratories, Inc.

7120763

Chloroethane	75-00-3	0.001	ppm
Chloroform	67-66-3	0.001	ppm
Chloromethane	74-87-3	0.001	ppm
cis-1,2-Dichloroethene	156-59-2	0.001	ppm
cis-1,3-Dichloropropene	10061-01-5	0.001	ppm
Cyclohexane	110-82-7	0.001	ppm
Dibromochloromethane	124-48-1	0.001	ppm
Dibromofluoromethane	1868-53-7	0.04	ppm
Methylene Chloride	75-09-2	0.001	ppm
Dichlorodifluoromethane	75-71-8	0.001	ppm
Methylcyclohexane	108-87-2	0.001	ppm
Ethyl Ether	60-29-7	0.001	ppm
Ethylbenzene	100-41-4	0.001	ppm
Heptane	142-82-5	0.001	ppm
Hexachlorobutadiene	87-68-3	0.001	ppm
Hexachloroethane	67-72-1	0.001	ppm
Iodomethane	74-88-4	0.001	ppm
Isopropylbenzene	98-82-8	0.001	ppm
Methyl Acetate	79-20-9	0.001	ppm
Xylene, Ortho	95-47-6	0.001	ppm
Dibromomethane	74-95-3	0.001	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
7090571	8260 Centurian Working Surrogate	Sep-13-07	Diane L. VanMale	Aug-31-08	Sep-18-07 10:47 by DLV	0.1
7120760	8260 Working Standard B 12-28-07	Dec-28-07	Diane L. VanMale	Feb-15-08	Dec-28-07 15:20 by DLV	0.001

Reviewed By Date

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By
1	VO7. -1													
2	VO7. -2													
3	VO7. -3													
4	VO7. -4													
5	VO7. -5													
6	VO7. -6													
7	VO7. -7													
8	VO7. -8													
9	VO7. -9													
10	VO7. -10													
11	VO7. -11													
12	VO7. -12													
13	VO7. -13													
14	VO7. -14													
15	VO7. -15													
16	VO7. -16													
17	VO7. -17													
18	VO7. -18													

Appendix AA

TriMatrix Laboratories, Inc.

Pipet Calibration Verification Acceptance Window Calculations

Pipet ID:	SPK-15	**Balance Used:**	IN-1
Manufacturer:	Socorex	**Manufacturer:**	Mettler
Model Number:	Calibra 822.1000	**Model Number:**	AE-163
Serial Number:	10111410	**Serial Number:**	B86211

I. 20 Weight (g) Measurements Using Each Pipet Calibration Mass

Date	Replicate Number	Volume 1 uL 100	Volume 2 uL 200	Volume 3 uL 250	Volume 4 uL 300	Volume 5 uL 500	Volume 6 uL 1000
05/04/01	1	0.0975	0.1978	0.2455	0.2924	0.4904	0.9819
05/04/01	2	0.0981	0.1978	0.2461	0.2944	0.5077	0.9958
05/04/01	3	0.0987	0.1980	0.2458	0.2945	0.5028	1.0019
05/04/01	4	0.0983	0.1973	0.2475	0.2933	0.5041	1.0011
05/04/01	5	0.1002	0.1986	0.2479	0.2936	0.4993	1.0030
05/04/01	6	0.0997	0.1984	0.2474	0.2945	0.5001	1.0006
05/04/01	7	0.1000	0.1973	0.2471	0.2942	0.5005	1.0034
05/07/01	8	0.0983	0.1965	0.2449	0.2940	0.4950	0.9930
05/07/01	9	0.0975	0.1971	0.2451	0.2936	0.4939	0.9922
05/07/01	10	0.0970	0.1933	0.2440	0.2948	0.4943	0.9943
05/07/01	11	0.0972	0.1970	0.2447	0.2927	0.4939	0.9938
05/07/01	12	0.0973	0.1963	0.2452	0.2935	0.4935	0.9928
05/07/01	13	0.0966	0.1970	0.2445	0.2939	0.4934	0.9935
05/07/01	14	0.0977	0.1961	0.2438	0.2937	0.4935	0.9920
05/08/01	15	0.0992	0.1969	0.2464	0.2973	0.4937	0.9884
05/08/01	16	0.0990	0.1970	0.2463	0.2953	0.4913	0.9918
05/08/01	17	0.0989	0.1959	0.2479	0.2977	0.4924	0.9841
05/08/01	18	0.0981	0.2012	0.2474	0.2963	0.4932	0.9858
05/08/01	19	0.0985	0.1954	0.2469	0.2962	0.4948	0.9856
05/08/01	20	0.0990	0.1976	0.2462	0.2975	0.4930	0.9865

II. Pipet Calibration Acceptance Window Calculations

Standard Deviation:	0.00100755	0.00151653	0.00128600	0.00156309	0.00469383	0.00647504
Random Error:	0.00302265	0.00454960	0.00385799	0.00468928	0.01408148	0.01942513
Average Percent Recovery	98.3%	98.6%	98.4%	98.2%	99.2%	99.3%
Acceptance Window Low:	0.0970	0.1955	0.2461	0.2953	0.4859	0.9806
Acceptance Window High:	0.1030	0.2045	0.2539	0.3047	0.5141	1.0194

TriMatrix Laboratories, Inc.

Metals Laboratory Spiking Pipet Calibration Logbook

Pipet ID	Calibration Volume	Acceptance Window (g)	Date: Initials:		Date: Initials:		Date: Initials:		Date: Initials:		Date: Initials:	
			g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail
B-8	20 uL	0.0192-0.0208										
	50 uL	0.0495-0.0505										
	100 uL	0.0981-0.1019										
SPK-5	10 uL	0.0096-0.0104										
	25 uL	0.0245-0.0255										
	50 uL	0.0485-0.0515										
	100 uL	0.0982-0.1018										
SPK-12	4.00 mL	3.91-4.09										
	8.00 mL	7.84-8.16										
	9.00 mL	8.84-9.16										
	10.00 mL	9.85-10.15										
SPK-15	100 uL	0.0970-0.1030										
	200 uL	0.1955-0.2045										
	250 uL	0.2461-0.2539										
	300 uL	0.2953-0.3047										
	500 uL	0.4859-0.5141										
	1000 uL	0.9806-1.0194										
SPK-16	100 uL	0.0953-0.1047										
	200 uL	0.1944-0.2056										
	250 uL	0.2457-0.2543										
	300 uL	0.2918-0.3082										
	500 uL	0.4922-0.5078										
	1000 uL	0.9641-1.0359										

Appendix AB

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Digestion of Mercury in Water, Wastewater and Aqueous Waste

EPA Method 245.1
SW-846 Method 7470A

APPROVALS:

Area Supervisor: _____ Date: 9-12-08
Denise S. Coffey

QA Officer: _____ Date: 9-11-08
Tom C. Boocher

Operations Manager: _____ Date: 9/12/08
Jeff P. Glaser

Procedure Number: GR-01-140
Revision Number: 0.3

Date Initiated: 2/19/03
Effective Date: 10/15/08

Date Revised: 8/4/08
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 21

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the digestion of total mercury (inorganic and organic) in samples of groundwater, potable water, surface water, saline water, mobility leachate, and in aqueous domestic and industrial waste.

1.2 The minimum reporting limit is 0.2 ug/L.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

2.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, all client samples and quality control must be digested to convert organo-mercury complexes to inorganic mercury.

3.2 A measured sample aliquot, acids and potassium permanganate-potassium persulfate are transferred to a block digestion vessel and refluxed for 2 hours at 90-95° C.

3.3 The digestate is then prepared for analysis by semi-automated cold vapor atomic absorption spectrometry with the addition of hydroxylamine hydrochloride to reduce excess permanganate.

3.4 Inorganic mercury is converted to mercury in the Hg^{2+} state during the digestion, for detection and quantitation.

4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision

5.3 TriMatrix SOP GR-01-123, *Mercury in Aqueous and Solid Samples by Semi-Automated Cold Vapor Atomic Absorption Spectrometry*, latest revision

Appendix AC

TriMatrix Laboratories, Inc.

Sample Collection, Packing and Return

All supplied containers are pre-cleaned, no additional cleaning is required. Some containers have preservatives present in them. Please do not rinse or overfill. Removal of some or all of the preservative may result in qualified data. Most of the chemicals used as preservatives are hazardous. Use caution when handling. Do not breathe or come in physical contact with these chemicals. For your safety, please read the enclosed Material Safety Data Sheets.

When conducting soil sampling, please clean off any residual soil from the outside of the containers. This will help prevent cross contamination of other samples in the cooler.

Please fill out all sample identification tags as completely as possible.

Please fill out the enclosed Chain of Custody form for adequate sample tracking.

The temperature requirement for the receipt of most environmental samples is 4 ±2° C. Temperatures that exceed this range are subject to qualification and data rejection by regulatory agencies. Following the instructions below provides the best chance of achieving and maintaining this temperature and avoiding qualified data.

- Samples should be collected and placed on ice as soon as possible. It is much more difficult to cool down warm samples.
- When possible, sample containers should be sealed in zip-lock containers. This prevents cross contamination and protects the sample labels from moisture that could render them illegible.
- Do not overfill the cooler with samples. Overfilling the cooler limits the space available for ice.
- Surround the sides and the tops of the sample containers with loose, cubed, ice. Surrounding the samples with ice is the most efficient way of cooling them. Do not use individual small bags of ice. Do not simply lay a bag of ice on top of the samples.
- Place the temperature blank in a representative location in the cooler, not in the middle of a bag of ice.
- Secure all paperwork in a zip-lock bag and place in the cooler. Seal the cooler closed.
- When shipping the coolers back to TriMatrix, complete the enclosed FedEx Airbill and attach it to the cooler. Samples shipped during the week for standard overnight delivery typically arrive the next day between 9:00 and 10:00 a.m. Saturday deliveries must be approved by your project chemist. When shipping samples for a Saturday delivery, select Priority Overnight and Saturday Delivery on the FedEx Airbill.

Please call your TriMatrix project chemist at 1-616-975-4500 if you require any further instructions, or to notify them of the pending arrival of any non-scheduled samples.

Thank You,
TriMatrix Laboratories, Inc.

MATERIAL SAFETY DATA SHEET

Sodium hydroxide, 50 wt% solution in water

Section 1 - Chemical Product and Company Identification

MSDS Name: Sodium hydroxide, 50 wt% solution in water

Catalog Numbers: 25986-0000, 25986-0025, 25986-0050, 38021-0000, 38021-0025, 38021-5000

Synonyms: Caustic soda

Company Identification:
Acros Organics BVBA
Janssen Pharmaceuticalaan 3a
2440 Geel, Belgium

Company Identification: (USA)
Acros Organics
One Reagent Lane
Fair Lawn, NJ 07410

For information in the US, call: 800-ACROS-01

For information in Europe, call: +32 14 57 52 11

Emergency Number, Europe: +32 14 57 52 99

Emergency Number US: 201-796-7100

CHEMTREC Phone Number, US: 800-424-9300

CHEMTREC Phone Number, Europe: 703-527-3887

Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name:	%	EINECS#	Hazard Symbols:	Risk Phrases:
1310-73-2	Sodium hydroxide	50	215-185-5	C	35
7732-18-5	Water	50	231-791-2		

Text for R-phrases: see Section 16

Hazard Symbols: C

Risk Phrases: 35

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Causes severe burns.

Potential Health Effects

Eye: Causes severe eye burns.

Skin: Causes skin burns. May cause deep, penetrating ulcers of the skin.

Ingestion: Causes gastrointestinal tract burns. Causes severe pain, nausea, vomiting, diarrhea, and shock. May cause corrosion and permanent tissue destruction of the esophagus and digestive tract.

Inhalation: Irritation may lead to chemical pneumonitis and pulmonary edema. Causes severe irritation of upper respiratory tract with coughing, burns, breathing difficulty, and possible coma. Causes chemical burns to the respiratory tract.

Chronic: Prolonged or repeated skin contact may cause dermatitis.

Section 4 - First Aid Measures

Eyes: Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid immediately.

Skin: Get medical aid immediately. Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Discard contaminated clothing in a manner which limits further exposure.

Ingestion: Do not induce vomiting. Get medical aid immediately.

Inhalation: Get medical aid immediately. Remove from exposure and move to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

Notes to Physician: Treat symptomatically and supportively.

Section 5 - Fire Fighting Measures

General Information: As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. May react with metals and lead to the formation of flammable hydrogen gas.

Extinguishing Media: Use foam, dry chemical, or carbon dioxide.

Section 6 - Accidental Release Measures

General Information: Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks: Absorb spill with inert material (e.g. vermiculite, sand or earth), then place in suitable container.

Section 7 - Handling and Storage

Handling: Wash thoroughly after handling. Use with adequate ventilation. Do not allow water to get into the container because of violent reaction. Do not breathe dust, vapor, mist, or gas. Do not get in eyes, on skin, or on clothing. Use only in a chemical fume hood.

Storage: Store in a cool, dry place. Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances. Corrosives area. Store under an inert atmosphere.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls:

Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower. Use adequate ventilation to keep airborne concentrations low. Use adequate general or local exhaust ventilation to keep airborne concentrations below the permissible exposure limits.

Exposure Limits

CAS# 1310-73-2:

United Kingdom, WEL - STEL: 2 mg/m3 STEL

United States OSHA: 2 mg/m3 TWA

Belgium - TWA: 2 mg/m3 VLE

France - VME: 2 mg/m3 VME

Germany: 2 mg/m3 TWA (inhalable fraction)

Japan: 2 mg/m3 Ceiling

Malaysia: 2 mg/m3 Ceiling

Spain: 2 mg/m3 VLA-EC

CAS# 7732-18-5:

Personal Protective Equipment

Eyes: Wear chemical splash goggles.

Skin: Wear appropriate protective gloves to prevent skin exposure.

Clothing: Wear appropriate protective clothing to prevent skin exposure.

Respirators: Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

Section 9 - Physical and Chemical Properties

Physical State: Viscous liquid

Color: clear colorless

Odor: Not available

pH: >13

Vapor Pressure: Not available

Viscosity: Not available

Boiling Point: 145 deg C (293.00°F)

Freezing/Melting Point: 12 deg C (53.60°F)

Autoignition Temperature: Not available

Flash Point: Not available

Explosion Limits: Lower: Not available

Explosion Limits: Upper: Not available

Decomposition Temperature: Not available

Solubility in water: Soluble

Specific Gravity/Density: 1.525

Molecular Formula: HNaO

Molecular Weight: 40

Section 10 - Stability and Reactivity

Chemical Stability: Stable at room temperature in closed containers under normal storage and handling conditions. Absorbs carbon dioxide from the air.

Conditions to Avoid: Incompatible materials, exposure to air.

Incompatibilities with Water, acids, aluminum, chlorinated solvents,

Other Materials	copper, copper alloys, magnesium, phosphorus, zinc, tin, organic materials.
Hazardous Decomposition Products	Sodium oxide.
Hazardous Polymerization	Will not occur.

Section 11 - Toxicological Information

RTECS#:
CAS# 1310-73-2: WB4900000
CAS# 7732-18-5: ZC0110000

LD50/LC50:
RTECS:
CAS# 1310-73-2: Draize test, rabbit, eye: 400 ug Mild;
Draize test, rabbit, eye: 1% Severe;
Draize test, rabbit, eye: 50 ug/24H Severe;
Draize test, rabbit, eye: 1 mg/24H Severe;
Draize test, rabbit, skin: 500 mg/24H Severe;
.
RTECS:
CAS# 7732-18-5: Oral, rat: LD50 = >90 mL/kg;
.
Other:

Carcinogenicity: Sodium hydroxide - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.
Water - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.

Other: See actual entry in RTECS for complete information.

Section 12 - Ecological Information

Not available

Section 13 - Disposal Considerations

Dispose of in a manner consistent with federal, state, and local regulations.

Section 14 - Transport Information

	IATA	IMO	RID/ADR
Shipping Name:	SODIUM HYDROXIDE SOLUTION	SODIUM HYDROXIDE SOLUTION	SODIUM HYDROXIDE SOLUTION
Hazard Class:	8	8	8
UN			

Number:	1824	1824	1824
Packing Group:	II	II	II

USA RQ: CAS# 1310-73-2: 1000 lb final RQ; 454 kg final RQ

Section 15 - Regulatory Information

European / International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols: C

Risk Phrases:

R 35 Causes severe burns.

Safety Phrases:

S 26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

S 37/39 Wear suitable gloves and eye/face protection.

S 45 In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

WGK (Water Danger/Protection)

CAS# 1310-73-2: 1

CAS# 7732-18-5: Not available

Canada

CAS# 1310-73-2 is listed on Canada's DSL List

CAS# 7732-18-5 is listed on Canada's DSL List

US Federal

TSCA

CAS# 1310-73-2 is listed on the TSCA Inventory.

CAS# 7732-18-5 is listed on the TSCA Inventory.

Section 16 - Other Information

Text for R-phrases from Section 2

R 35 Causes severe burns.

MSDS Creation Date: 7/16/1996

Revision #1 Date 5/05/2004

Revisions were made in Sections: General revision.

The information above is believed to be accurate and represents the best

information currently available to us. However, we make no warranty of merchantibility or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall the company be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential, or exemplary damages howsoever arising, even if the company has been advised of the possibility of such damages.

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR THE COLLECTION OF VOLATILE ORGANIC DRINKING WATER SAMPLES

Open the water tap and allow the system to flush until the water temperature has stabilized (usually about 10 minutes). Reduce the water flow and carefully collect a set of duplicate samples. It is important that the flow is slow enough that no air bubbles pass through the sample as the vial is being filled. Each 40 mL vial has been pre-preserved with 25 mg of ascorbic acid preservative. Fill sample vials to just overflowing, taking care not to flush out the ascorbic acid.

Prior to sealing the set of vials, each sample must also be preserved with 1:1 hydrochloric acid. Using the supplied eyedropper and vial of HCl, carefully add 2 drops of HCl to each vial. The HCl must be added after the collection of the sample. DO NOT add the HCl to the sample vial prior to collecting the sample.

CAUTION: The 1:1 HCl is very acidic. Handle with care.

NOTE: If the sample foams vigorously when the HCl is added, discard that set of samples. Collect a new set, omitting the addition of the HCl. These samples must be flagged as "not acidified" on the chain of custody.

Seal the vials, invert, and mix for 1 minute. Verify that the sealed and mixed vial is bubble and headspace free. Sample data generated from vials received with headspace will be qualified accordingly.

The samples must be chilled to about 4° C when collected, and maintained at that temperature until analysis. Samples must be packaged for shipment with sufficient ice to ensure they arrive at the laboratory with a substantial amount of ice remaining in the cooler. Do not use Blue Ice. Surrounding the samples with crushed or cubed ice is strongly recommended. Samples received at the laboratory within 6 hours of collection may not have sufficient time to cool to 4° C. Provided that they have been correctly packed in ice, no qualifications will be necessary. Samples received in excess of 6 hours of the time of collection that exceed the required preservation temperature will be qualified accordingly.

Please call 1-616-975-4500 and speak to your project chemist if you have any questions. Thank you.

Dissolved Sulfide Sample Collection and Preservation

To measure dissolved sulfide, insoluble matter in the sample must first be removed. This is accomplished by producing an aluminum hydroxide floc using sodium hydroxide and aluminum chloride. The flocculent is allowed to settle and the supernatant decanted off and preserved with zinc acetate. It is important that there is no headspace present in the bottle after the addition of the aluminum chloride. The vials containing the final decanted sample must also be headspace free. If you have any questions on the treatment procedures described below, please contact your project chemist at 1-616-975-4500.

Supplies

Quantity	*Item*
1 per sample	250 mL amber bottle containing 0.5 mL (10 drops) 6N NaOH
2 per sample	40 mL VOA vials, each containing 0.1 mL (2 drops) 2N Zinc Acetate per sample
2 or 3	eye droppers
1	Container of Aluminum Chloride. Enough has been sent to allow for the addition of 10 drops (0.5 mL) to each 250 mL sample.

Procedure

1.0 Collect the sample in the 250 mL amber bottle containing the NaOH. Completely fill the bottle (must be enough sample so when capped it is headspace free).

2.0 Immediately add 10 drops of the Aluminum Chloride solution.

3.0 Mix the sample by holding the bottle in an upright position and rotating your wrist back and forth for 1 minute.

4.0 Allow the sample to settle for 5 to 15 minutes (long enough to allow the flocculent to settle to the bottom of the bottle but not longer than 15 minutes). Wait only as long as necessary to collect 80 mL of supernatant.

5.0 Carefully decant the supernatant into the (2) 40 mL VOA vials containing the 2N zinc acetate. Completely fill the vials with sample so they are headspace free.

6.0 The sample remaining in the 250 mL amber bottle is caustic. Please return the partially filled bottle to TriMatrix for disposal.

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR SULFIDE SAMPLE COLLECTION

The amber, 500 mL, light green-tagged bottles supplied for sulfide sample collection have been pre-preserved with 1 mL of 2N zinc acetate. Sulfide samples must also be preserved with sodium hydroxide to a pH of ≥9; however, to correctly preserve the sulfide in the sample the addition of the sodium hydroxide must be made *after* the sample has been combined with the zinc acetate. A 4 mL vial containing 2 mL of 10N sodium hydroxide has been included with every 500 mL sulfide sample bottle for this purpose.

With a minimum of aeration, fill a 500 mL bottle up to the neck with sample. Cap and gently swirl to mix the sample and the zinc acetate. Open the sample bottle and transfer all of the sodium hydroxide from one of the 4 mL vials. Carefully add more sample to fill the 500 mL bottle, cap and mix. The filled sample container should be headspace free.

CAUTION: The 10N sodium hydroxide solution is very caustic. Handle with care.

Please call 1-616-975-4500 and speak to your project chemist with any questions. Thank you.

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR AVAILABLE CYANIDE SAMPLE COLLECTION

Two sample containers must be collected at each sample point. One container will be treated with lead carbonate and sodium hydroxide, and the second with only sodium hydroxide (see below and the attached flowchart). A form titled "Available Cyanide Sample Treatment Record" has been provided to document all field pre-treatment activities. Please complete it as you collect and treat each sample. If you have any questions on the treatment procedures described below, please contact your project chemist at 1-616-975-4500.

IMPORTANT: To avoid analyte loss it is **required** that all sample treatments occur within 15 minutes of sample collection.

CAUTION: All containers labeled as Sodium Hydroxide and Lead Carbonate/Sodium Hydroxide contain 1.3 mL of 10N sodium hydroxide. This solution is very caustic. Avoid skin contact. Handle with care.

CAUTION: All containers labeled as Lead Carbonate contain 0.25 g of solid lead carbonate. Avoid inhalation and skin contact.

1.0 Sample Collection Equipment

Per Sample
- One membrane filter
- One plastic powder funnel
- One sheet of filter paper
- One Lead Carbonate bottle
- One Lead Carbonate/Sodium Hydroxide bottle
- One Sodium Hydroxide bottle

A hand pump (not provided) is also required to perform this procedure

2.0 Collecting a Lead Carbonate/Sodium Hydroxide Pre-Treated Sample

If the sample contains particulates, begin with section 2.1. If the sample is particulate free, begin with section 2.2.

2.1 Sample Contains Particulate Matter

If the sample contains particulate matter that would be removed upon filtration, the sample must be filtered prior to the lead carbonate pre-treatment to avoid the loss of any cyanides associated with the particulate matter. Using a powder funnel and a sheet of filter paper, filter the sample into the bottle labeled Lead Carbonate. Filter enough sample to fill the bottle up to its neck. Place the used filter paper into the bottle labeled Lead Carbonate/Sodium Hydroxide. Cap the Lead Carbonate bottle and gently swirl to mix the sample and the lead carbonate. The sulfide will react with the lead carbonate

and precipitate out as lead sulfide. The sample must now be filtered through a membrane filter to prevent the loss of any cyanide through reaction with the precipitated lead sulfide. Using a new membrane filter apparatus and a hand pump, filter the sample. Transfer the filtrate into the Lead Carbonate/Sodium Hydroxide bottle containing the used filter paper. Do not pre-rinse the container or fill to overflowing, as a loss of the particulate matter and sodium hydroxide will result. Proceed to section 3.0.

2.2 Sample Particulate Free

With a minimum of aeration, fill the 250 mL bottle labeled Lead Carbonate up to the neck with sample. Cap and gently swirl to mix the sample and the lead carbonate. The sulfide will react with the lead carbonate and precipitate out as lead sulfide. The sample must now be filtered through a membrane filter to prevent the loss of any cyanide through reaction with the precipitated lead sulfide. Using a new membrane filter apparatus and a hand pump, filter the sample. Transfer the filtrate collected into the bottle labeled Lead Carbonate/Sodium Hydroxide. Do not pre-rinse the container or fill to overflowing to avoid the loss of the sodium hydroxide.

3.0 Collecting a Sodium Hydroxide Pre-Treated Sample

With a minimum of aeration fill the 250 mL bottle labeled Sodium Hydroxide with sample. Do not pre-rinse the container or fill to overflowing to avoid the loss of the sodium hydroxide.

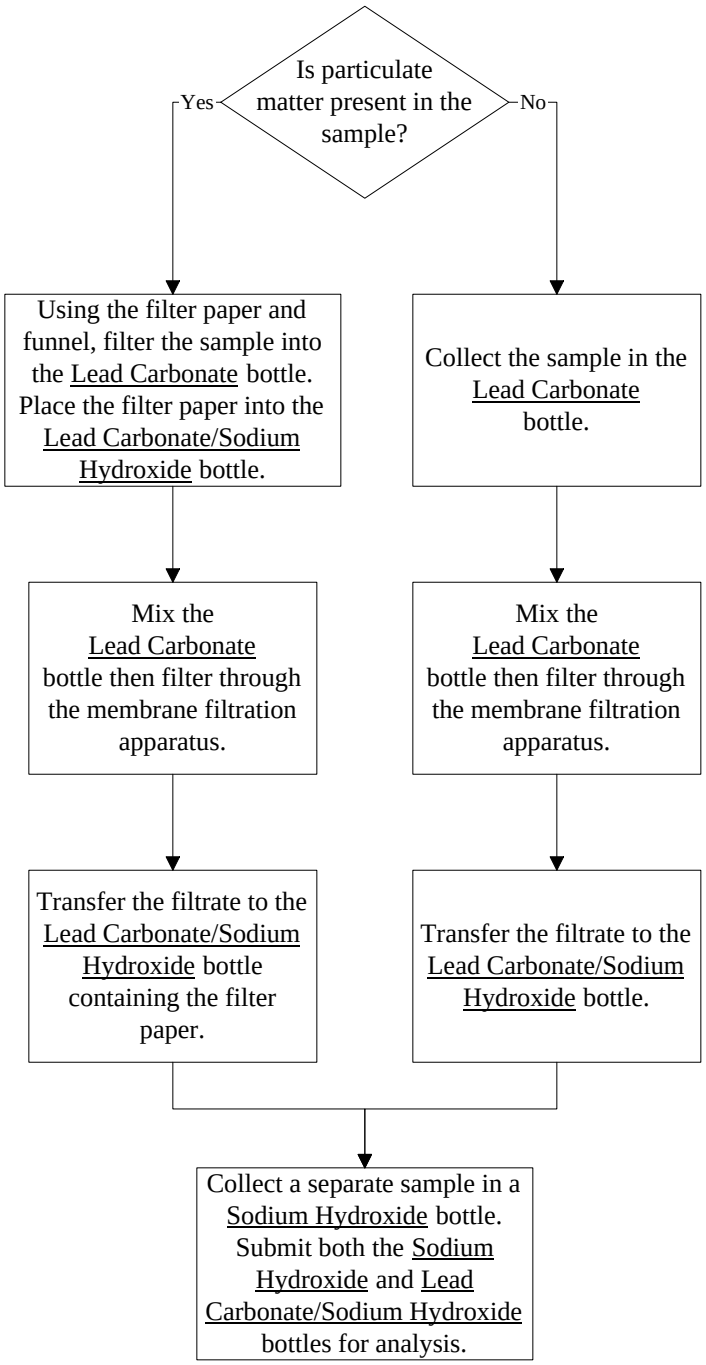
4.0 Collect all Paperwork and Return the Samples to TriMatrix

Place all samples in the cooler. Surround the samples with ice. To avoid data qualification all samples must be received at a temperature of between 0 and 6° C. Seal all paperwork in the resealable bag. Place the sealed bag containing the paperwork. Place all plastic powder funnels and unopened membrane filters in the cooler. Seal the cooler and return it to TriMatrix.

If you have any questions, please call TriMatrix at 1-616-975-4500 and speak with your project chemist. Thank you.

TriMatrix Laboratories, Inc.

Available Cyanide Sample Collection Flowchart



Appendix AD

TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court SE Grand Rapids, MI 49512
Phone (616) 975-4500 Fax (616) 942-7463
www.trimatrixlabs.com

Chain of Custody Record

COC No.

For Lab Use Only

Cart

VOA Rack/Tray

Receipt Log No.

Project Chemist

Work Order No.

Client Name

Address

Phone

Fax

Project Name

Client Project No. / P.O. No.

Invoice To
(●) Client ( ) Other (comments)

Contact/Report To

Analyses Requested

Container Type (corresponds to Container Packing List)

Schedule	Matrix Code	Laboratory Sample Number		Sample ID	Cooler ID	Sample Date	Sample Time	Comp / Grab	Matrix	Number of Containers Submitted	Total	Sample Comments
			1									
			2									
			3									
			4									
			5									
			6									
			7									
			8									
			9									
			10									

⇦ PRESERVATIVES

A NONE pH~7
B HNO_3 pH<2
C H_2SO_4 pH<2
D 1+1 HCl pH<2
E NaOH pH>12
F ZnAc/NaOH pH>9
G MeOH
H Other (note below)

Sampled By (print)

Sampler's Signature

Company

How Shipped?

Tracking No.

Comments

1. Relinquished By Date Time

1. Received By Date Time

2. Relinquished By Date Time

2. Received By Date Time

3. Relinquished By Date Time

3. Received For Lab By Date Time

Appendix AE

TriMatrix Laboratories, Inc.

pH Strip Calibration Logbook

Date	Lot #	pH 4	pH 7	pH 10	Area

pH STRIP CALIBRATION CRITERIA CORRECTIVE ACTION

1. The acceptance range for the strips is to read the exact pH of the buffer being checked. The wide range strips must pass this criteria at all three levels, 4, 7, and 10. The narrow range pH 5-7 strips are only checked at a pH of 7.

2. If the pH strips do NOT read at their appropriate levels, that lot number must NOT be used. Return them to purchasing.

Appendix AF

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

The collection of the sample is the starting point for the generation of quality data. It is the responsibility of TriMatrix to provide the client who collects the sample with sample collection instructions, which ensure sample integrity. Also, where applicable TriMatrix also supplies the client with appropriate clean sample containers and preservative chemicals; these glass containers are purchased new and certified as clean and vendors such as I-Chem Research and Fischer Scientific.

Sampling and Preservation Requirements for certain common environmental analyses are listed in the following table: (NOTE: Holding times are based on EPA guidelines for CLP, NPDES, and RCRA).

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
ORGANICS							
Volatile	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	8015, 8021, 8260	Yellow/Black
Halocarbons*	Water	14 days	4° C	2-40 mL VOA vials	40 mL each	601	Yellow/Black
	Water	14 days	4° C/HCl to pH <2	2-40 mL VOA vials	40 mL each	601, 8015, 8021, 8260	Yellow
	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar	fill the jar	8015, 8021, 8260	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8015, 8021, 8260	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8015, 8021, 8260	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8015, 8021, 8260	Light Yellow
Volatile	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	602	Yellow/Black
Aromatics*	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	602, 8021, 8260	Yellow
	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar or	fill the jar	8021, 8260	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8021, 8260	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8021, 8260	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8021, 8260	Light Yellow
Acrolein*	Water	3 days	4° C	2-40 mL VOA vials	40 mL each	624	Yellow/Black
	Water	14 days	4° C/HCl to pH 4-5	2-40 mL VOA vials	40 mL each	624	Yellow
Acrylonitrile*	Water	14 days	4° C	2-40 mL VOA vials	40 mL each	624	Yellow/Black
	Water	14 days	4°C/HCl to pH 4-5	2-40 mL VOA vials	40 mL each	624	Yellow
TPH-GRO	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	8015	Yellow/Black
	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	8015	Yellow
TPH-GRO/PVOC	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	Wisconsin PUBL-SW-140	Yellow
TPH-GRO	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar or	fill the jar	8015	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8015	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8015	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8015	Light Yellow
TPH-GRO/PVOC	Soil (Encore)	48 hours/21 days	4° C	10 or 25 g Encore	See Table 1 in Method	Wisconsin PUBL-SW-140	Label on Bag

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	Wisconsin PUBL-SW-140	Light Yellow
Petroleum	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8015	Salmon
Hydrocarbons	Water	7 days/47 days	4° C/HCl to pH <2.0	1000 mL amber glass bottle	1000 mL	Wisconsin PUBL-SW-141	Gray
(DRO)	Soil/Waste (High Level Bulk)	14 days/54 days	4° C	60 mL glass jar or	fill the jar	8015	Manila
	Soil/Waste	10 days/47 days	4° C	Tared VOC vial	See Table 1 in Method	Wisconsin PUBL-SW-141	Gray
Pesticides	Water	7 days/47 days	4° C/pH 5-9	1000 mL amber glass bottle	1000 mL	608	Yellow/White
PCBs	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	608, 8082	Salmon
Methoxychlor	Water	7 days/47 days	4° C/pH 6-8	1000 mL amber glass bottle	1000 mL	608.2	Yellow/White
Pesticides	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8081	Manila
PCBs	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8082	Manila
PCB Oils	Oil	N/A	None	40 mL VOA vial	20 mL	8082	Manila
Organo-phosphorous	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8141	Salmon
Pesticides	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8141	Manila
Phenoxy Acid Herbicides	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8151	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8151	Manila
Polynuclear aromatic	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	610, 8100	Salmon
Hydrocarbons*	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8310, 8270	Manila
Acid Extractables	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8041, 8270	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8041, 8270	Manila
Base/Neutral Extractables	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8270	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8270	Manila
TCLP-							
Volatiles	Soil/Waste	14 days/28 days	4° C	60 mL glass jar	100 g	1311	Yellow/Black
Semi-Volatiles	Soil/Waste	14 days/21 days/61 days	4° C	125 mL glass jar	250 g	1311	Manila
Metals	Soil/Waste	180 days/360 days (Hg-28 days/56 days)	None	125 mL glass jar	250 g	1311	Manila
Pesticide/Herbicide	Soil/Waste	14 days/21 days/61 days	4° C	125 mL glass jar	250 g	1311	Manila
Dioxins/	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	Screen-625	Salmon

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Furans							
	Soil/Waste	None Required	4° C	60 mL glass jar	fill the jar	Screen-625	Manila

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
METALS							
Metals, Total (including phosphorus)	Water	6 months	HNO_3 to pH <2.0	500 mL plastic bottle	500 mL	6010/6020/200.7/200.8	Red
Metals, Dissolved (including phosphorus)	Water	6 months	HNO_3 to pH <2.0	500 mL plastic bottle	500 mL	6010/6020/200.7/200.8	Red/White Stripe
	Soil/Waste	6 months	None	250 mL plastic bottle	50 g	6010/6020	White
Mercury Cold Vapor	Water	28 days	HNO_3 to pH <2.0	500 mL plastic bottle	500 mL	245.1, 7470	Red
	Soil/Waste	28 days	4° C	250 mL plastic bottle	50 g	7471	White
Low-Level	Water	28 days	None	500 mL borosilicate glass bottle**	500 mL	1631	Label on Bag

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
INORGANICS							
Color (Apparent)	Water	48 hours	4° C	125 mL plastic bottle	100 mL	110.2	Green
Color (True)	Water	48 hours	4° C	125 mL plastic bottle	100 mL	110.2	Green
Oil & Grease (HEM and SGT)	Water	28 days	4° C/H_2SO_4 to pH <2.0	1000 mL glass bottle	1000 mL	9070/1664	Dark Blue
	Soil/Waste	28 days	None	60 mL glass jar	50 g	9071	Manila
Specific Conductance	Water	28 days	4° C	125 mL plastic bottle	100 mL	2510 B./120.1/9050	Green
Acidity	Water	14 days	4° C	125 mL plastic bottle	100 mL	2310 B.	Green
pH	Water	24 hours	4° C	125 mL plastic bottle	100 mL	150.1/9041/4500-H B.	Green
	Soil/Waste	24 hours	4° C	60 mL glass jar	50 g	9040/9041/9045	
Alkalinity	Water	14 days	4° C	125 mL plastic bottle	100 mL	310.1/2320 B.	Green
Hardness	Water	6 months	HNO_3 to pH <2.0	125 mL plastic bottle	100 mL	130.2/2340 C.	Red
Biochemical Oxygen Demand (BOD)	Water	48 hours	4° C	1000 mL plastic bottle	1000 mL	5210 B.	Green
Chemical Oxygen Demand (COD)	Water	28 days	4° C/H_2SO_4 to pH <2.0	125 mL plastic bottle	100 mL	410.4/5220 D.	Dark Blue
Chromium (Hexavalent)	Water	24 hours	4° C	500 mL plastic bottle	500 mL	7196A, 3500-Cr B.	Green
	Soil/Waste	30 days/24 hours	4° C	60 mL glass jar	50 g	7196A	Manila
Organic Carbon (TOC)	Water	28 days	4° C/H_2SO_4 to pH <2.0	3-40 mL VOA vials	40 mL	415.1/5310 D./9060	Salmon
	Soil/Waste	28 days	4° C	60 mL glass jar	10 g	MSA 29-3.5.2/415.1/9060	Manila

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Ortho-Phosphate	Water	48 hours	4° C	125 mL plastic bottle	100 mL	365.1/4500-P E.	Green
Total Phosphorus	Water	28 days	H_2SO_4 to pH <2.0	125 mL plastic bottle	100 mL	365.1/4500-P F.	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	365.1/4500-P F.	Manila
Total Kjeldahl Nitrogen (TKN)	Water	28 days	4° C/H_2SO_4 to pH <2.0	125 mL plastic bottle	100 mL	351.2	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	351.2	Manila
Ammonia	Water	28 days	4° C/H_2SO_4 to pH <2.0	125 mL plastic bottle (500 mL for wastewater)	100 mL (200 mL for wastewater)	350.1/4500-NH_3 G.	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	350.1/4500-NH_3 G.	Manila
Nitrite	Water	48 hours	4° C	125 mL plastic bottle	100 mL	300.0/9056/353.2/354.1/ 4500 NO_2-B/4500 NO_2-F	Green
	Soil/Waste	28 days/48 hours	4° C	60 mL glass jar	50 g	353.2/9056	Manila
Nitrate	Water	48 hours	4° C	125 mL plastic bottle	100 mL	300.0/9056/353.2/4500 NO_3-F	Green
	Soil/Waste	28 days/48 hours	4° C	60 mL glass jar	50 g	9056/353.2/4500 NO_3-F	Manila
Nitrite plus Nitrate (No distinction between NO_2 and NO_3)	Water	28 days	4° C/H_2SO_4 to pH <2.0	125 mL plastic bottle	100 mL	353.2/4500 NO_3-F	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	353.2/4500 NO_3-F	Manila
Total Volatile Solids	Water	7 days	4° C	125 mL plastic bottle	100 mL	160.4	Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	2540-G	Manila
Turbidity	Water	48 hours	4° C	125 mL plastic bottle	100 mL	180.1/2130 B.	Green
Sulfate	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/375.4/9038	Green
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	9056/375.2/9038/4500 SO_4-F	Manila
Sulfite	Water	48 hours	4° C/3 mL 1% EDTA	125 mL plastic bottle	100 mL	377.1	Manila

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Sulfide, Total	Water	7 days	4° C/Pre-Preserved with Zinc Acetate; NaOH Added in field to pH ≥9	125 mL plastic bottle	100 mL	9034/376.1/376.2/4500 S_2-D 4500 S_2-F	Light Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	9034	Manila
Cyanide*	Water	14 days	4° C/NaOH to pH >12	1000 mL plastic bottle	1000 mL	335.2/335.4/9012/9014	Light Blue
	Soil/Waste	14 days	4° C	60 mL glass jar	50 g	9012/9014	Manila
Cyanide, Available	Water	14 days	1 Lead Carbonate bottle 1 Lead Carbnate/NaOH bottle 1 NaOH bottle	125 mL amber glass bottles	125 mL	OIA-1677	Light Blue
Coliform Fecal and Total	Water	24 hours	4° C/$Na_2S_2O_3$	Sterile plastic bottle or Whirl-Pak	100 mL	9222-D/9223-B	White
Bromide	Water	28 days	4° C	125 mL plastic bottle	100 mL	9056/ASTM D1246-88	Green
Chloride	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/325.2/4500-Cl E.	Green
	Soil	28 days	4° C	60 mL glass jar	50 g	9056/325.2/4500-Cl E.	Manila
Chlorine Residual	Water	Analyze Immediately	4° C	125 mL plastic bottle	100 mL	HACH-8167	Green
Total Solids (% Moisture)	Water	7 days	4° C	125 mL plastic bottle	100 mL	160.3/2540 B.	Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	3550	Manila
Total Dissolved Solids (TDS)	Water	7 days	4° C	1000 mL plastic bottle	1000 mL	160.1/2540 C.	Green
Total Suspended Solids (TSS)	Water	7 days	4° C	1000 mL plastic bottle	1000 mL	160.2/2540 D.	Green
Fluoride	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/4500-F C.	Green
	Soil	28 days	4° C	60 mL glass jar	50 g	9056	Manila

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Organic Halogen (TOX)	Water	28 days	4° C/H_2SO_4 to pH <2.0	500 mL amber glass bottle	500 mL	9020	Lilac
	Soil	28 days	4° C	60 mL glass jar	50 g	9023	Manila
Phenolics	Water	28 days	4° C/H_2SO_4 to pH <2.0	500 mL amber glass bottle	100 mL	420.2/420.4/9066	Brown
	Soil	28 days	4° C	60 mL glass jar	50 g	9066	Manila
Surfactants (MBAS)	Water	48 hours	4° C	1000 mL plastic bottle	400 mL	425.1/5540 C.	Green
Flash Point	Solid/Liquid/Waste	N/A	None	Clear glass wide mouth jar. 60 mL unless otherwisespecified.	100 g	1010/1020	White
	Waste	N/A	None		100 g	1010/1020	White
Corrosivity (pH and Method 1110)	Waste	N/A	None	(Appropriate to Sample) 500 mL glass or plastic bottle	500 mL	9040/9041/1110	White
Paint Filter (Free Liquids)	Soil/Waste	N/A	None	(Appropriate to Sample) 250 mL glass jar or 125 mL plastic bottle	100 g	9095	White
Radiologicals (Alpha + Beta, Alpha, Beta, Ra 226, Ra 228	Water	6 months	HNO_3 to pH <2.0	1000 mL plastic bottles or 1000 mL glass bottle	1000 mL		White
Reactivity (Releasable CN and S)	Waste	14 days CN, 7 days S	4° C	(Appropriate to Sample 125 mL plastic bottle or 60 mL glass jar	10 g	SW- 846 Chapter 7	White

*Sample must also be preserved with Sodium Thiosulfate or Ascorbic Acid if chlorinated
**All low-level mercury bottles are stored filled with 5 mL of concentrated HCl and Millipore water
NOTE: For Organics parameters, container lid should be Teflon.
NOTE: For Inorganic parameters, container lid should be plastic or Teflon lined.
NOTE: When testing for several like parameters (ICP metals, Ion Chromatograph anions), one container per sample is sufficient. For example, a sample to be tested for the 13 priority pollutant metals needs one 500 mL container.

Appendix AG

Client: **S**
Project: **Analytical Services**

Project Manager: **Jennifer L. Rice**
Date Received: **Dec-08-08 10:00**

Department: **Metals** Analysis: _____

Lab Number / Sample Name	*Container*	*Removed by (Signature)*	*Date & Time Removed*	*Date & Time Returned*	*Consumed?*	*Extract Container*
0812144-01 Pugged CKD						
0812144-02 Pugged CKD						
0812144-03 Pugged CKD						
0812144-04 Pugged CKD						

Appendix AH

TriMatrix Laboratories, Inc.

Non-Conformance Investigation Report

Client:_____ **Project Number:**_____

Sample Number(s):_____ **Date Initiated:**_____ **Date Due:**_____

Initiated By:_____ **Document Control Number:** _____

Investigation Resulting From: ☐ Internal Observation ☑ **Client Complaint** ☐ Audit ☐ Failing PT Sample

I. Area of Non-Conformance:

☐ Sample Receiving / Storage ☐ Bottle Prep ☐ Client Services / Reporting ☐ Other_____

☑ Inorganic (**Wet Chemistry** / Metals) Laboratory ☐ Organic (Volatile / Semi-Volatile / Extraction) Laboratory

II. Description of Non-Conformance:

_____

_____

_____

_____

III. Explanation of Investigation into Non-Conformance:

_____

_____

_____

_____ Initials: _____ Date: _____

IV. Resolution:

_____

_____

_____

_____

_____ Initials: _____ Date: _____

V. Follow-Up (if required):

_____

_____

_____

_____ Initials: _____ Date: _____

VI. Reviewed By:

QA Manager:_____ **Area Manager:**_____

Date Completed:_____

Appendix AI

TriMatrix Laboratories, Inc.

Preventive Action Investigation

Initiated By: _____ **Document Control Number:** _____

Date Initiated: _____ **Date Due:** _____

Investigation Resulting From: ☐ Internal Observation ☐ Client Complaint ☐ Audit ☐ Failing PE Sample

I. Area of Preventive Action:

☐ Sample Receiving / Storage ☐ Bottle Prep ☐ Client Services / Reporting ☐ Other _____

☐ Inorganic (Wet Chemistry / Metals) Laboratory ☐ Organic (Volatile / Semi-Volatile / Extraction) Laboratory

II. Description and Proposed Solutions:

_____

_____

_____

_____

III. Action Plan and Implementation Schedule:

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____ Initials: _____ Date: _____

V. Follow-Up to Monitor Effectiveness:

_____

_____

_____

_____

_____

_____ Initials: _____ Date: _____

VI. Reviewed By:

QA Manager: _____ **Area Manager:** _____

Date Completed: _____

8.0 GLOSSARY OF TERMS

ABSORBANCE - a measure of the decrease in incident light passing through a sample into the detector. It is defined mathematically as:

$$A = \left(\frac{I\,(solvent)}{I\,(solution)} \right) - \frac{\log Io}{I}$$

ALIQUOT - a measured portion of a field sample taken for analysis.

ANALYSIS DATE/TIME - the date and time of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE - the element or ion an analysis seeks to determine; the component of interest.

ANALYTICAL SAMPLE - any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification and continuing calibration blank. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, interference check samples (ICS), CRDL standard for AA (CRA), CRDL standard for ICP (CRI), laboratory control sample (LCS), method preparation blank (MPB), laboratory fortified blank (LFB), and linear range analysis sample (LRS).

AUTOZERO - zeroing the instrument at the proper wavelength. It is equivalent to running a blank to set the absorbance to zero.

AVERAGE INTENSITY - the average of two different responses from a detector.

BACKGROUND CORRECTION - a technique to compensate for background contribution to the instrument signal in the determination.

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank, Instrument Blank, Storage Blank, and Sulfur Blank.

BATCH - a group of samples prepared at the same time in the same location using the same method.

BREAKDOWN - a measure of the decomposition of certain analytes (i.e. DDT and Endrin) into by-products.

4-BROMOFLUOROBENZENE (BFB) - the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses.

CALIBRATION - the establishment of an analytical curve based on the measured response of known standards.

CALIBRATION BLANK - a volume of laboratory reagent or other inert carrier matrix.

CALIBRATION STANDARDS - a series of known standards used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected during external calibration. The calibration factor is analogous to the Response Factor (RF) calculated during internal calibration.

CASE - a finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office. A Case consists of one or more Sample Delivery Groups.

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.

CONTINUING CALIBRATION VERIFICATION - analytical standard run at periodic intervals to verify the initial calibration of the system.

CONTRACT REQUIRED DETECTION LIMIT (CRDL) - minimum level of detection acceptable as specified by the project to report.

CONTROL LIMITS - a range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

CORRELATION COEFFICIENT - the number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more dependent they are the closer the number (r) is. Determined on the basis of the least squares regression.

DAY - unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - an official record of the sample preparation (digestion).

DISSOLVED METALS - analyte elements which have not been digested prior to analysis and which will pass through a 0.45 um filter.

DRY WEIGHT - the weight of a sample analyzed based on percent solids. The weight after drying in an oven.

DUPLICATE - a second aliquot of sample that is treated the same as the original in order to determine the precision of the collection.

EXTRACTED ION CURRENT PROFILE (EICP) - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).

EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.

FIELD BLANK - any sample submitted from the field identified as a blank.

FIELD SAMPLE - Material received to be analyzed that is contained in single or multiple containers and identified by a unique Sample Number.

GAS CHROMATOGRAPH (GC) - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are either volatized directly from the sample (VOA water and low-soil), from the sample extract (VOA medium soil), or injected as an extracted sample (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture Detector (ECD). In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

HOLD TIME - the maximum allowable elapsed time expressed in hours or days from the time the sample is collected until the time of its pre-treatment or analysis.

INDEPENDENT STANDARD – an externally prepared standard solution composed of analytes from a different source than those used in the standards for the initial calibration.

INDUCTIVELY COUPLED PLASMA (ICP) - a technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

IN-HOUSE - at the laboratories facility.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the instrument.

INJECTION - introduction of the analytical sample into the instrument excitation system for the purpose of measuring concentration of an analyte.

INSTRUMENT CALIBRATION - Series of analytical standards at different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument.

INSTRUMENT DETECTION LIMIT (IDL) - determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

INSTRUMENT CHECK SAMPLE - a solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INSTRUMENT CHECK STANDARD - a multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.

INTERFERENTS - substances which affect the analysis for the element of interest.

INTERNAL STANDARDS - compounds added to analytical and quality control samples at a known concentration prior to analysis. In the methods that require them, internal standards are used as the basis for quantitation of the target compounds.

INSTRUMENT/ANALYTICAL BLANK - a blank designed to determine the level of contamination associated with the analytical instrument.

INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

SECOND SOURCE CALIBRATION VERIFICATION (SCV) STANDARD - a standard prepared from a source other than that used to prepare the quantitation standard, and used to verify the initial calibration curve.

BLANK SPIKE - a control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

LABORATORY RECEIPT DATE - the date on which a sample is received as recorded on the chain of custody.

LINEAR RANGE, LINEAR DYNAMIC RANGE - the concentration range over which the determinative instrument's analytical curve remains linear.

MATRIX - the predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

MATRIX EFFECT - in general, the effect of the particular sample matrix on the constituents with which is contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

MATRIX SPIKE - aliquot of a matrix spiked with known quantities of target compounds and subjected to the entire analytical procedure. Matrix spikes are used to indicate the efficiency of the method on the matrix by measuring the recovery of the spiked analyte.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method relative to the matrix.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards that are carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD OF STANDARD ADDITIONS (MSA) - the addition of 3 increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept and y-intercept are determined by least-square analysis. The analyte concentration is determined by the absolute value of the x-intercept.

Ideally, the spike volume is low relative to the sample volume (approximately 10% of the volume). Standard addition may counteract matrix effects; it will not counteract special effects. Also referred to as Standard Addition.

m/z - Mass to charge ratio, synonymous with "m/e"

NARRATIVE - portion of the data package which includes laboratory, contract, case and sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

PERCENT DIFFERENCE (%D) - to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference).

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105° C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105° C, including water.

PERCENT SOLIDS - the proportion of solid in a soil sample determined by drying an aliquot of the sample at 105° C.

PERFORMANCE EVALUATION MIXTURE - a calibration solution of specific analytes used to evaluate both recovery and percent breakdown as measures of performance.

PERFORMANCE TESTING (PT) SAMPLE - a single blind sample of known composition obtained from an external provider for analysis. Used by clients and regulatory agencies to evaluate laboratory performance.

PREPARATION BLANK (reagent blank, method blank) - an analytical control that contains distilled/deionized water and reagents, which is carried through the entire analytical procedure – digested/distilled/extracted and analyzed. An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.

PRIMARY QUANTITATION ION - a specific ion used to quantitate a target analyte.

PROTOCOL - a compilation of procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control.

PURGE AND TRAP (DEVICE) - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto a gas chromatographic column.

PURGEABLES – non-water soluble volatile organic compounds.

QUALITY CONTROL SAMPLE - a solution obtained from an outside source having known concentration values to be used to verify the calibration.

REAGENT BLANK - a volume of deionized, distilled water containing the same reagent matrix as the calibration standards carried through the entire analytical scheme.

REAGENT WATER - water in which an interferent is not observed at or above the minimum detection limit of the parameters of interest.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - The relative percent difference is based on the mean of two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RTc}{RTis}$$

where,

RTc = Retention time for the target or surrogate compound in continuing calibration.

Rtis = Retention time for the internal standard in calibration standard or in a sample.

RELATIVE STANDARD DEVIATION (RSD) - the variation of a series of results based on the standard deviation and average. Typically used in the evaluation of initial calibration curves.

$$RSD = \frac{SD}{\text{Average RF}}$$

RESOLUTION - the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RESPONSE - or Instrumental Response: a measurement of the output of the detector in which the intensity of the signal is proportionate to the concentration detected.

RESPONSE FACTOR (RF) - a measure of the relative response of an analyte compared to an internal standard. The RF is determined by the following equation:

$$RF = \left(\frac{Ax}{Ais} \times \frac{Cis}{Cx} \right)$$

where:

A = area of the characteristic ion measured

C = concentration

is = internal standard

x = analyte of interest

RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is

dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

ROUNDING RULES - If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no known figures beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

RUN - a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements.

SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE NUMBER - a unique identification number designated for each sample. The Sample Number appears on all laboratory documents which contain information on that sample.

SEMIVOLATILE COMPOUNDS - compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

SENSITIVITY - the slope of the analytical curve, i.e., functional relationship between emission intensity and concentration.

SERIAL DILUTION – a series of dilutions to attain a less concentrated solution.

SOIL - synonymous with soil/sediment or sediment as used herein.

SONICATOR - a device that uses the energy from controlled ultrasound applications to mix, disperse, and dissolve organic materials from a given solid matrix.

SPECTRA - a plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

STORAGE BLANK - a reagent water aliquot stored with samples and analyzed on a weekly basis for VOCs. The storage blank is used to determine the potential for sample contamination occurring during storage.

STOCK SOLUTION - a standard solution prepared from neat materials diluted to derive other standards.

SURROGATES (Surrogate Standard) - for semivolatiles, volatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be present in the sample.

SUSPENDED - those particulates in suspension which are retained by a 0.45 um membrane filter.

TENTATIVELY IDENTIFIED COMPOUNDS (TIC) - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

TOTAL METALS – analytes from the sample which have been digested to complete solvency prior to analysis.

TWELVE-HOUR TIME PERIOD - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the

laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. The injection time of the last analyses in the batch must be made within 12 hours of the injection time of BFB of DFTPP.

VOLATILE COMPOUNDS – non-water soluble compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

WET WEIGHT - the mass of a sample aliquot including moisture (un-dried) that is used for analysis.

WIDE BORE CAPILLARY COLUMN - a gas chromatographic column with an internal diameter (ID) that is greater than 0.32 mm. Columns with lesser diameters are classified as capillary columns.

APPENDIX D

WESTON Procured Subcontractor Laboratory Preventative Maintenance Procedures

APPENDIX D

WESTON Procured Subcontractor Laboratory Preventative Maintenance Procedures

Kirstin McCracken: QA Manager, BA Geography, 15 years experience

The QA Manager (QM) is responsible for ensuring the laboratory's quality system and quality assurance manual meet the requirements given in the company's Corporate Quality Management Plan (CQMP). The QAM implements, maintains and improves the quality system. The QAM provides quality system and ethics training to all new personnel ensuring all personnel understand their contributions to the quality system and the QAM evaluates the effectiveness of the training program. The QAM performs and oversees internal systems, data, and special audits and performs other surveillance activities to monitor for trends and opportunities for continuous improvement. The QAM oversees the maintenance of QA records, certifications and accreditations. The QAM is responsible for ensuring communication regarding the effectiveness of the quality system takes place at all levels within the laboratory. The QAM has the final authority to accept or reject data and to stop work in progress in the event the practice compromises the validity or integrity of analytical data. The QAM has an indirect reporting relationship to an assigned Quality Director, is independent of laboratory operations and has responsibility and authority to ensure the continuous implementation of the quality System.

Jim Madison: Project Manager, BS Geology/Environmental Science, 25 years exp.

The Project Manager(s) is responsible for direct communication with the client, coordination of laboratory services, work scheduling and dissemination of project requirements to the laboratory operation. The PM writes project narratives, performs tertiary data review, investigates and resolves technical and service related issues that arise during the course of the project.

Cover Page:

Quality Assurance Manual

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Facility Distribution No. Electronic **Distributed To: Facility Server**

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Title Page:

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
BR-QA-003	Document Control & Updating (However Named, Sec. 3.4.1)
BR-QA-004	Complaint Resolution (However Named, Sec .10.1)
BR-QA-011	Lab Training (However Named, Sec. 17.3)
See Corporate SOP	Writing SOPs (However Named, Sec. 19.2)
BR-QA-011	DOCs (However Named, Sec. 19.4.2)
BR-QA-005	MDLs (However Named, Sec. 19.7)
BR-QA-006	MI (However Named, Sec. 19.14.1)
BR-QA-020	Subsampling (However Named, 22.5)
BR-SM-001	Sample Receipt / Login, etc… (However Named, Sec. 23.2.1.3)

SECTION 3. INTRODUCTION (NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Burlington'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 IIIB, November 2004, Final Update IV, January 2008.*
- *U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.1, April 2009.*
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Statement of Work for Inorganics & Organics Analysis, SOM 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.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- 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 solids and sediments, drinking water, non-potable water, waste, tissue, air and saline/estuarine samples. Specialty capabilities include air toxics testing, geotechnical testing, and tissue preparation and analysis. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical 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 on the company's website or may be obtained from any laboratory Project Manager (PM). 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 Corporate Quality Management Plan (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 for Document Control, laboratory SOP No. BR-QA-003.

SECTION 4. ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica Burlington 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 Burlington 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 Burlington laboratory.

4.2.2 Laboratory Director

The Laboratory Director (LD) has responsibility and authority for the overall quality, safety, financial, technical, human resource and service performance of the laboratory. The LD oversees the daily operations of the laboratory and provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program. The LD responsibilities include supervision of staff, setting goals and objectives for both the business and the employees and achieving the financial, business, technical and quality objectives of the laboratory. The LD ensures timely compliance with audits and corrective actions, and is responsible for maintaining a working environment that encourages open, constructive problem solving for continuous improvement.

4.2.3 Quality Assurance Manager

The QA Manager (QM) is responsible for ensuring the laboratory's quality system and quality assurance manual meet the requirements given in the company's Corporate Quality Management Plan (CQMP). The QAM implements, maintains and improves the quality system. The QAM provides quality system and ethics training to all new personnel ensuring all personnel understand their contributions to the quality system and the QAM evaluates the effectiveness of the training program. The QAM performs and oversees internal systems, data, and special audits and performs other surveillance activities to monitor for trends and

opportunities for continuous improvement. The QAM oversees the maintenance of QA records, certifications and accreditations. The QAM is responsible for ensuring communication regarding the effectiveness of the quality system takes place at all levels within the laboratory. The QAM has the final authority to accept or reject data and to stop work in progress in the event the practice compromises the validity or integrity of analytical data. The QAM has an indirect reporting relationship to an assigned Quality Director, is independent of laboratory operations and has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025 including:

- 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.

4.2.4 Technical Director (TD)

The Technical Director is responsible for compliance with the ISO 17025 Standard. The Technical Director solves day to day technical issues, provides technical training and guidance to laboratory staff, project managers, and clients, investigates technical issues identified by QA, and directs evaluation of new methods.

4.2.5 Customer Service Manager (CSM)

The Customer Service Manager is responsible for supervision of the project management staff. The CSM compiles and interprets the receipts forecast and tracks and maintains information for various revenue reports. The CSM is responsible for the evaluation and preparation of bids and proposals for new business opportunities and overseeing the project management bid activity for existing client base.

4.2.6 Project Manager (PM)

The Project Manager(s) is responsible for direct communication with the client, coordination of laboratory services, work scheduling and dissemination of project requirements to the laboratory operation. The PM writes project narratives, performs tertiary data review, investigates and resolves technical and service related issues that arise during the course of the project.

4.2.7 Department Manager/Supervisor/Coordinator

The Department Manager has responsibilities for a defined portion of the laboratory that include work scheduling, development, execution and supervision of analytical procedures including SOP review and revision, secondary data review, staff training, goal setting and monitoring lab activities to achieve the quality objectives set forth in the LQM and standard operating procedures. A department supervisor or coordinator may be designated by the Department Manager to perform some of these job responsibilities. Department Supervisors or Coordinators report to the Department Manager.

4.2.8 Chemist/Analyst

Chemists and analysts responsible for analysis of samples and generation of analytical data in accordance with the requirements set forth in the CQMP, this document, company policy and procedure, test method and process standard operating procedures, and project specifications.

4.2.9 Sample Custodian

The Sample Custodian(s) is responsible for the receipt and handling of samples within the laboratory. Responsibilities include adherence to the laboratory sample acceptance policy, initiation of internal chain of custody, when needed, sample log-in and tracking, sample security and storage, and sample disposal.

4.2.10 IT Staff

The IT Staff are responsible for the design and maintenance of the laboratory's computer hardware and software. Responsibilities include preparation and maintenance of the Information Systems Quality Manual (ISQM), implementation and validation of new data systems, network administration, hardware and software maintenance, review, creation of electronic data deliverables (EDD) and the provision of technical support to all laboratory staff.

4.2.11 Environmental Health & Safety Coordinator

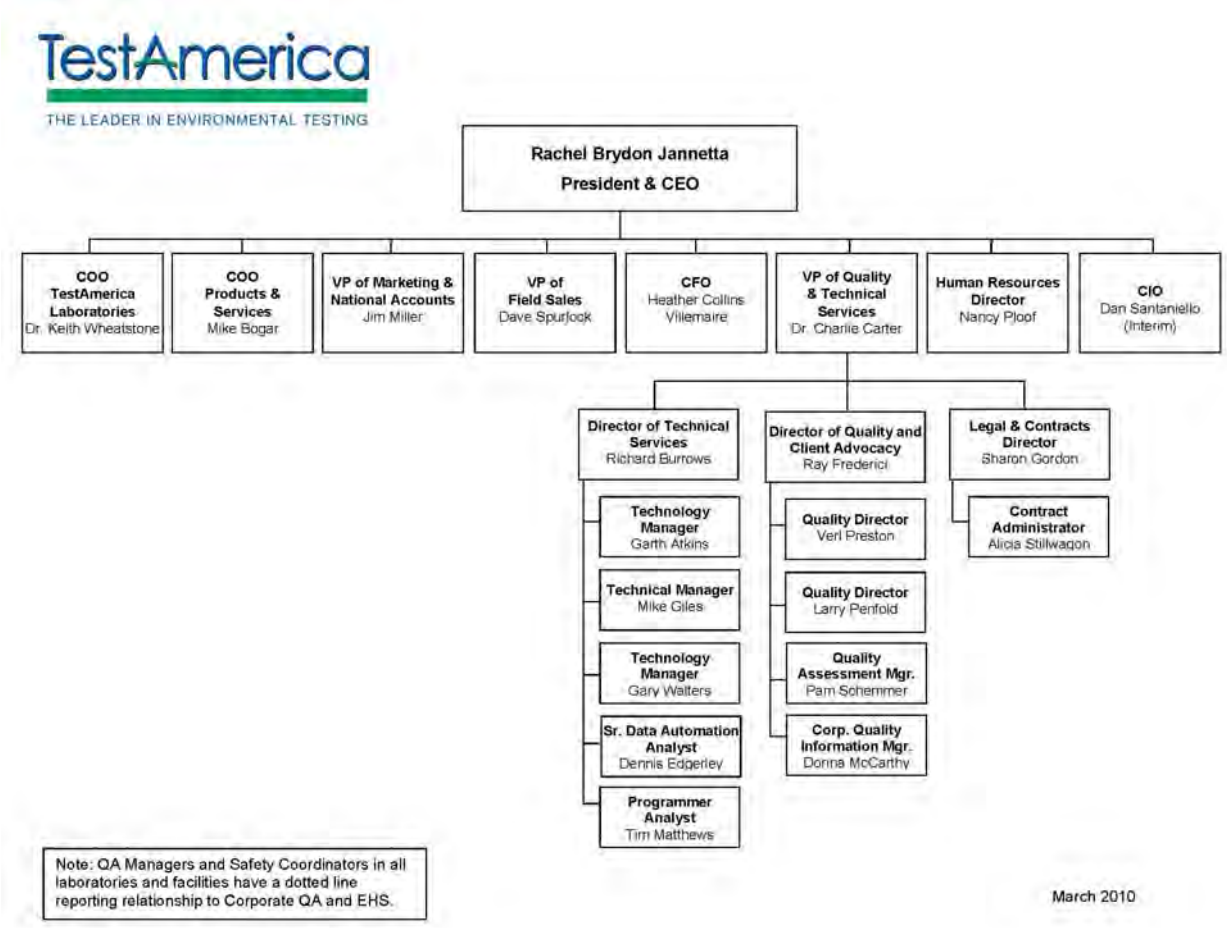
The Employee Health and Safety Coordinator is responsible for administering the EH&S program in order to provide a safe, healthy working environment for all employees. The EH&S Coordinator responsibilities include the monitoring of all work areas to detect unsafe conditions, acts, and potential hazards, enforcement of environmental, health, and safety policies and procedures and ensuring regulatory compliance with local, state, and federal laws. The EH& S Coordinator provides safety and health recommendations to laboratory management in conjunction with the facility safety committee, develops the facility Integrated Contingency Plan and coordinates the facility's Emergency Response Team.

4.3 DEPUTIES

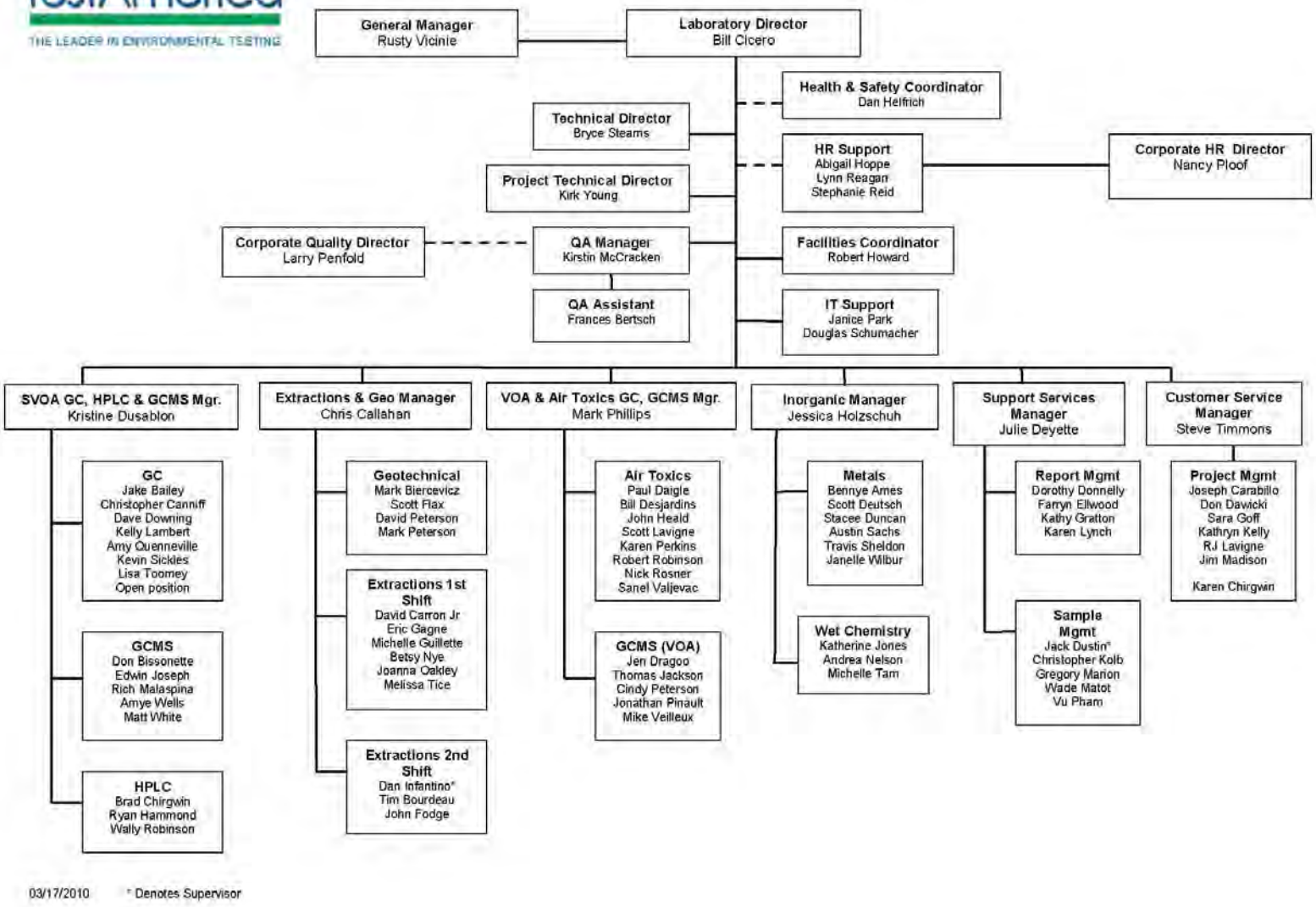
The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
William S. Cicero Laboratory Director	Bryce E. Stearns, Technical Director Kirstin L. McCracken, QA Manager Steve Timmons, Customer Service Manger
Kirstin L. McCracken QA Manager	William S. Cicero, Laboratory Director Bryce E. Stearns, Technical Director Frances S. Bertsch, QA Assistant
Bryce E. Stearns Technical Director	William S. Cicero, Laboratory Director Kirstin L. McCracken, QA Manager
Dan E. Helfrich EHS Coordinator	William S. Cicero, Laboratory Director

Figure 4-1. Corporate and Laboratory Organization Charts



Burlington 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 quality assurance manual 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 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 Limit of Detection) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains the LIMS database that summarizes the precision and accuracy acceptability limits for performed analyses. The database includes an effective date, is updated each time new limits are generated and are 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 is contained in laboratory SOP BR-QA-013.

5.6 STATISTICAL QUALITY CONTROL

If a method defines the QC limits, the method limits are used. In the absence of method specific or project specific limits, the laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. These limits are maintained by the QA Manager or her designee in the Laboratory Information Management System (LIMS).

Statistical limits for spikes and surrogates are generated from recent data in the LIMS database following the guidelines described in Section 24.

Current QC limits are entered and maintained in the LIMS 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

Trend analysis is performed to determine if adjustments need to be made or for corrective actions to methods. These procedures are provided in laboratory SOP BR-QA-013.

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 BR-QA-003.

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 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. 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 annually 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. BR-QA-003. A controlled copy of the QA Manual is issued under controlled distribution to the controlled distribution directory located on a network server. Uncontrolled copies must not be used within the laboratory. Previous revisions of the Quality Manual are stored by the QA department.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP.

Forms, worksheets, work instructions and information are organized by department and are maintained by QA. The procedure for the care of these documents is in SOP BR-QA-003.

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 BR-QA-003.

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 review the work request at each stage of evaluation.

For routine projects a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. 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.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which network laboratory will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. 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. Appropriate personnel include but are not limited to:

- Legal & Contracts Director
- General Manager
- Laboratory Director
- Laboratory Customer Service Manager
- Laboratory Project Manager
- Laboratory QA Manager and Technical Director
- Laboratory Department Managers

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 local account representative submits the final proposal to the client.

The Legal & Contracts Director maintains copies of all signed contracts and a copy is kept locally in the project file.

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. Records of review are retained by the Project Manager.

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.

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.

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 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 also communicated to the laboratory by the PM and 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.

The Technical Director, QA Staff or Department Managers are 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 because 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 SOP's 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) and any relevant program requirements, such as compliance to DoD QSM 4.1. 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 Department of Defense US Army Corps of Engineers and the USDA, require notification prior to placing such work.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM or Regional Account Executive (RAE) or Customer Service Manager 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, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director 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 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's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-1) and the form is retained in the project folder. 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. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

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 - Subcontracted Sample Form

Date/Time: _____

Subcontracted Laboratory Information:

- Subcontractor's Name: _____
- Subcontractor Point of Contact: _____
- Subcontractor's Address: _____
- Subcontractor's Phone: _____
- Analyte/Method: _____
- Certified for State of Origin: _____
- NELAC Certified: Yes_____No_____
- USDA Permit (__Domestic __ Foreign) Yes_____No_____
- A2LA (or ISO 17025) Certified: Yes_____No_____
- CLP-like Required: Yes_____No_____
  (Full doc required)
- Requested Sample Due Date: _____
  (Must be put on COC)

Project Manager: _____

Laboratory Sample # Range: _____
(Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #): _____

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature_____**Date**_____

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. The laboratory maintains an on-site consignment system for frequently used items. Analysts may check items out of the on-site consignment system as needed or place orders through the purchasing system. Orders placed through the purchasing system are approved by designated personnel.

9.3.2 Receiving

It is the responsibility of each department manager to receive the shipment. All orders are checked on receipt to ensure the material received matches material ordered and 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 SOPs expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.
- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained in the relevant laboratory section where the chemical is used.

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 daily. The minimum total pressure must be 500 psig for Argon/Methane and Hydrogen an all cylinders directly connected to instruments. The minimum total pressure must be 120 psig for Helium, 100 psig for liquid Argon and 30 psig for Nitrogen. If pressure exceeds the minimum pressure the tank must be replaced. 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- umho/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/Supervisors 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.

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 the LIMS. 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 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.

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 IT Department. Software certificates supplied by the vendors are filed with the IT Department. The manufacturer's operation manual is retained at the bench.

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 or the 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 SOP BR-QA-004.

10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to laboratory SOP BR-QA-004.

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 (DM) for resolution. The DM may elect to discuss it with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents the approach in the analytical record and the PM includes a discussion of the departure in the case narrative.

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. Project specific procedures must be documented by the PM in the project record.

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, the Technical Director, QA Manager or Department Manager 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, and the Department Managers. 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, 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 ECO's 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, Department Manager) 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 Corrective Action Reports (CAR) (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) – may be 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.

12.2.2 **Corrective Action Report (CAR)** – may be used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of nonconformance
- 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

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 CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 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 Laboratory Director, QA Manager or Technical Director 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 NCR 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 CAR is entered into a spreadsheet for tracking purposes. The spreadsheet is subsequently used to ensure CAR are closed and actions taken effective.
- The QA Manager reviews corrective actions 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 ~~15.2.4~~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 NCR or project memo.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table 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 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 - Corrective Action Report

CORRECTIVE ACTION REPORT (CAR)		Tracking Number:	
Initiated By:		Assigned To:	
Initiation Date:		CC:	
Due Date:			
Section 1: Describe Problem & Attach Supporting Documentation As Needed			
Corrective Action Prompted By:			
Recurring NCR	Internal Audit	External Audit	Complaint / Other
Section 2: Root Cause Analysis			
Section 3: Describe Actions Required to Correct & Prevent Problem			
Section 4: QA Review and Close Out			
Action Taken Was:	Acceptable	Not Acceptable	Other
Comments:			
Close Out Date:		Closed By:	
Section 5: Follow Up (From Close-Out Date)			
Time Frame:	Performed By:	Date:	Is action taken preventing recurrence?
1 Month			
3 Month			
6 Month			
Comments:			

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Table 12-1.

Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Primary Analyst, Secondary Data Review Analyst)*	- See details in Method SOP	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Primary Analyst, Secondary Data Review Analyst)*	- See details in Method SOP	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Primary Analyst, Secondary Data Review Analyst)*	- % Recovery within limits documented in SOP.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Primary Analyst, Secondary Data Review Analyst)*	- See details in Method SOP	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Primary Analyst, Secondary Data Review Analyst)*	- % Recovery within limits documented in SOP.	- 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) *(Primary Analyst, Secondary Data Review Analyst)*	- % Recovery within limits documented in SOP.	- 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 *(Primary Analyst, Secondary Data Review Analyst)*	- % Recovery within limits documented in SOP.	- Individual sample must be repeated. Place comment in LIMS.

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Method Blank (MB) *(Primary Analyst, Secondary Data Review Analyst)*	< Reporting Limit or as specified by regulatory program, such as DoD.	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.
Proficiency Testing (PT) Samples *(Primary Analyst, Secondary Data Review Analyst, Department Manager)*	- 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, 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, QA Manager)*	- SOP BR-QA-004	- 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 17 for an example) *(QA Manager, Lab Director)*	- 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, Department Manager)*	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

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 such as the addition of new equipment or personnel. Procedures for minimization of potential risks inherent with a new event or change are described in various laboratory standard operating procedures.

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. 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 retained by QA, Department Managers, electronically or by report management depending on the record type.

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
	EH&S Manual, Permits, Disposal Records	7 years

	Record Types [1]:	Retention Time:
	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 an offsite location 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 access logs are maintained. 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. Example: 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 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. 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 chain of custody should 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. These procedures are described in laboratory SOP BR-QA-014. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. 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 electronic bench sheets are used to record and file data. Standard and reagent information is 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 BR-QA-014.

- 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.
- Instrumentation identification and instrument operating conditions/parameters.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation
- 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.

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. These procedures are described in laboratory SOPs BR-QA-003 and BR-QA-014.

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 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: Air, Potable Water, Non-Potable Water and Soil.

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. When requested, 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 tracked using a spreadsheet. 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 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 and 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 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 may 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 staffs that possess 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.

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 **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
Technical Director - Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of 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 are 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 BR-QA-011.

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 22,000 sq 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.

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

Electronic access cards are issued to each employee and building keys are distributed to authorized employees as necessary.

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.
- 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 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*

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.

A demonstration of capability (DOC, Lab SOP BR-QA-011) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel.

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.

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 Figure 19-1 as 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.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

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 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. 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.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP BR-QA-005 for details on the laboratory's MDL process.

19.8 INSTRUMENT DETECTION LIMITS (IDL)

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.

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.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

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 for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). 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. 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.

For DoD QSM work, once the detection limit 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 BR-QA-005 Method Detection Limits (MDLs/DLs) for further details.

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 QSM work, 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 $\pm$ 1 reporting limit for samples $\leq$ 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 Area Supervisor 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. The laboratory is currently running the TALS LIMS system which is a 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 an SQL database 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.

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 laboratory SOP BR-QA-006.

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. 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 the LIMS formatter specified by the PM.

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.

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 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 out lined in several SOP BR-QA-019 to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP for manual integration, BR-

QA-005. 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. Approximately 15% 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 Department Manager, Project Manager, QA Manager or Technical Director, as necessary. Corrective action is initiated whenever necessary.

19.14.4.4 The results are then entered or directly transferred into the computer database and a report is prepared for the client.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the report for 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.

19.14.5 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. BR-QA-005 entitled Manual Integration.

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

Demonstration of Capability
Certification Statement

Date: _____ Page ____ of _____

Laboratory Name: TestAmerica Burlington
Laboratory Address: 30 Community Drive, Suite 11, South Burlington, VT 05403

Analyst Name: _____

Matrix: _____

Test Method: _____ Prep Method: _____

We, the undersigned, CERTIFY that:

The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the various* Program, have method the Demonstration of Capability.

The test method(s) was performed by the analyst identified on this certification.

A copy of the test method(s) and the laboratory-specific SOP are available for all personnel on-site.

The data associated with the demonstration of capability are true, accurate, complete and self-explanatory (1).

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 assessors.

Bryce E. Stearns		
Technical Director	Signature	Date
Kirstin L. McCracken		
QA Manager	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with Supporting Data
Accurate: Based on good laboratory practices consistent with sound scientific principles / practices.
Complete: Includes the results of supporting performance testing.
Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

* Various – includes any regulatory program for which the laboratory performs work including but not limited to: NELAC accreditation program, Department of Defense, USEPA CLP, state and other federal programs.

FQA013.03.30.10.2
TestAmerica Burlington

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 be / are also outlined in analytical SOPs or instrument manuals.

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, field sampling devices, 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.

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 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 the analytical record. More information on this subject can be found in the laboratory SOP for the calibration of thermometers, BR-QA-012 and individual laboratory SOPs.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each day.

Ovens, waterbaths and incubators are monitored on days of use.

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/or the analytical record.

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. Glass micro-syringes are considered the same as Class A glassware.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements. Laboratory procedures for the verification of mechanical pipette are described in laboratory SOP BR-QA-008.

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.

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.

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 (Avg 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.

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 at a different time or a different preparation 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 initially and 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 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 here 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.

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. Instrumentation List

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Automated Distillation Apparatus	Westco	Easy Dist	1090	2002	NEW
Automated Distillation Apparatus	Westco	Easy Dist	1091	2002	NEW
COD	HACH	UNKNOWN	11000022452	UNKNOWN	UNKNOWN
CVAA	Leeman (CV3)	HydraAA112-0064-1	2031	2003	NEW
CVAA	Leeman (CV4)	HydraAA112-0064-1	8015	2008	NEW
GC/ECD/ECD	Agilent (7424)	6890	US10332093	2003	NEW
GC/ECD/ECD	Hewlett-Packard (2620)	5890II	3203A41056	1998	UNKNOWN
GC/ECD/ECD	Agilent (3283)	6890II	US10202136	2008	NEW
GC/ECD/ECD	Hewlett-Packard (2618)	5890II	3203A41055	1987	UNKNOWN
GC/ECD/ECD	Hewlett-Packard (2624)	5890II	3203A41057	1998	UNKNOWN
GC/ECD/ECD	Agilent (7227)	6890II	CN10602095	2006	NEW

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/ECD/ECD	Agilent (0825)	6890II	US10202136	2002	NEW
GC/ECD/ECD	Agilent (5253)	6890N	CN10723008	2007	NEW
GC/ECD/ECD	Agilent (0911)	6890II	US10230082	2002	NEW
GC/ECD/ECD	Agilent (5005)	6890II	CN10615005	2009	USED
GC/FID/ECD	Hewlett-Packard (Screen)	5890	GC 2415A01109	UNKNOWN	UNKNOWN
GC/FID/FID	Hewlett-Packard (3328)	5890A	333A58806	2008	USED
GC/FID/FID	Hewlett-Packard (3012)	5890II	3235A45259	1984	UNKNOWN
GC/FID/FID/TCD	Varian (CP3800)	3800	S/N 10328	2003	NEW
GC/FID/TCD	Varian (VR3600)	3600	1467	1998	UNKNOWN
GC/FPD/FPD	Hewlett-Packard (2860)	5890	2950A27078	1990	UNKNOWN
GC/FPD/FPD	Hewlett-Packard (2622)	5890II	3203A41058	1987	UNKNOWN
GC/MS	Hewlett-Packard (N)	5890II / 5971	418803507	1998	NEW
GC/MS	Hewlett Packard (V)	5890 / 5971	3549A03239	1998	NEW
GC/MS	Agilent (B)	6890 / 5973	US30965342	2003	NEW
GC/MS	Agilent (C)	6890 / 5973	US41720738	UNKNOWN	NEW
GC/MS	Agilent (G)	6890 / 5973	US43110515	UNKNOWN	USED
GC/MS	Agilent (E)	6890 / 5973	US44621242	2005	NEW
GC/MS	Agilent (F)	6890 / 5973	US52420622	2005	NEW
GC/MS	Hewlett-Packard (L)	5890II / 5971	3188A03410	1998	NEW
GC/MS	Hewlett-Packard (M)	5890II / 5971	3188A03486	1998	NEW
GC/MS	Agilent (D)	6890N / 5973	US43120962	2004	NEW
GC/MS	Hewlett-Packard (P)	5890II / 5971	3188A03495	1992	USED
GC/MS	Hewlett-Packard (Q)	5890II / 5971	3188A03498	1992	NEW
GC/MS	Hewlett-Packard (R)	5890II / 5971	3188A03506	1992	NEW
GC/MS	Hewlett-Packard (U)	5890II / 5972	3549A03238	1997	NEW
GC/MS	Agilent (H)	6890 / 5973	US+0532425	2006	NEW
GC/MS	Agilent (Z)	6890 / 5973	US02440321	2000	NEW
GC/MS	Agilent (J)	6890 / 5973	US41720746	2009	USED
GPC	ABC	1000	9137SI	UNKNOWN	UNKNOWN
GPC	J2 Scientific (I)	Autoinject 110	02D-1030-2.1	2002	NEW
GPC	J2 Scientific (H)	Autoinject 110	02D-1031-2.1	2001	NEW
GPC	J2 Scientific (J)	AccuPrep	03G1076-3.0	2003	NEW
GPC	J2 Scientific (K)	Autoinject 110	02A-102.3-2.1	2007	USED
HPLC/UV	Dionex (1488)	P680	1680407	1991	UNKNOWN
HPLC/UV/PDA	Waters (1208)	600	600-4790RP	1988	NEW
Hydrogen Generator	Parker Hannafin	H2-800	h2-800081C	2006	NEW
Hydrogen Generator	Parker Hannafin	H2-800	h2-800099C	2006	NEW
IC	Dionex (LC2723)	ICS 2000-ICAS40	4100753	2005	UNKNOWN
ICP-MS	Thermo Elemental (2)	X7	X0288	2003	NEW
ICP-OES	Thermo Electron Corp (7)	iCAP 6000	ICP20063302	2006	NEW
LC/MS/MS	Waters (1111)	Acquity/Quattro micro	QAA929	2005	NEW
LC/MS/MS	Waters (3062)	616	MX5NM6829M	UNKNOWN	NEW

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
pH Meter	Beckman	45	166928	1991	UNKNOWN
Soxtherm	Gerhardt (SOXA)	4012396	35172	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXB)	4022047	35171	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXC)	4022046	35169	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXD)	4022045	35170	UNKNOWN	UNKNOWN
TKN Digestion System	Tecator	1015	UNKNOWN	1991	UNKNOWN
TOC	Carlo Erba	EA-1108	220465	1991	UNKNOWN
TOC	Costech	4010	231009973	2005	UNKNOWN
TOC	Shimadzu	TOC-5000A	37401209A	1997	UNKNOWN
Turbidimeter	HF Scientific	Micro 100	208463	2001	UNKNOWN
UV/VIS	Genesys	Spectronic 20	35GB029021	1999	UNKNOWN

Table 20-2. Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check Peristaltic Pump tubing	As required
	Lubricate Autosampler rods	Monthly
	Clean Autosampler	Weekly
	Check and fill Rinse Vessel	As required
	Check and fill Stannous Chloride	As required
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
ICP	Check Peristaltic Pump tubing	As required
	Clean Torch	Daily
	Replace Torch	As required
	Check and fill Rinse Vessel	As required
	Check and fill IS Vessel	As required
	Fill Standards Cup	Daily
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
	Check and clean Cones	As required
	Perform Auto Peak Adjustment	As required
ICP MS	Check Peristaltic Pump tubing	As required
	Clean Torch	As required
	Check and fill Rinse Vessel	As required
	Check and fill IS Vessel	As required
	Fill standards cup	Daily
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
	Check and clean Cones	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Wavelength verification check	As required
	Clean Cuvette with Cuvette Cleaning Solution	As required
Hewlett Packard GC/MS (VOA)	Clean Injection Port and Liner	As required
	Change Septa	As required
	Cut 2-3 inches from GC Column	As required
	Fill Autosampler rinse vials	As required
	Clean Purge and Trap mount and purge vessel	As required
	Check Purge Flow	As required
Hewlett Packard GC/MS (SVOA)	Clean Injection Port and Liner	Daily
	Change Septa	Daily
	Replace or clip Guard Column	Daily
	Replace or clip Analytical Column	Daily
	Fill Autosampler rinse vials	Daily
Hewlett Packard GC/MS (Air)	Check GC / Entech Column Interface	As required
	Check Nitrogen Tank Volume	As required
	Check Nitrogen Valves Software and Valves	As required
	Cut 2-3 inches from GC Column	As required
Gas Chromatograph	Replace Septa	As required
	Clean and replace Injection Port Liner	As required
	Replace or clip Guard Column	As required
	Replace or clip Analytical Column	As required
	Bake, Re-foil, Refurbish Detector	As required

Instrument	Procedure	Frequency
Zero Air Generator	Change pre-filter cartridge	Annually
	Replace catalyst module	Indicator Light Blinks
	Check Indicator Beads in Moisture Filters	Daily
	Bake and Refill Mol Sieve Dry Rite Beads	As required
Hydrogen Generator	Fill Water Reservoir	Daily
	Replace Water in Water Reservoir	Semi-Annually
	Replace Ionic Bags in Water Reservoir	Semi-Annually
HPLC	Change Transfer Lines	As required
	Replace Guard Column	As required
	Replace Analytical Column	As required
	Replace or clean Pump Head Check Valves	As required
	Change Plunger Seals	As required
	Change Suppressor	As required
	Change Eluent Generator Cartridge and CR-ATC	As required
LC/MS/MS	Replace Guard Column	As required
	Replace Analytical Column	As required
	Replace or clean Pump Head Check Valves	As required
	Change Plunger Seals	As required
	Change In Line Filter	As required
	Clean or Change Sample Cone	As required
	Clean Source	As required
Balances	Class “1” traceable weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	Annually
Latchet	Change Tubing	As required
	Replace Bulb	As required
Conductivity Meter	Calibrate	Daily
Turbidimeter	Calibrate	As required
	Check light bulb	Daily, when used
Drying Ovens	Temperature monitoring	Daily
	Temperature adjustments	As required
Refrigerators/ Freezers	Temperature monitoring	Daily
	Temperature adjustment	As required
	Defrosting/cleaning	As required
pH/Specific Ion Meter	Calibrate	Daily
	Clean electrode	As required
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring	Daily, when used
	Water replaced	Monthly or as needed

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 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.

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 each laboratory section and in the LIMS. 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.

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.

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 within the LIMS.

- Standard ID
- Description of Standard
- Department
- 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)
- LIMS Standard ID
- 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 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 or verified time of sample receipt.

22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the laboratory SOPs 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 BR-QA-020.

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 minimum 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, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

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 the following sections.

Sample receipt procedures are described in laboratory SOP BR-SM-001.

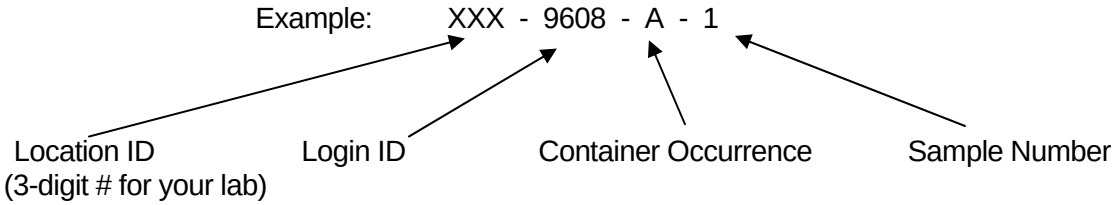
23.2.1 Laboratory Receipt

After samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample is determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

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.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Burlington Laboratory (Location XXX). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: XXX - 9608 - A - 1 - A ← Secondary Container Occurrence

Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

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 inspection and sample acceptance is verified, samples are logged into the LIMS then placed 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.

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 as per the storage conditions specified for the matrix and test method in laboratory SOPs. 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 at the frequency specified in the laboratory SOP for storage blanks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample. All unused portions of samples are returned to the secure sample control area where the samples are kept until disposal. Unless otherwise specified for each project, samples are disposed of thirty days after issuance of the data report. Special arrangements may be made to store samples for longer periods of time.

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

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only.

23.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with appropriate thermal preservation such as dry ice or sufficient wet ice to ensure the samples remain just above freezing and at or below 6.0°C during transit or samples may be shipped frozen or at ambient temperature depending on the preservation requirements of the methodology requested. 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 if a trip blank was provided with the sample set received by the client. (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: BR-EH-001. 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.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. When requested, the laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated).

Figure 23-1. Example: Chain of Custody (COC)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Burlington
30 Community Drive, Suite 11
South Burlington, VT 05403 Tel: 802 660 1990

CHAIN OF CUSTODY RECORD

Report to:
Company:
Address:
Contact:
Phone:
Fax:
Contract/Quote:

Invoice to:
Company:
Address:
Contact:
Phone:
Fax:

ANALYSIS REQUESTED

Lab Use Only
Due Date:
Temp. of coolers when received (C°):
1 | 2 | 3 | 4 | 5
Custody Seal N / Y
Intact N / Y
Screened For Radioactivity ☐

Sampler's Name
Sampler's Signature

Proj. No.
Project Name

Matrix¹	Date	Time	Comp	Grab	Identifying Marks of Sample(s)	No/Type of Containers²: VOA	A/G 1 Lt.	250 ml	P/O	Lab/Sample ID (Lab Use Only)

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Date	Time	Remarks
Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Date	Time	
Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Date	Time	Client's delivery of samples constitutes acceptance of TestAmerica terms and conditions contained in the Price Schedule.

¹Matrix WW - Wastewater W - Water S - Soil L - Liquid A - Air bag C - Charcoal Tube SL - Sludge O - Oil
²Container VOA - 40 ml vial A/G - Amber / Or Glass 1 Liter 250 ml - Glass wide mouth P/O - Plastic or other ______

TestAmerica Cannot accept verbal changes. Please Fax written changes to (802) 660-1919

TAL-8234(1007)

Figure 23-2. Example: Sample Acceptance Policy

The receipt of samples is acknowledged on the chain of custody (COC) form with the signature and date/time of the sample custodian. The condition of samples upon receipt is documented on checklists designated for this purpose. Any deficiencies identified during sample receipt are recorded and communicated to the laboratory project manager (PM), who will contact the client and fully document any decision to proceed with analysis in the project record. Consultation with the client should be immediate and timely (next business day or as specified in the project plan). Correspondence records and/or records of conversations concerning the decision to proceed with analysis and/or the disposition of rejected samples is maintained in the project record, and should be maintained in association with the sample receipt checklist. All data associated with samples that did not meet the sample acceptance criteria must be qualified with a Non-Conformance Report (NCR) and/or noted in the project narrative that accompanies the final test report.

Sample receipt is considered deficient when the following conditions are observed:

- Shipping cooler and/or samples are received outside the temperature specification
- Sample bottles are received broken or leaking
- Samples are received beyond holding time
- Samples are received without the appropriate preservation
- Samples are not received in appropriate containers
- Chain of Custody does not match the samples received
- Chain of Custody was not received or is incomplete*
- Custody seals are broken
- Evidence of tampering with the cooler and/or samples
- Headspace in 40mL or 22 mL VOA vials
- Seepage of extraneous water or other material into the samples
- Inadequate sample volume
- Illegible, impermanent ink, or non-unique sample labeling
- One or more coolers missing from a multi parcel shipment
- Shipping container is damaged

**Complete documentation shall include sample identification, the location date/time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample.*

Figure 23-3. Example: Cooler Receipt Form

SAMPLE RECEIPT & LOG IN CHECKLIST										
Client:		Date Received:				Log In Date:				
ETR:		Time Received:				By:				
SDG:		Received By:				Signature:				
Project:		# Coolers Received:				PM Signature:				
Samples Delivered By: □ Shipping Service □ Courier □ Hand □ Other (specify)						Date:				
List Air bill Number(s) or Attach a photocopy of the Air Bill:										
COOLER SCREEN						**YES**	**NO**	**NA**	**COMMENTS**	
There is ***no*** evidence to indicate tampering										
Custody seals are present and intact										
Custody seal numbers are present										
If yes, list custody seal numbers:										
Thermal Preservation Type: □ Wet Ice □ Blue Ice □ None □ Other (specify)										
IR Gun ID:		Correction Factor (CF) =		°C						
Cooler 1:	°C	Cooler 6	°C	Cooler 11	°C	Cooler 16	°C			
Cooler 2:	°C	Cooler 7	°C	Cooler 12	°C	Cooler 17	°C			
Cooler 3:	°C	Cooler 8	°C	Cooler 13	°C	Cooler 18	°C			
Cooler 4:	°C	Cooler 9	°C	Cooler 14	°C	Cooler 19	°C			
Cooler 5	°C	Cooler 10	°C	Cooler 15	°C	Cooler 20	°C			
Unless otherwise documented, the recorded temperature readings are adjusted readings to account for the CF of the IR Gun										
EPA Criteria: 0-6°C, except for air and geo samples which should be at ambient temperature and tissue samples, which may be frozen.										
Some clients require thermal preservation criteria of 2-4°C or other such criteria. The PM must notify SM when alternate criteria is specified.										
SAMPLE CONDITION						**YES**	**NO**	**NA**	**COMMENTS**	
Sample containers were received intact										
Legible sample labels are affixed to each container										
CHAIN OF CUSTODY (COC)						**YES**	**NO**	**NA**	**COMMENTS**	
COC is present and includes the following information for each container:										
▪ Sample ID / Sample Description										
▪ Date of Sample Collection										
▪ Time of Sample Collection										
▪ Identification of the Sampler										
▪ Preservation Type										
▪ Requested Tests Method(s)										
▪ Necessary Signatures										
Internal Chain of Custody (ICOC) Required										
If yes to above, ICOC Record initiated for every Worksheet										
SAMPLE INTEGRITY / USABILITY						**YES**	**NO**	**NA**	**COMMENTS**	
The sample container matches the COC										
Appropriate sample containers were received for the tests requested										
Samples were received within holding time										
Sufficient amount of sample is provided for requested analyses										
VOA vials do not have headspace or a bubble >6mm (1/4" diameter)										
Appropriate preservatives were used for the tests requested										
pH of inorganic samples checked and is within method specification										
If no, attach Inorganic Sample pH Adjustment Form										
ANOMALY / NCR SUMMARY										

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.
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 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 as needed. 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). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

- The maximum acceptable recovery limit will be 150%.

- 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. Procedures for control charts and control limits are described in laboratory SOP BR-QA-013.

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 Department 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 exceedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits. If the laboratory allows use of marginal exceedance for a test method, the specification for use will be described in the test method SOP.

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 on laboratory letterhead, 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.
- 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 (e.g., 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. For DoD work, the date and time of preparation and analysis are essential information regardless of holding time. Test reports for DoD QSM compliance must include both the data and of sample preparation and analysis.

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 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 routinely offers four levels of quality control reporting.

- Level I is a report with the features described in Section 25.2 above except QC summary information is not included.
- Level II is a Level I report plus QC summary information.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Burlington 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.

3.1.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

3.1.2 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.

3.1.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

3.1.4 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.

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 the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) 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 as is the original report. The revised report will have the word "revised" or "amended" next to the date rather than the word "reported".

When the report is re-issued, a notation of "report re-issue "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 11/3/08 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/08 at 10:47am.*

25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

25.9.1 Policy on Data Omissions or Reporting Limit Increases

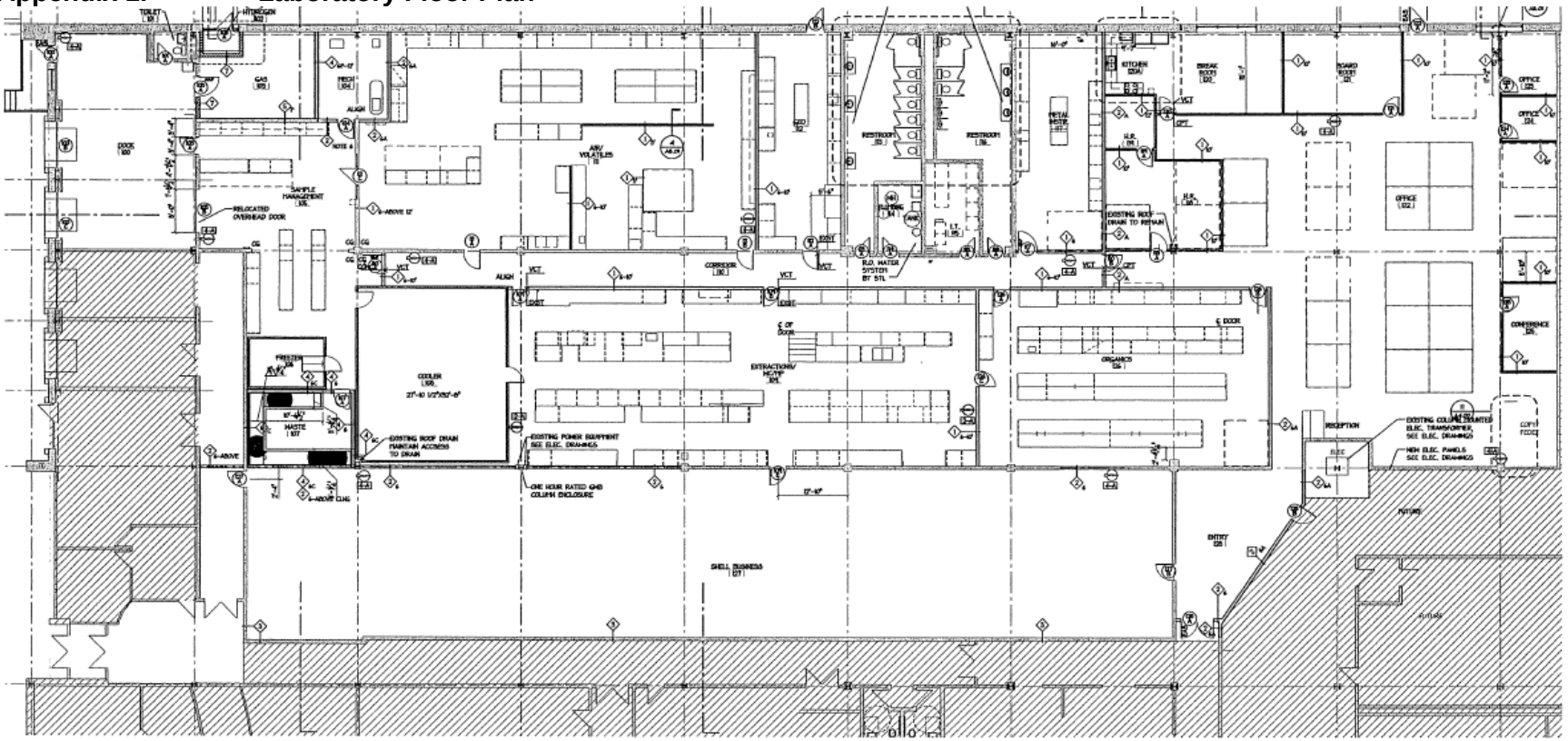
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- 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 work order 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



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.

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.

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)

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 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)

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.

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.

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 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.

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)

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)

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 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.

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.

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:

BS – Blank Spike
BSD – Blank Spike Duplicate
CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
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
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
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
PT – Performance Testing
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
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Burlington 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. Contact the laboratory for the most current information regarding certifications, accreditation and licenses held by the laboratory. As of the effective date of this document, the certifications maintained by the Burlington laboratory are:

Organization	Certificate Number
Connecticut	PH-0751
Delaware	DNREC
Florida	E87467
Maine	VT0008
Minnesota	050-999-436
New Hampshire	200606
New Jersey	VT972
New York	10391
Pennylvania	68-00489
Rhode Island	LAO00298
USDA	S-66352
Vermont	VT-4000

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 following offices: QA, marketing, and project management.

From: Madison, Jim [Jim.Madison@testamericainc.com]
Sent: Tuesday, June 29, 2010 12:53 PM
To: Balla, Tonya
Subject: RE: St. Louis and Howard's Bay
Attachments: QA and PM roles.doc

Tonya - here is info on our QA Manager and Myself as PM.

For spare parts and maintenance:

Tributyltin.
HP 7673A, HP5890 with GC/FPD Detectors
Trim column ends, and/or replace columns, Rtx-35 (30m x 0.32 mmID x 0.25um) and Rtx-5 (30m x 0.32mmID x 0.25um).
Injection Port Maintenance
Install new guard column
Replace Septa

TOC/BC
Carlo Erba Elemental Analyzer EA1108 and/or NA 1500
5mm x 9mm tin capsules
Quartz column
Check for column leaks, replace column.

Grain Size
Spare Sieves
1000ml sedimentation cylinders
Hydrometer: ASTM 151H in specification E 100.

From: Madison, Jim
Sent: Monday, June 28, 2010 3:18 PM
To: 'Balla, Tonya'
Subject: RE: St. Louis and Howard's Bay

Tonya - Here is our Quality Plan and the applicable SOPs. Note that we have separate extraction and analytical SOPs for tributyltin. I'm also sending an Excel worksheet with RL/MDL/Control Limit, container and holding time data. I'm working on blurbs for QA and PM, and instrument maintenance.

For these methods, we can provide the SEDD 2a, as well as the Region 5 EQuiS (Version 2 from August 2008).

From: Balla, Tonya [mailto:T.Balla@WestonSolutions.com]
Sent: Monday, June 28, 2010 12:23 PM
To: Madison, Jim
Subject: St. Louis and Howard's Bay

Jim,

I would like to award the St. Louis and Howard's Bay Grain Size, Tributyl tin, black carbon and the TOC samples (~42) which are co-located with the black carbon analysis to Test America Burlington. I need the following items by COB on Tuesday:

- Laboratory QAPP

- Method Specific SOPs
- Reporting limit and method detection limits (excel format)
- Accuracy and precision limits for surrogates, laboratory control sample and matrix spike (control limits and RPDs) – (excel format)
- Project Manager and QA Manager names with couple sentence blurb on responsibilities of each
- Confirmation of which level of SEDD (2a, 2b, or 3) and that EquIS EDD will be provided
- Preventative maintenance and spare parts related to instruments/method of award.
- Bottle requirements/holding times for inclusion into the project specific QAPPs

The laboratory should make efforts to meet the 21 day TAT for grain size but that parameter (only) will be allowed to extend to 30 days due to labs overall throughput capacity and the number of projected samples for these projects.

Hopefully that is it. Please let me know if there are any questions/concerns.

Thanks,
Tonya

Tonya Balla
Senior Project Manager
847.918.4094 (o) 847.528.2623 (c)

Weston Solutions Inc.
750 E. Bunker Ct. Suite 500 Vernon Hills, IL 60061
t.balla@westonsolutions.com www.westonsolutions.com

From: Craig Widby [widbyc@gmail.com]
Sent: Tuesday, June 29, 2010 4:54 PM
To: Balla, Tonya
Cc: harveylm@trimatrixlabs.com
Subject: RE: St. Louis and Howard's Bay (TriMatrix Laboratories - DRO/ORO analysis)
Attachments: TriMatrix 2010 QAM.PDF; GR-03-122_REV 2.3(04-16-10)_DRO_8015B.PDF; GR-09-123_REV 0.2(04-16-10)_EXTRACTION SOIL DRO_3550B 8015C.PDF; Weston Solutions_St Louis and Howards Bay STD BID 28 Jun 10 1538_TriMatrix Laboratories.xls; DRO ROUTINE PREVENTIVE MAINTENANCE SPARE PARTS_TriMatrix Laboratories.pdf

Hi Tonya:

Good afternoon.

Attached and below is the information you have requested from TriMatrix in reference to the St. Louis and Howard's Bay DRO/ORO analyses. Upon review, please let me know if you have any questions or need anything further. I am here to help.

- Laboratory QAPP (attached PDF)
- Method Specific SOPs (attached PDF)
- Reporting limit and method detection limits (attached Excel format)
- Accuracy and precision limits for surrogates, laboratory control sample and matrix spike (control limits and RPDs) – (attached Excel format)
- Project Manager and QA Manager names with couple sentence blurb on responsibilities of each

  Rick Wilburn – Quality Assurance Manager

  Mr. Wilburn is responsible for all aspects of the laboratory Quality Control/Quality Assurance (QA/QC) Program. Primary responsibilities include conducting internal and external auditing of the laboratory, procurement and maintenance of state and federal certifications, and ensuring that all facets of the quality control program remain at the highest level possible. Mr. Wilburn also manages the external and internal Quality Control check sample programs.

  Mr. Wilburn directed TriMatrix's efforts in successfully becoming a charter member in the first round of the National Environmental Laboratory Accreditation Program (NELAP). He has a working knowledge of several major corporate environmental laboratory programs, including programs with Ford Motor Company, General Motors Corporation, and Honeywell International. Mr. Wilburn is experienced in working closely with laboratory personnel to ensure that QA/QC requirements are identified and followed for specific corporate programs.

  Lisa Harvey – Laboratory Senior Project Chemist

  Ms. Harvey's responsibilities include all aspects of laboratory project management. Therefore, a Project Chemist is responsible for various laboratory client services including client contact, project management, problem solving, invoicing, marketing, report generation and meeting mandated agency report deadlines and client reporting deadlines.

  Ms. Harvey has vast experience with various matrix types and project requirements. Her client list includes Federal, State and Local Governmental agencies, automotive industrial clients, manufacturing industries, and a large list of environmental consulting and engineering firms. Types of projects she currently is responsible for are Soil Remediation and Clean-up projects, Wastewater/NPDES reporting, Groundwater Monitoring for Landfills, former industrial sites, and current manufacturing sites. Many of her projects require data validation for EPA Region 5.
- Confirmation of which level of SEDD (2a, 2b, or 3) and that EQuIS EDD will be provided

  TriMatrix will provide the SEDD 2b and the Weston project specific EQuIS format for all analyses performed at our laboratory. We have requested the status of the SEDD 3 EDD from our LIMS vendor and will provide this format if available prior to the project start date.
- Preventative maintenance and spare parts related to instruments/method of award (attached PDF)
- Bottle requirements/holding times for inclusion into the project specific QAPPs

  Sample Container/Preservation – SW846 8015 DRO/ORO

  Sample Containers - 125mL or 250mL WM Clear Glass
  Preservation - Cool to 4° C

Analysis Hold time(s) - 14-days (Extraction); 40-days (Analysis)

Again, thank you for the opportunity to partner with Weston on these projects. We look forward to working with you.

Best regards,

Craig Widby
Director, Corporate Programs

TRIMATRIX LABORATORIES

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Direct: 773.271.1218
Lab: 616.975.4500
Fax: 616.942.7463
www.trimatrixlabs.com

From: Balla, Tonya [mailto:T.Balla@WestonSolutions.com]
Sent: Monday, June 28, 2010 11:17 AM
To: craig.widby@trimatrixlabs.com
Subject: St. Louis and Howard's Bay

Craig,

I would like to award the St. Louis and Howard's Bay **DRO/ORO** analysis to TriMatrix. I need the following items by COB on Tuesday:

- Laboratory QAPP
- Method Specific SOPs
- Reporting limit and method detection limits (excel format)
- Accuracy and precision limits for surrogates, laboratory control sample and matrix spike (control limits and RPDs) – (excel format)
- Project Manager and QA Manager names with couple sentence blurb on responsibilities of each
- Confirmation of which level of SEDD (2a, 2b, or 3) and that EquIS EDD will be provided
- Preventative maintenance and spare parts related to instruments/method of award.
- Bottle requirements/holding times for inclusion into the project specific QAPPs

Hopefully that is it. Please let me know if there are any questions/concerns.

Thanks,
Tonya

Tonya Balla
Senior Project Manager
847.918.4094 (o) 847.528.2623 (c)

Weston Solutions Inc.
750 E. Bunker Ct. Suite 500 Vernon Hills, IL 60061
t.balla@westonsolutions.com www.westonsolutions.com

Columbia Analytical Services

Project Manager - Howard Holmes

Responsibilities:

Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies.

QA Manager - Julie Gish

Responsibilities:

Responsible for the overall implementation of the laboratory QA program. Responsible for the Quality Assurance Manual, certifications, documenting SOPs, and maintaining proficiency testing (PT) records. Maintains certifications/accreditations for regulatory agencies and client certifications or approval programs. Conducts internal audits and make recommendations for corrective action.

EDD's – SEDD 2a & EPA Region 5 EquIS EDD

Preventative Maintenance & Spare Parts for TOC analysis

This information is discussed in the SOP. In addition, CAS stocks a spare thermocouple and lamp. CAS has arranged a loaner instrument from the manufacturer in case of a catastrophic failure.

Bottle Requirements/Preservatives/Holding Times

WM Glass jar with Teflon lined cap, typically 4oz / cool to 4C / 28 days

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

1317 South 13th Avenue
Kelso, Washington 98626
(360) 577-7222

Effective Date: October 30, 2009

Laboratory Director/Technical Director: _____
Jeff Christian

Quality Assurance Manager: _____
Julie Gish

Technical Director – Metals: _____
Jeff Coronado

Technical Director – Metals R & D: _____
Nicholas Bloom

Technical Director – Organics: _____
Jeff Grindstaff

Technical Director – Organic Extractions: _____
Dr. Gregory Salata

Technical Director – Inorganics/Microbiology: _____
Harvey Jacky

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

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3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is an employee-owned professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, and other material.

Quality Management Systems are established, implemented and maintained by management. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory in involved.

This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities. Policies and procedures are established in order to meet the quality objectives of clients, accrediting authorities, and certifying organizations. Columbia Analytical Services, Inc. is committed to operate in accordance to: ISO/IEC 17025:2005 International Standards, The NELAC Institute (TNI) National Environmental Laboratory Accreditation Program (NELAP), and DoD Environmental Laboratory Accreditation Program. Quality Systems are established to meet the requirements of these standards.

Laboratory management is committed to continually improve the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set fourth in the *Columbia Analytical Services, Inc. Quality and Ethics Policy Statement March* 2009 and in this *Kelso Quality Assurance Manual* (QAM). We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

Columbia Analytical maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

Columbia Analytical is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, Columbia Analytical also operates laboratories in California, Florida, New York, Arizona, and Texas.

The information in this document has been organized according to the format described in *EPA Requirements for Quality Management Plans, EPA QA/R-2,* USEPA, 2001; *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* USEPA, 2001, and *ISO17025 International Standard.*

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at Columbia Analytical is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of Columbia Analytical:

> "The mission of Columbia Analytical Services, Inc. is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

4.1 Quality Management Systems

In support of this mission, the Kelso laboratory has developed Quality Management Systems to ensure all products and services meet our client's needs. These systems incorporate the requirements of ISO17025 standards. Quality Management Systems Include:

- Standard Operating Procedures
- Sample Management
- Chain of Custody Procedures
- Statistical Control Charting
- Standards Traceability
- Core Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the Quality Management System is assessed in several ways:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Complaints
- Internal and External Proficiency Testing

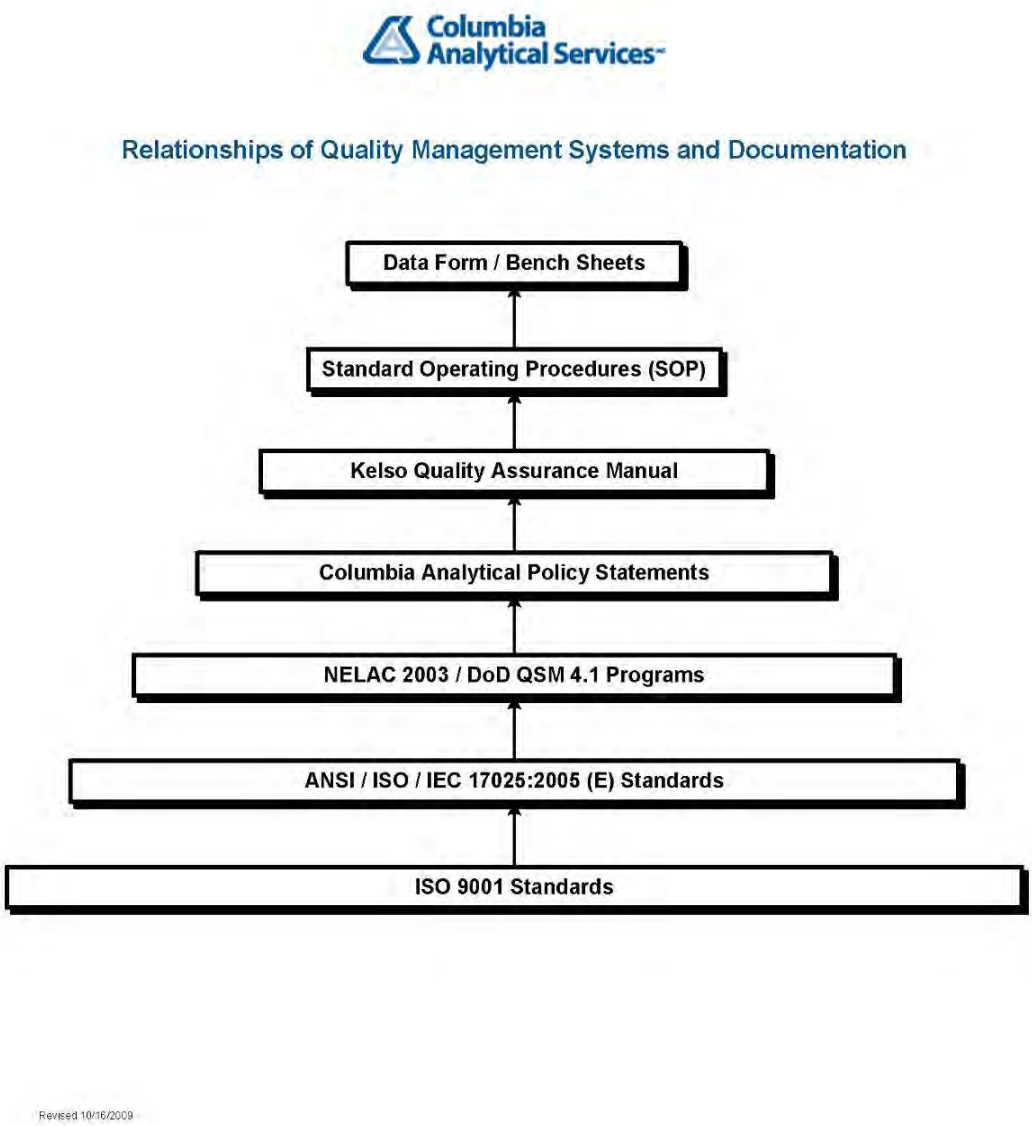


Figure 4-1

Kelso Quality Management Systems are based upon ISO 17025:2005 standards. Fundamental programs (NELAC 2003 and DoD QSM) are based upon these standards. Implementation and documentation against these standards are communicated in corporate policy statements, and Kelso's Quality Assurance Manual. Actual procedures, actions and documentation are defined in both administrative and technical SOP's.

4.2 Facilities and Equipment

Columbia Analytical features over 45,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, Columbia Analytical minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals "clean room" sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Metals R&D Laboratory
- Water Chemistry & General Chemistry Laboratories (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography/High Performance Liquid Chromatography Laboratories
- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Semi-volatile Organics Drinking Water Laboratories (2)
- Volatile Organics Laboratory
    - Separate sample preparation laboratory
    - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water Systems (2)
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.3 Technical Elements of the Quality Assurance Program

The laboratory's technical procedures are based upon procedures published by various agencies or organizations (See Section 18). The Quality Assurance Program provides to the laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained by the QA department. This includes method approvals and audit administration. In addition,

internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventative maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.4 Operational Assessments

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients including; On-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

Columbia Analytical utilizes a number of different methods to ensure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results. All Requests for Proposal (RFP) documents are reviewed by the Project Chemist and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Plans (QAPPs) if available. A weekly status meeting is also conducted with the laboratory staff by the Client Services Manager to inform the staff of the status of incoming work, future projects, or project requirements.

4.5 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, certificates of analysis, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled Columbia Analytical documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

Columbia Analytical maintains a records system that ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH).*

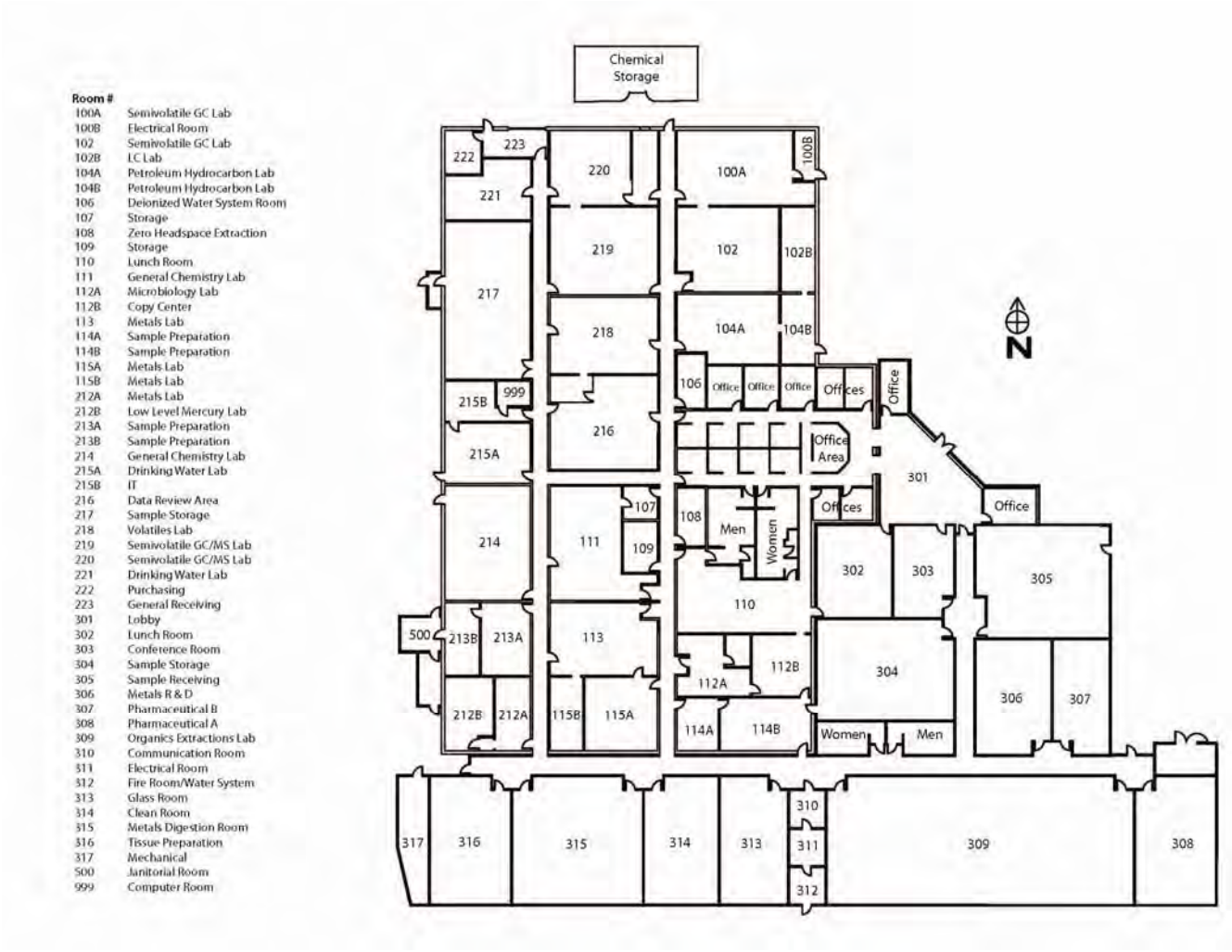
4.6 Subcontracting

Analytical services are subcontracted when Columbia Analytical/Kelso needs to balance workload or when the requested analyses are not performed by Columbia Analytical/Kelso. Subcontracting is only done with the knowledge and approval of the client and to qualified laboratories. Subcontracting to another Columbia Analytical laboratory is preferred over external-laboratory subcontracting. Further, sub-contracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories (ADM-SUBLAB).* The Corporate Quality Assurance staff is responsible for qualifying and oversight of subcontract laboratories.

4.7 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned (by the laboratory user) as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Purchasing through CAS Purchasing Department in Kelso (SOP ADM-PUR).* Also, refer to section 10.4 for a discussion of reference materials.

Figure 4-2
Columbia Analytical/Kelso Laboratory Floor Plan



5.0 PROFESSIONAL CONDUCT AND ETHICAL PRACTICES

One of the most important aspects of the success of Columbia Analytical is the emphasis placed on the integrity of the data provided and services performed. To promote product quality, employees are required to comply with certain standards of conduct and ethical practices. The following examples of Columbia Analytical policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action. Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible. Employee discipline is progressive in its severity and each situation is handled individually in that the discipline is designed to fit the circumstances. Potential disciplinary actions may include a verbal warning, written warning, a second written notice (more severe and more strongly worded than a warning), suspension without pay, demotion, or termination.
- It is the responsibility of all Columbia Analytical employees to safeguard sensitive company and client information. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action.

All employees are required to sign and adhere to the requirements set forth in the Columbia Analytical *Confidentiality and Conflicts of Interest Employee Agreement* and the Columbia Analytical *Commitment to Excellence in Data Quality Policy.* All employees receive in-house ethics training and are periodically reminded of their data quality and ethical conduct responsibilities.

Columbia Analytical makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the Columbia Analytical Employee Handbook. This includes the Columbia Analytical Ombudsman Program, the Columbia Analytical Open Door Policy, and the use of flexible work hours. Operational assessments are regularly made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads (Section 4.3). Procedures for subcontracting work are established, and within the Columbia Analytical laboratory network additional capacity is typically available for subcontracting, if necessary.

6.0 ORGANIZATION AND RESPONSIBILITIES

The Columbia Analytical/Kelso staff, consisting of approximately 130 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

Columbia Analytical is committed to providing an environment that encourages excellence. Everyone within Columbia Analytical shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 6-1 lists the Columbia Analytical/Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The responsibility of the **Quality Assurance Manager** (QAM) is to oversee implementation of the quality program and to coordinate QA activities within the laboratory. The QAM works with laboratory production units to establish effective quality control and assessment plans. The QAM has the authority to stop work in response to quality problems. The QAM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and coordinating the annual review of each SOP; maintaining QA records such as metrological records, archived logbooks, PT sample results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; performing internal QA audits; preparing QA activity reports; etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the Columbia Analytical Quality Assurance Director. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

  The Chief Quality Officer (CQO) is responsible for the overall QA program at all the Columbia Analytical laboratories. The CQO is responsible for ensuring that annual internal audits are performed at each Columbia Analytical laboratory; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of Columbia Analytical-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Organics Department Manager (for the Laboratory Director) and the CQO or Laboratory Director (for the QA Manager).

- The **Environmental Health and Safety Officer** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to Columbia Analytical's EH&S Director.

- The **Client Services and Sample Management Office Manager** is responsible for the Client Services Department (customer services/project chemists, and Electronic Data Deliverables group) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.

- The **Project Chemist** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the Columbia Analytical laboratory and administrative staff to ensure that client-specific needs are understood, and that the services Columbia Analytical provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within the department. Each **Department Manager and Supervisor** has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.

- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.

- **Information Technology** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 6-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	30	Laboratory Director
Julie Gish, M.S.	18	Quality Assurance Manager
Lynda Huckestein, B.S.	20	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	19	Metals Department Manager
Nicolas Bloom, M. S.	29	Metals R & D Manager
Harvey Jacky, B.S.	20	General Chemistry Department Manager
Gregory Salata, Ph.D.	9	Extractions Department Manager
Jeff Grindstaff, B.S.	20	Organics Chromatography & Mass Spectrometry Department Manager
Loren Portwood, B.S.	18	Organics Drinking Water Department Manager
Eileen Arnold, B.A.	27	Environmental Health and Safety Officer
Mike Sullivan, B.S.	8	Information Technology Director
Lee Wolf, B.S.	23	Chief Quality Officer
Steve Vincent, B.S.	33	President

7.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. Columbia Analytical management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major automated data processing equipment.

7.1 Software Quality Assurance Plan

Columbia Analytical has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the *CAS Software Quality Assurance Plan (SQAP)*. The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of this plan are policies for software validation and control.

7.2 IT Support

The local Columbia Analytical Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, Columbia Analytical corporate IT provides support for network-wide systems. Columbia Analytical also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

7.3 Information Management Systems

Columbia Analytical has various systems in place to address specific data management needs. The Columbia Analytical Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request. Included on the service request is a summary of client information, sample identification, required analyses, work instructions, deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats, including Staged Electronic Data Deliverable (SEDD) format.

7.4 Backup and Security

Columbia Analytical laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non- Columbia Analytical access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. Columbia Analytical uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. Email attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems.

8.0 SAMPLE MANAGEMENT

8.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. Columbia Analytical recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

Columbia Analytical uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 18 for complete citations). The container, preservation and holding time information for these references is summarized in Table 8-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

Columbia Analytical routinely provides sample containers with appropriate preservatives for our clients. Containers are purchased as precleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container.

Figure 8-1 shows the chain-of-custody form routinely used at Columbia Analytical and included with sample kits. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to Columbia Analytical. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. Columbia Analytical keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. Columbia Analytical also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When Columbia Analytical ships environmental samples to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures (SMO-GEN) for maintaining the samples' chain of custody, packaging and shipment. Dry ice gel ice is the only temperature preservative used by Columbia Analytical, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures (SMO-GEN) are established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received.

Once samples are delivered to the Columbia Analytical sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 8-2 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);

- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Chemist and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Chemist for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. Columbia Analytical has five walk-in cold storage units which house the majority of sample containers received at the laboratory. In addition, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. Columbia Analytical also has seven sub-zero freezers capable of storing samples at -20° C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

Columbia Analytical adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is printed or written on the analytical raw data. For analyses with a holding time prescribed in hours it is essential that the sample collection time is provided, so holding time compliance can be demonstrated. If not, the sample collection time is assumed as the earliest in the day (i.e. the most conservative).

Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature for 30 days, soil samples are retained at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Environmental Health and Safety Manual.* All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

8.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN)*. Figure 8-1 is a copy of the chain-of-custody form routinely used at Columbia Analytical.

Facility security and access is important in maintaining the integrity of samples received at Columbia Analytical/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The Columbia Analytical facility is equipped with an alarm system and Columbia Analytical employs a private security firm to provide nighttime and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC).*

8.4 Project Setup

The analytical method(s) used for sample analysis are chosen based on the client's requirements. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Chemist will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Chemist ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the service request. To communicate and specify project-specific requirements, a Tier V form (Figure 8-3) is used and accompanies the service request form.

Table 8-1
Sample Preservation and Holding Times

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert (Standard Methods)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total (Standard Methods)	W, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci (SM 9230B)	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia (SM 4500NH3)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Chlorine, Total Residual (SM 4500Cl F)	W, DW	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500CN E,G)	W, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable (SM 4500CN I)	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron (CAS SOP)	W, DW	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0)	W, DW	P,G	None Required	28 days
Fluoride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Hardness (SM 2340C)	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrite (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Orthophosphate (EPA 365.3)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Perchlorate (EPA 314.0)	W, DW	P,G	Protect from temp. extremes	28 days
Phenolics, Total (EPA 420.1)	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total (EPA 160.3 & SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS) (SM 2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500SiO2 C)	W	P Only	Cool, 4°C	28 days
Specific Conductance (EPA 120.1 & SM 2510B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Sulfide (SM 4500S2 F)	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite (SM 4500SO3 B)	W	P,G	None Required	24 hours
Surfactants (MBAS) (SM 5540C)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Metals				
Metals, except CrVI and Mercury	W, DW	P,G	HNO_3 to pH<2	6 months
(EPA 200.7, 200.8, 200.9, 6010, 6020)	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
(EPA 245.1, 7470, 7471)	S	P,G	Cool, 4°C	28 days
1631E	W	F	Cool, 4°C, HCl or H_2SO_4 to pH<2	90 days
1631E	S	F	Freeze < -15ºC	1 Yr
Methyl Mercury 1630	W	F	HCL to pH<2	6 months
Organic Tests				
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (EPA 415.1, 9060 & SM 5310C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (EPA 9020)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total (EPA 8015)	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction
Pharma Personal Care Products 1694	W	Amber G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	14 days until extraction; 40 days after extraction
Nitroaromatics and Nitramines 8330, 8330B	W,S	G, Teflon-Lined Cap	Cool, 4°C	S 14, W 7 days until extraction; 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Organic Test				
Methanol in Process Liquid NCASI 94.03	L	G, Teflon-Lined Cap	Cool, 4°C	30 days
HAPS – Condensates NCASI 99.01		G, Teflon-Lined Cap	Cool, 4°C	14/30 days
HAPS – Impinger/Canisters NCASI 99.02			Cool, 4°C	21 days
Perfluorinated Compounds HPLC/MS/MS	W	P	Cool, 4°C	14 days until extraction; 40 days after extraction
PBDE/PBB – ROHS GC/MS			RT	40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics) (EPA 8015)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days
Purgeable Halocarbons (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE) (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile (EPA 624, 8260)	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
EDB and DBCP (EPA 8260)	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Alcohols and Glycols (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270, 8310)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides (EPA 8141, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides (EPA 8151)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organotins (CAS SOP)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics (EPA 1653A)	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Resin and Fatty Acids (NCASI 85.02)	W	G, Teflon-Lined Cap	NaOH to pH $\geq$10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined Septum Cap	Ascorbic Acid, HCl to pH≤2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	DW	G, Teflon-Lined Septum Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Carbamates, Carbamoyloximes (EPA 531.1)	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH<3; 80 mg/L $Na_2S_2O_3$ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/4omL NaS; Cool, <6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$ if Res.Cl., Cool, 4°C,	7days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate (EPA 547)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$, Cool, 4°C	14 days
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Nitrosoamines (EPA 521)	DW	G, Amber, Teflon-Lined Cap	Dechlorinate at collection[g] Cool, 4°C	14 days until extraction; 28 days after extraction
Selected Pesticides and Flame Retardants (EPA 527)	DW	G, Amber, Teflon-Lined Cap	See method Cool, 4°C	14 days until extraction; 28 days after extraction
Explosives (EPA 529)	DW	G, Amber, Teflon-Lined Cap	See method Cool, 4°C	14 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Mercury (EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics (EPA 1311/8260)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction

a For EPA SW-846 methods the method number is listed generically, without specific revision suffixes.
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass, F- Fluoropolymer
d For chlorinated water samples
e The maximum holding time is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

Figure 8-1
Chain of Custody Form

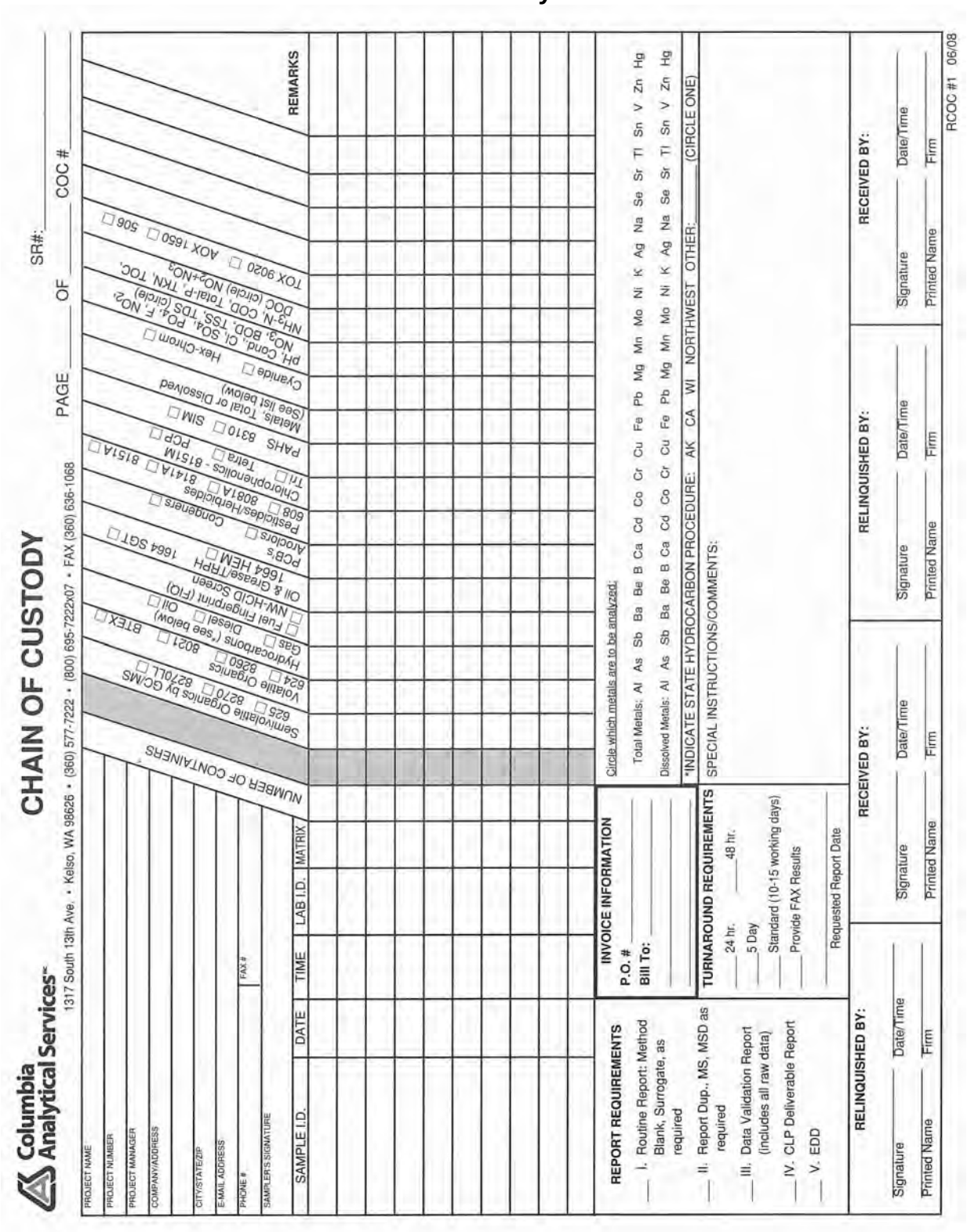


Figure 8-2

Columbia Analytical Services, Inc. PC_____
Cooler Receipt and Preservation Form

Client / Project:_____ Service Request *K09*_____

Received:_____ Opened:_____ By:_____

1. Samples were received via? ***US Mail*** ***Fed Ex*** ***UPS*** ***DHL*** ***GH*** ***GS*** ***PDX*** ***Courier*** ***Hand Delivered***
2. Samples were received in: (circle) ***Cooler*** ***Box*** ***Envelope*** ***Other***_____ ***NA***
3. Were custody seals on coolers? NA Y N If yes, how many and where?_____
   If present, were custody seals intact? Y N If present, were they signed and dated? Y N
4. Is shipper's air-bill filed? If not, record air-bill number:_____ NA Y N

5. **Temperature of cooler(s) upon receipt (°C):** _____ _____ _____ _____
   Temperature Blank (°C): _____ _____ _____ _____
   Thermometer ID: _____ _____ _____ _____
6. If applicable, list Chain of Custody Numbers: _____ _____ _____ _____
7. Packing material used. ***Inserts*** ***Baggies*** ***Bubble Wrap*** ***Gel Packs*** ***Wet Ice*** ***Sleeves*** ***Other***_____
8. Were custody papers properly filled out (ink, signed, etc.)? NA Y N
9. **Did all bottles arrive in good condition (unbroken)?** *Indicate in the table below.* NA Y N
10. Were all sample labels complete (i.e analysis, preservation, etc.)? NA Y N
11. Did all sample labels and tags agree with custody papers? *Indicate in the table below* NA Y N
12. **Were appropriate bottles/containers and volumes received for the tests indicated?** NA Y N
13. Were the pH-preserved bottles tested* received at the appropriate pH? *Indicate in the table below* NA Y N
14. Were VOA vials and 1631 Mercury bottles received without headspace? *Indicate in the table below.* NA Y N
15. **Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?** NA Y N
16. Was C12/Res negative? NA Y N

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume added	Reagent Lot Number	Initials

**Does not include all pH preserved sample aliquots received. See sample receiving SOP (SMO-GEN).*

Additional Notes, Discrepancies, & Resolutions:_____

Figure 8-3
Tier V Form

Client :

Project Chemist :

Project Name :

Service Request :

Project Number :

SMO LimsTemplate ID :

Project Description :

QAPP/SOW Information :

Reporting

Tier Level :

PDF:

Report to :

In result field use :

EDD :

Flagging Requirements :

Other Requirements :

Sample Considerations

Sample Limitations :

Sample Prep/Analysis :

Non-Standard Holdtimes :

Historical Data :

Comments :

9.0 ANALYTICAL PROCEDURES

Columbia Analytical employs methods and analytical procedures from a variety of external sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 18.0. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by Columbia Analytical is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix E. Further details are described below.

9.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

Columbia Analytical maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

9.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

9.3 Modified Procedures

Columbia Analytical strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

9.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that Columbia Analytical has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.
2) All (field) samples in a batch are of the same matrix.
3) The QC samples to be processed with the (field) samples include:
   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b) Laboratory Control Sample

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.
4) A single lot of reagents is used to process the batch of samples.
5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.

7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

9.5 Specialized Procedures

Columbia Analytical not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are trace-level Mercury and Methylmercury analyses, reductive precipitation metals analysis, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs).

9.6 Sample Cleanup

Columbia Analytical commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods (3620, 3630, 3640, 3660, and 3665) for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

10.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at Columbia Analytical are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files.

Records are maintained to provide traceability of reference materials.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standards. All analytical measurements generated at Columbia Analytical are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type. This information is provided to the client when requested.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective; is taken out of service until it is repaired. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

10.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators. Bound record books are kept which contain daily-recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC).* The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standard on an annual basis.

10.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC. The weights are recertified using NIST traceable standards by an accredited metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by an accredited metrology organization.

10.3 Water Purification Systems

Columbia Analytical uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Following a written SOP, the status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily in a bound record book. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked on a daily basis at a point downstream of the purification system at a tap in the laboratory.

10.4 Source and Preparation of Standard Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. Columbia Analytical relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

10.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

10.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

10.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be “dropped” from the resulting calibration curve.

10.8 GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working

range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.10 LC/MS Systems

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.11 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be “dropped” from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at Columbia Analytical include total phenolics, phosphates, surfactants and tannin-lignin.

10.12 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be “dropped” from the resulting calibration curve. Standard Columbia Analytical acceptance limits are used to evaluate the calibration curve prior to sample analysis.

10.13 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be “dropped” from the resulting calibration curve. A correlation coefficient of $\geq$ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and drinking water inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

10.14 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

10.15 Ion-selective electrode

The method-prescribed numbers of standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

10.16 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP Checking Volumetric Labware (ADM-VOLWARE)*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

10.17 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

11.0 QUALITY CONTROL

A primary focus of Columbia Analytical's Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. Columbia Analytical has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

11.1 Quality Control Objectives

11.1.2 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria is met.

11.1.3 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination). Columbia Analytical utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

11.1.4 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

11.1.5 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the Quality Assurance Manager. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

11.1.6 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. Columbia Analytical has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample.

These include the *SOP for Subsampling and Compositing of Samples* and the *SOP for Tissue Sample Preparation*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

11.1.7 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using Columbia Analytical or project-specified data qualifiers.

11.2 Method Detection Limits and Method Reporting Limits

Method Detection Limits (MDL) for methods performed at Columbia Analytical/Kelso is determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation (ADM-MDL),* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation- LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

11.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

11.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

11.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

11.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

11.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

11.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in the calibration. Once it is determined that there is no reference material defect or systematic error in preparation of the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

11.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

11.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

11.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

11.3.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in

the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

11.3.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$Relative\ Percent\ Difference\ (RPD) = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

11.3.13 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

11.3.14 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

11.3.15 Control Charting

The generation of control charts is routinely performed at Columbia Analytical. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

11.3.16 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at Columbia Analytical undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at Columbia Analytical; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

Columbia Analytical reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the *SOP for Laboratory Data Review Process.*

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the “before” and “after” integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (SOP ADM-INT).

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.
    2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
    2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS Methods - Two criteria are used to verify identification:
    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.
- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures as described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are

performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including alternative analysis.

- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP *Confirmation Procedure for GC and HPLC Analysis (SOC-CONF).* If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including additional cleanup.
- Surrogate Results (Organic) – Following sample analysis and data reduction, the percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.
- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 are used to determine if the LCS is effective in validating the analytical system and the associated samples.

- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. Columbia Analytical maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Reporting and Report Generation* addresses the flagging and qualification of data. The Columbia Analytical-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the project chemist to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Chemist verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Chemist accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the Columbia Analytical client.

12.5 Documentation

Columbia Analytical maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the *SOP for Data Archiving*.

12.5.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

12.6 Deliverables

In order to meet individual project needs, Columbia Analytical provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) to following specialized deliverables:

- ADEC – Alaska Department of Conservation specified data package
- ACOE/HTRW – Army Corps of Engineers specified data package and reporting requirements (HTRW, CERP, FUDS, etc.)
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, Columbia Analytical provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. Columbia Analytical is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 12-1
Descriptions of Columbia Analytical Standard Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results and acceptance criteria for applicable organic methods
5. Chain of custody documents
6. Dates of sample preparation and analysis for all tests

Tier II and IIA. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Tier IIA also includes Laboratory Control Sample (LCS) result(s) with calculated recovery and including associated acceptance criteria

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or Quality Control check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Summary forms for associated QC and calibration parameters
6. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Results and spectra of TIC compounds will be included upon request.

Tier IV. CLP-Level Data Validation Package.

A complete Data Validation Package containing all sample results, quality control and calibration results, and raw data necessary to fulfill all deliverable requirements of an EPA Contract Laboratory Program (CLP) data package.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of Columbia Analytical/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of Columbia Analytical/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which Columbia Analytical/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of Columbia Analytical/Kelso are conducted regularly under the direction of the Quality Assurance Manager. The internal audit procedures are described in the *SOP for Internal Audits*. The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that systems, technical operations, hardcopy data, and electronic data are assessed.
- Hardcopy report audits – minimum of 3 per quarter.
- Electronic audit trail reviews – each applicable instrument per quarter.

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

13.2 Performance Audits

Columbia Analytical/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. Columbia Analytical routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the Columbia Analytical Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformity (NCAR) procedures.

Table 13-1
Current Columbia Analytical Performance and System Audit Programs

Federal and National Programs

- The TNI (The NELAC Institute) National Environmental Laboratory Accreditation Program (NELAP) Accredited Drinking Water, Non-Potable Water, Solid & Hazardous Waste, and Biological Tissue Laboratory
- ANSI-ASQ National Accreditation Board/ACLASS ISO 17025:2005
- DoD- ELAP Environmental Laboratory Accreditation Program
- Naval Facilities Engineering Service Center
  Validated Laboratory for NFESC Parameters
- U.S. Army Corps of Engineers
  Approved Laboratory for USACE Projects
- U.S. EPA Region 8
  Approved Drinking Water Laboratory

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory, Lab I.D. UST040
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of Arkansas, Department of Environmental Quality
  Certified Environmental Laboratory, Lab I.D. 88-0637
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Colorado, Department of Public Health and Environment
  Certified Drinking Water Laboratory
- State of Florida, Department of Health
  Primary NELAP Accreditation No. E87412
- State of Georgia, Department of Natural Resources
  Certified Drinking Water Laboratory
- State of Hawaii, Department of Health
  Certified Drinking Water Laboratory
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Indiana, Department of Health
  Certified Drinking Water Laboratory, Lab I.D. C-WA-01
- State of Louisiana, Department of Environmental Quality
  Accredited Environmental Laboratory, Lab I.D. 3016
- State of Louisiana, Department of Health and Hospitals
  Accredited Drinking Water Laboratory, Lab I.D. LA080001
- State of Maine, Department of Human Services
  Certified Environmental Laboratory, Lab I.D. WA0035
- State of Michigan, Department of Environmental Quality
  Certified Drinking Water Laboratory, Lab I.D. 9949

Table 13-1 (continued)

State and Local Programs (continued)

- State of Minnesota, Department of Health
  Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nevada, Division of Environmental Protection
  Certified Drinking Water Laboratory, Lab I.D. WA35
- State of New Jersey, Department of Environmental Protection
  Accredited Environmental Laboratory, Lab I.D. WA005
- State of New Mexico, Environment Department
  Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
  Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
  Accredited Environmental Laboratory, Lab I.D. WA200001
- State of South Carolina, Department of Health and Environmental Control
  Certified Environmental Laboratory, Lab I.D. 61002
- State of Utah, Department of Health, Division of Laboratory Services
  Accredited Environmental Laboratory
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
  Accreditation No. C1203
- State of Wisconsin, Department of Natural Resources
  Accredited Environmental Laboratory, Lab I.D. 998386840

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at Columbia Analytical (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at Columbia Analytical contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at Columbia Analytical before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at Columbia Analytical. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D.

This inventory or “parts list” also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix D for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form. The laboratorys procedure and responsibilities for addressing nonconforming work is defined in the SOP ADM-CA *Corrective Action.*

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives outlined in Section 11, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist is notified in order to ascertain if the client needs to be notified.

In the event that analyses produce nonconformances with data or results, the problem and the corresponding corrective actions taken are documented on a *Nonconformity and Corrective Action Report* (See Figure 15-1) following the requirements in the *SOP for Corrective Action* (SOP No. ADM-CA). This form is utilized to determine the root cause of the nonconformity and to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the project chemist) is responsible for documenting the complaint. If the project chemist is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (ADM-FDBK).

Figure 15-1

Nonconformity and Corrective Action Report

NCAR No: *Assigned by QA*

PROCEDURE (SOP or METHOD): EVENT DATE: _____

EVENT:
- ☐ Missed Holding Time
- ☐ Method Blank Contamination
- ☐ Equipment Failure
- ☐ SOP Deviation
- ☐ QC Failure
- ☐ Login Error
- ☐ Unacceptable PT Sample Result
- ☐ Other (describe):
- ☐ Lab Error (spilled sample, spiking error, etc.)
- ☐ Project Management Error

INCLUDE NUMBER OF SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED

DETAILED DESCRIPTION

ORIGINATOR: _____ DATE: _____

PROJECT MANAGER(S): _____ NOTIFIED BY: _____ DATE: _____

ROOT CAUSE OF NON-CONFORMITY (POTENTIAL CAUSES COULD BE TRAINING, COMMUNICATION, SPECIFICATIONS, EQUIPMENT, KNOWLEDGE)

What is the cause of the error or finding:

CORRECTIVE ACTION AND OUTCOME

Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent its reoccurrence. Include Project Manager Instructions here.

Is the data to be flagged in the Analytical Report with an appropriate qualifier? ☐ **No** ☐ **Yes**

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____ Date: _____
Comments:

QA PM Verification and Approval of Corrective Action _____ Date: _____
Comments:

Project Manager Verification and Approval of Corrective Action _____ Date: _____
Comments:

Customer Notified by ☐ Telephone ☐ Fax ☐ E-mail ☐ Narrative ☐ Not notified

(Attach record or cite reference where record is located.)

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by Columbia Analytical personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists as required (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QAM provides the Laboratory Director with quarterly reports that summarize the various QA/QC activities that occurred during the previous quarter. The report addresses such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

The Laboratory Director also performs an annual management review of the quality and management systems to identify any necessary changes or improvements to the quality system or quality assurance policies. This review is documented in a report *Management Quality System and Testing Review* and sent to senior management.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at Columbia Analytical are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at Columbia Analytical when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at Columbia Analytical. Safety training begins with the reading of the *Environmental Health and Safety Manual.* Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer.

Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s). Quality Systems training begins with Quality Assurance orientation for new employees and reading the *Quality Assurance Manual.* During the employees first year, the employee attends *Core Ethics* training and learns about Columbia Analytical Services quality systems. Each employee participates in annual *Ethics Refresher* training, which is part of the Columbia Analytical Improper Practices Prevention Program.

Columbia Analytical also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The “CAS University” education system, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained in the QA department. Training requirements and its documentation are described in the SOP (ADM-TRANDOC) *Documentation of Training.* A training plan is developed whenever an employee starts a new procedure to new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

17.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible, special requirements are as follows:
    - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
    - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
    - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 17-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

17.2 Continuing Demonstration of Proficiency

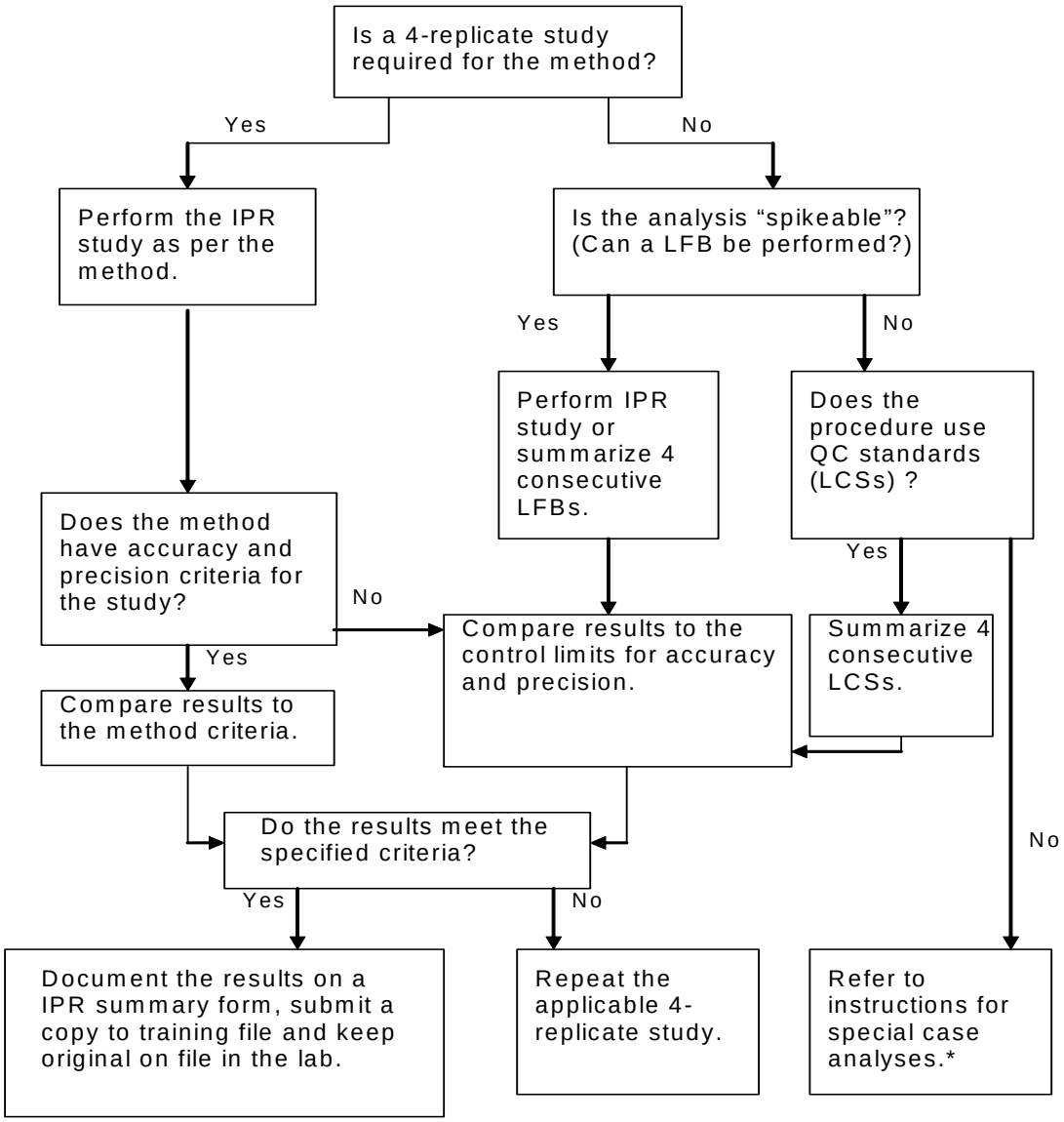
A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

17.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and Columbia Analytical resumes. QA maintains a database to record the various technical skills and training acquired while employed by Columbia Analytical. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Training (SOP No. ADM-TRANDOC).*

Figure 17-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per NELAP requirements
*Total Settleable Solids: Successful PT sample analysis and duplicate results with RPD<10%.
*Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
* Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES – EXTERNAL DOCUMENTS

The analytical methods used at Columbia Analytical generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at Columbia Analytical are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E)
- *Department of Defense Quality Systems Manual for Environmental Laboratories*, Final Version 3 (January 2006).
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 4.1, 4/22/2009
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).
- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4.
- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 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) and Supplements.
- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.
- *Standard Methods for the Examination of Water and Wastewater*, 18th Edition (1992); 19th Edition (1995), 20th Edition (1998). See Introduction in Part 1000.
- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.
- 40 CFR Part 141, National Primary Drinking Water Regulations.

- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.
- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).
- Annual Book of ASTM Standards, Part 31, Water.
- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM03.1, OLM03.2, OLM04.2, and OLM04.3.
- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0, ILM04.1, and ILM05.2.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).
- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).
- *Identification and Listing of Hazardous Waste,* California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).
- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

and

STANDARD OPERATING PROCEDURES

QA Program Files

Quality Assurance Manual	10/2/2009
Software Quality Assurance Plan	7/11/05
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	AppSignatories.pdf
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
Quality Control Acceptance Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001
Standard Operating Procedure Database	TrainDat.mdb

Corporate – Policies

POLICY TITLE	POLICY DATE	DATE APPROVED	DATE EFFECTIVE
CAS Quality and Ethics Policy Statement	March 2009	3/19/09	3/19/09
Policy for Data Review and Validation	May 2009	5/5/09	7/1/09
Policy for Internal Quality Assurance Audits	May 2009	5/5/09	7/1/09
Policy for Standards and Reagents Expiration Dates	September 2009	Final draft	9/28/09
Policy for Quality Assurance for Non-Regulated Testing	Draft	-	-
Policy for Use of Accreditation Organization's Name, Symbols, and Logos	Draft	-	-
Policy for Conducting Research, Technical Investigations, and Method Development	In development	-	-

Administrative SOP Corporate

SOP TITLE	SOP Code	Rev	SOP Date
SOP for Checking New Lots of Chemicals for Contamination	ADM-CTMN	4	1/26/09
SOP for Control Limits	ADM-CTRL_LIM	6	9/28/07
SOP for Corrective Action	ADM-CA	5	9/12/07
SOP for Data Recall	ADM-DATARECALL	0	9/21/07
SOP for Document Control	ADM-DOC_CTRL	7	1/27/09
SOP for Documentation of Training	ADM-TRANDOC	10	12/6/07
SOP for Estimation of Uncertainty of Measurements	ADM-UNCERT	4	12/30/08
SOP for Handling Customer Feedback	ADM-FDBK	4	12/10/07
SOP for Making Entries into Logbooks and onto Benchsheets	ADM-DATANTRY	8	9/8/09
SOP for Managerial Review of the Laboratory's Quality Systems	ADM-MGMTRVW	2	11/7/07
SOP for Manual Integration of Chromatographic Peaks	ADM-INT	3	8/28/07
SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	ADM-MDL	9	9/8/09
SOP for Preparation of Electronic-data for Organic Analyses for Electronic-data Audits	ADM-E_DATA	3	8/29/07
SOP for Preparation of SOPs	ADM-SOP	8	11/14/08
SOP for Preventive Action	ADM-PA	0	11/14/08
SOP for Proficiency Testing Sample Analysis	ADM-PTS	1	9/28/07
SOP for Purchasing Through SOP Purchasing Agent in Kelso	ADM-PUR	2	12/10/07
SOP for Qualification of Subcontract Laboratories Outside of SOP Network	ADM_SUBLAB	4	12/29/08
SOP for Significant Figures	ADM-SIGFIG	8	1/28/09

Administrative SOP Kelso

SOP Title	FILE NAME
CHECKING PIPETTE CALIBRATION	**ADM-CPIP**
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	**ADM-ECP**
CONTROL CHARTING QUALITY CONTROL DATA	**ADM-CHRT**
DATA ARCHIVING	**ADM-ARCH**
DATA REPORTING AND REPORT GENERATION	**ADM-RG**
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	**ADM-DOD**
ELECTRONIC DATA BACKUP AND ARCHIVING	**ADM-EBACKUP**
INTERNAL QUALITY ASSURANCE AUDITS	**ADM-IAUD**
LABORATORY BALANCE MONITORING AND CALIBRATION	**ADM-BAL**
LABORATORY DATA REVIEW PROCESS	**ADM-DREV**
PROJECT MANAGEMENT	**ADM-PCM**
REAGENT LOGIN AND TRACKING	**ADM-RLT**
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	**ADM-SEMC**
SAMPLE BATCHES	**ADM-BATCH**
SAMPLE MANAGEMENT SOPS	**FILE NAME**
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC

Technical SOP Kelso

SOP Title	FILE NAME
COLIFORM, TOTAL (DRINKING WATER)	BIO-9221DW
COLIFORM, FECAL	BIO-9221FC
COLIFORM, TOTAL	BIO-9221TC
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D
COLILERT® and COLITAG	BIO-9223
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B
COLILERT® COMPLETED TEST VERIFICATION OF E. COLI IN MUG CULTURES	BIO-CCT
ENTEROLERT	BIO-ENT
HEPTEROTROPHIC PLATE COUNT	BIO-HPC
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN
EPA CLP ORGANICS ANALYSES	CLP_ORGA
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510
CONTINUOUS LIQUID - LIQUID EXTRACTION	EXT-3520
SOLID PHASE EXTRACTION	EXT-3535
SOXHLET EXTRACTION	EXT-3540
AUTOMATED SOXHLET EXTRACTION	EXT-3541
ULTRASONIC EXTRACTION	EXT-3550
WASTE DILUTION EXTRACTION	EXT-3580
SILICA GEL CLEANUP	EXT-3630
REMOVAL OF SULFUR USING COPPER	EXT-3660
REMOVAL OF SULFUR USING MERCURY	EXT-3660M
SULFURIC ACID CLEANUP	EXT-3665
CARBON CLEANUP	EXT-CARCU
DIAZOMETHANE PREPARATION	EXT-DIAZ
FLORISIL CLEANUP	EXT-FLOR
ORGANIC EXTRACTIONS GLASSWARE CLEANING	EXT-GC
PREPARATION OF REAGENTS AND BLANK MATRICES USED IN SEMIVOLATILE ORGANICS ANALYSIS	EXT-REAG
ADDITION OF SPIKES AND SURROGATES	EXT-SAS
SOLID PHASE DISPERSION IN TISSUES	EXT-SPD
MEASURING SAMPLE WEIGHTS AND VOLUMES FOR ORGANIC ANALYSIS	EXT-WVOL
FACILITY AND LABORATORY CLEANING	FAC-CLEAN
OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS	FAC-WATER
FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020
COLOR	GEN-110.2
HARDNESS, TOTAL	GEN-130.2
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-160.1
SOLIDS, TOTAL SUSPENDED (TSS)	GEN-160.2
TOTAL SOLIDS	GEN-160.3
SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES	GEN-160.4
SETTEABLE SOLIDS	GEN-160.5
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300.1
ACIDITY	GEN-305.2
ALKALINITY TOTAL	GEN-310.1

PERCHLORATE BY ION CHROMATOGRAPHY	GEN-314.0
CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)	GEN-325.3
CHLORINE, TOTAL/FREE RESIDUAL	GEN-330.4
TOTAL RESIDUAL CHLORINE - METHOD 330.5	GEN-330.5
TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION	GEN-335
AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1
AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE	GEN-350.3
NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS	GEN-353.2
NITRITE BY COLORIMETRIC PROCEDURE	GEN-354.1
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3
DISSOLVED SILICA	GEN-370.1
GRAVIMETRIC SULFATE	GEN-375.3
SULFIDE, TITRIMETRIC (IODINE)	GEN-376-1
SULFIDE, METHYLENE BLUE	GEN-376-2
PHENOLICS, TOTAL	GEN-420.1
MBAS	GEN-425.1
HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION	GEN-5050
BIOCHEMICAL OXYGEN DEMAND	GEN-5210B
HALIDES, ADSORBABLE ORGANIC (AOX) - SM 5320B	GEN-5320B
TANNIN AND LIGNIN	GEN-5550
CYANIDE EXTRACTION OF SOLIDS AND OILS	GEN-9013
HALIDES, TOTAL ORGANIC (TOX)	GEN-9020
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030
TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY	GEN-9076
CARBON, TOTAL ORGANIC IN SOIL	GEN-ASTM
AUTOFLUFF	GEN-AUTOFLU
SULFIDES, ACIDS VOLATILE	GEN-AVS
HEAT OF COMBUSTION	GEN-BTU
CYANIDE, WEAK ACID DISSOCIABLE	GEN-CNWAD
CHEMICAL OXYGEN DEMAND	GEN-COD
CONDUCTIVITY IN WATER AND WASTES	GEN-COND
CORROSIVITY TOWARDS STEEL	GEN-CORR
HEXAVALENT CHROMIUM - COLORIMETRIC	GEN-CR6
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109
FERROUS IRON IN WATER	GEN-FeII
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26
MERCURY IN COAL SAMPLE PREPARATION BY PARR BOMB COMBUSTION	GEN-HGPREP
HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD
TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS
ION CHROMATOGRAPHY	GEN-IONC
COLOR, NCASI	GEN-NCAS
OXYGEN CONSUMPTION RATE	GEN-O2RATE
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU
Ph IN SOIL AND SOLIDS	GEN-Phs

Ph IN WATER	GEN-Phw
PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM
PARTICLE SIZE DETERMINATION	GEN-PSP
SULFIDES, REACTIVE	GEN-RS
TOTAL SULFIDE BY PSEP	GEN-S2PS
SULFITE	GEN-SO3
SPECIFIC GRAVITY	GEN-SPGRAV
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS
THIOCYANATE	GEN-THIOCN
NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN
POST DIGESTION DETERMINATION OF TOTAL KJELDAHL NITROGEN BY SEMIAUTOMATED COLORIMETRY	GEN-TKNAA
TOTAL ORGANIC CARBON IN WATER	GEN-TOC
TURBIDITY MEASUREMENT	GEN-TURB
ULTIMATE BOD	GEN-UBOD
GLASSWASHING FOR INORGANIC ANALYSES	GEN-WASH
Quantitative Determination of Carbamate Pesticides by High Performance Liquid Chromatography/Tandam Mass Spectrometry (HPLC/MS/MS)	LCP-8321
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY(HPLC)	LCP-8330B
QUANTITATION OF NITROAROMATICS AND NITRAMINES IN WATER, SOIL, AND TISSUE BY LIQUID CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY (LC-MS/MS)	LCP-LCMS4
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	LCP-NITG
QUANTITATION OF NITROPHENOLS IN SOLIS BY LIQUID CHROMATOGRAPHYAND TANDEM MASS SPECTORMETRY (LC-MS/MS)	LCP-NITRO
METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630S
METHYL MERCURY IN TISSUE BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630T
METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630W
MERCURY IN WATER BY OXIDATION, PURGE&TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY	MET-1631
MERCURY IN WATER	MET-245.1
METALS DIGESTION	MET-3005A
METALS DIGESTION	MET-3010A
METALS DIGESTION	MET-3020A
METALS DIGESTION	MET-3050B
CLOSED VESSEL OIL DIGESTION	MET-3051M
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020
ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7062
METALS DIGESTION	MET-7195
MERCURY IN LIQUID WASTE	MET-7470A
MERCURY IN SOLID OR SEMISOLID WASTE	MET-7471A/B
SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7742
CATION-EXCHANGE CAPACITYOF SOILS (SODIUM ACETATE) - METHOD 9081	MET-9081
SAMPLE PREPARATION OF AQUEOUS SAMPLES BY "CLEAN" TECHNIQUES	MET-ACT
BIOACCESSIBILITY OF METALS IN SOIL AND SOLID WASTE	MET-BIOACC
METALS DIGESTION	MET-DIG
FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRIC ANALYSES	MET-FAA
SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT
METALS LABORATORY GLASSWARE CLEANING	MET-GC

DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)	MET-GFAA
DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES	MET-ICP
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICP.MS
MULTIPLE EXTRACTION PROCEDURE	MET-MEP
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC
METALS AND SEMIVOLATILES TCLP EXTRACTION (EPA METHOD 1311)	MET-TCLP
SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS	MET-TDIG
TISSUE SAMPLE PREPARATION	MET-TISP
GRAVIMETRIC DETERMINATION OF HEAXANE EXTRACTABLE MATERIAL (1664)	PET-1664
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	PET-GRO
ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS	PET-SVF
ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS	PHC-WIDRO
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND INTERNAL CHAIN OF CUSTODY	SMO-SCOC
SAMPLE DISPOSAL	SMO-SDIS
CHLORINATED PHENOLICS BY IN-SITU ACETYLATION AND GC/MS	SOC-1653A
PHARMACEUTICALS, PERSONAL CARE PRODUCTS AND ENDOCRINE DISRUPTING COMPOUNDS IN WATER BY HPLC/TANDEM MASS SPECTROMETRY (HPLC/MS/MS)	SOC-1694
1,8-DIHYDROXYANTHRAQUINONE BY GC/MS SIM	SOC-18DHYDRAQ
GEL PERMEATION CHROMATOGRAPHY	SOC-3640A
ACETAMIDE HERBICIDE DEGRADATES IN DRINKING WATER BY SPE AND HPLC/MS/MS	SOC-535
ORGANOCHLORINE PESTICIDES AND PCBs (METHOD 608)	SOC-608
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-625
GLYCOLS	SOC-8015M
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081
PCBS AS AROCLORS - METHOD 8082A	SOC-8082AAr
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD - METHOC 8082A	SOC-8082ACo
PCBS AS AROCLORS	SOC-8082Ar
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD	SOC-8082C
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141
CHLORINATED HERBICIDES	SOC-8151
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - METHOD 8270D	SOC-8270D
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE	SOC-8270L
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SOC-8270P
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SOC-8270S
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	SOC-8310
ALDEHYDES BY HPLC	SOC-8315A

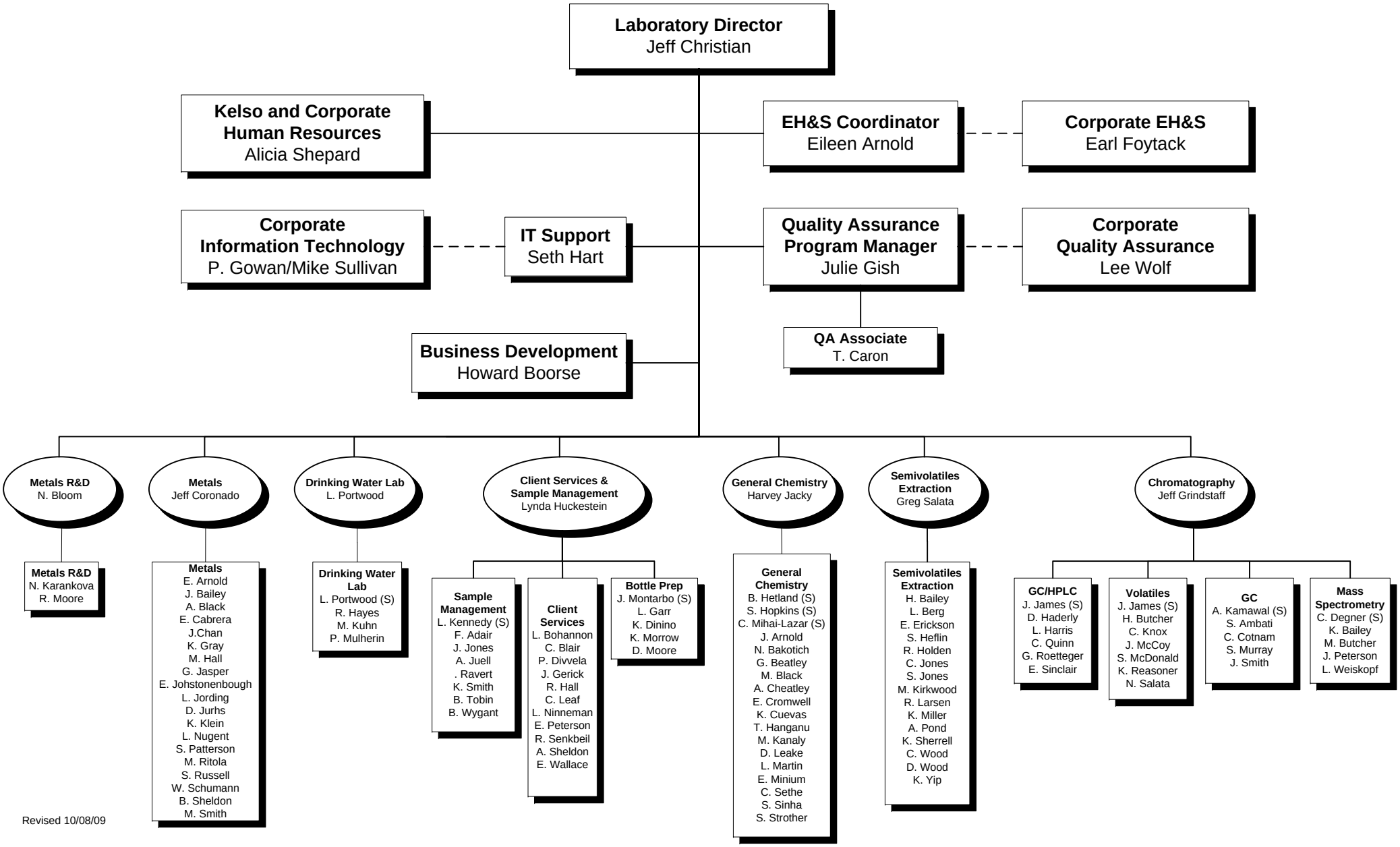
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8330
NITROGLYCERIN AND PETN BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8332
RESIN AND FATTY ACIDS BY GC/MS - NCASI METHOD 85.02 MODIFIED	SOC-85.02
METHANOL IN PROCESS LIQUIDS AND STATIONARY SOURCE EMISSIONS	SOC-9403
HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901
HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES	SOC-9902
BUTYLTINS	SOC-BUTYL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES USING EPA 8000C	SOC-CAL8000C
CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES	SOC-CONF
CPSC PHTHALATES BY GC/MS SELECTIVE ION MONITORING	SOC-CPSC
DIMP	SOC-DIMP
DMD SYNTHESIS	SOC-DMD
TOTAL OLEANOLIC ACID SAPONINS IN WATER BY ACID HYDROLYSIS AND HPLC/MS/MS	SOC-LCMS3
PERCENT LIPIDS IN TISSUE	SOC-LIPID
MONOCHLOROACETIC ACID BY GC-ECD	SOC-MCA
NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES	SOC-NONYL
ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC	SOC-OALC
EXTRACTION METHOD FOR ORGANOTINS IN SEDIMENTS, WATER, AND TISSUE	SOC-OSWT
CHLORINATED PESTICIDES BY GC/MS/MS, EPA METHOD 1699 MODIFIED	SOC-PESTMS2
PERFLUORINATED COMPOUNDS BY HPLC/MS/MS	SOC-PFC
PICRIC ACID AND PICRAMIC ACID BY HPLC	SOC-PICRIC
POLYBROMINATED DIPHENYL ETHERS (PBDEs) AND POLYBROMINATED BIPHENYLS (PBBs) BY GC/MS	SOC-ROHS
SEMI-VOLATILE ORGANICS SCREENING	SOC-SCR
1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC	SVD-504
ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515_4
N-NITROSAMINES BY GC/MS/MS	SVD-521
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)	SVD-525
SELECTED PESTICIDES AND FLAME RETARDANTS IN DRINKING WATER BY GC/MS (EPA METHOD 527)	SVD-527
DETERMINATION OF EXPLOSIVES AND RELATED COMPOUNDS IN DRINKING WATER BY GC/MS	SVD-529
CARBAMATES AND CARBAMOYLOXIMES IN WATER BY POST-COLUMN DERIVITIZATION HPLC	SVD-531 -1
GLYPHOSATE IN DRINKING WATER BY HPLC	SVD-547
ENDOTHALL IN DRINKING WATER BY GC/MS	SVD-548
DIQUAT AND PARAQUAT BY HPLC	SVD-549
HALOACETIC ACIDS IN DRINKING WATER	SVD-552
PURGE AND TRAP FOR AQUEOUS SAMPLES	VOC-5030
PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM	VOC-5035
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-524.2
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 602	VOC-602BTEX
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-624
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 8021	VOC-8021BTEX
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-8260
VOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING	VOC-8260S

VOA STORAGE BLANKS	VOC-BLAN
SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES	VOC-BVOC
ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)	VOC-ZHE

APPENDIX B

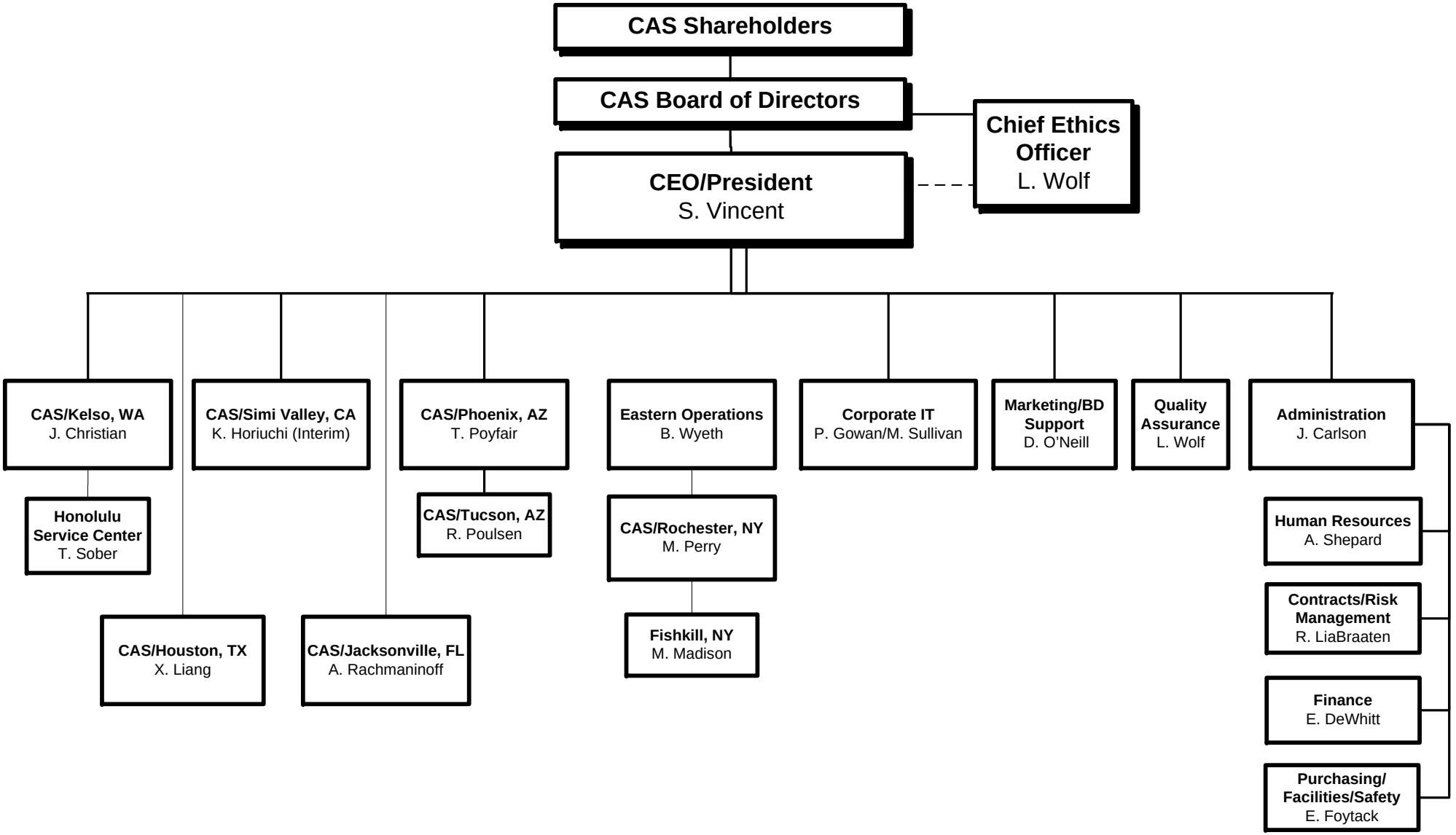
ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

Environmental and General Testing Division
Kelso, Washington
Laboratory Organization



Revised 10/08/09

Laboratory Division Organization



Revised .12/1/09

JEFFREY D. CHRISTIAN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**VICE PRESIDENT/NW REGIONAL DIRECTOR –** 1996 to Present
Responsibilities	Responsible for all phases of laboratory operations at the Kelso (WA) facility, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory (i.e. serves as the Kelso Laboratory Director, 1993-present). Also responsible for additional duties acquired as a member of the Columbia Analytical Services Holdings, Inc., Board of Directors.
Experience	**Laboratory Director, Kelso Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance. **Operations Manager, Kelso Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts. **Project Chemist and Manager, Metals Analysis Laboratory,** *Columbia Analytical Services, Kelso, Washington,* 1989-1992. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories. **Scientist,** *Weyerhaeuser Technology Center, Federal Way, Washington,* 1986-1989. Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods. **Lead Technician, Metals Lab,** *Weyerhaeuser Technology Center, Federal Way, Washington*, 1981-1986. Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing. **Research Assistant,** *ITT Rayonier, Olympic Research Division, Shelton, Washington,* 1978-1981. Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry.
Education	**B.S., Chemistry,** *Evergreen State College, Olympia, Washington,* 1993. **ICP/MS Training Course,** *VG-Elemental,* 1992. **Coursework,** *Pacific Lutheran University, Tacoma, Washington.* 1988-1989. **Coursework,** *Tacoma Community College, Tacoma, Washington.* 1970-1971, 1988-1989. **Perkin-Elmer Advanced Furnace,** *Norwalk, Connecticut,* 1986. **CERTIFICATION, Chemistry,** *L.H. Bates Technical, Tacoma, Washington,* 1978. **Coursework,** *Central Washington University, Ellensburg, Washington.* 1969-1970.
Publications/ Presentations	*Mr. Christian has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.*

JULIE GISH

1996 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER I, KELSO LAB QUALITY ASSURANCE MANAGER** – 2008 to Present
Responsibilities	Responsible for the overall implementation of the laboratory QA program. Responsible for the Quality Assurance Manual, certifications, documenting SOPs, and maintaining proficiency testing (PT) records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits and provides audit responses and initiates any corrective actions. Coordinate the analysis and reporting of PT samples. Conduct internal audits and make recommendations for corrective action.
Experience	**Scientist IV, Semi-Volatile Mass Spectrometry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Primary responsibilities were analysis, interpretation and report generation for semivolatile organics by GC/MS. Analyses included EPA 625, 8270, SIM, and other miscellaneous methodology.
	Technical Manager I, Semi-Volatile GC Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1999-2002. Primary responsibilities include supervision and oversight of semi-volatile GC department. This includes initiating new methods, staff training, workload management, and instrument maintenance/troubleshooting. Duties include departmental compliance with CAS QA and Safety policies. Responsible for analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, EPA 8141A, Organotins, and CLP Pesticides.
	Scientist III, Semi-Volatile Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-1999. Primary responsibilities were analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, and CLP-Pesticides. Secondary responsibilities include organics semi-volatile sample preparation.
	Scientist, Volatile Organics Sample Preparation, *Employer's Overload, Longview, Washington – assigned to the Columbia Analytical Services, Inc., Kelso, Washington facility,* 1996. Primary duties included the preparation of water, soil, sediment and tissue samples using EPA Methods 3510, 3520, 3540, 3550, and 3545. Other duties were the further clean up of extracts using EPA Methods 3620 (Florsil), 3610 (Alumina), 3630 (Silica gel), 3650 (Acid/Base Partitioning), and 3660 (Sulfur).
	Organics Chemist and GC/MS Chemist, *Coffey Laboratories, Portland, Oregon,* 1990-1996. Primary responsibilities included sample preparation and analysis for EPA FID, ECD, and HPLC using various EPA SW-846 and 500-series methods, as well as other methodology. Later, moved to GC/MS position which included sample preparation, analysis, and associated instrument maintenance for EPA Methods 625, 8027, and 525 BNA's. Also responsible for data review and approval of data packages.
	QC Manager/QC Supervisor and Product Manager, Corn Products, *Frito-Lay, Inc., Vancouver, Washington,* 1982-1990. Manager of the QC department overseeing three supervisors and approximately 30 technicians. Responsible for department cost, accuracy, timeliness of data and safety performance. Later, responsible for production oversight of brand name snacks. Responsible for cost, quality and safety performance over three shifts. Managed four supervisors directly and approximately 60 employees indirectly.
	Food Technologist, QA Department, *Kraft, Inc., Buena Park, California*, 1978-1981. Responsible for audits, formulations, finished product evaluation, batch reviews and technical support.
Education	**MS, Food Science, Minor in Industrial Engineering,** *Oregon State Univ. Corvallis, Oregon,* 1978. **BS, Food Science, Minor in Business Administration,** *Utah State University, Logan, Utah,* 1975
Publications/ Presentations	*Quality Improvement Team Leader,* Coffey Laboratories, Portland, Oregon. 1991
	Methods Improvement Program, Coffey Laboratories, Portland, Oregon. Seminars on Development and Implementation 1990.
	Statistical Process Control and Total Quality Management, Frito-Lay, Vancouver, Washington. Routine Training Classes 1986-1988.

GREGORY G. SALATA

2003 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**PROJECT/EXTRACTIONS MANAGER V** – 2003 to Present
Responsibilities	Responsibilities include Project Management, including quotation preparation and data reporting, as well as providing technical support to the laboratory as needed. Responsibilities also include oversight of the organic extractions lab, managing resources and providing technical support for all organic preparation work flows. 2003-Present.
Experience	**Project Manager,** *B&B Laboratories, College Station, Texas,* 1999-2003. Supervisor/responsible for analysis of TPH (waters, tissues, sediments), organotins (waters, tissues, sediments), Atterberg Limits (sediments), and total organic/inorganic carbon (sediments, waters). Also responsible for report generation on specific projects. Instrumentation operated included GCs with FID and FPD detectors, Combustion TOC, Water TOC, and Dionex Accelerated Solvent Extractor.
	Graduate Student, *Texas A&M University, College Station, Texas,* 1991-1999. While working toward MS in Oceanography, performed organic extractions for pesticides, PCBs, PAHs, and butyltins. While working toward Ph.D. in Oceanography determined stable carbon isotope ratios in sediments, waters, and bacterial phospholipid fatty acids. Other responsibilities included field sample collection, and operation/maintenance of FinniganMAT 252 isotope ratio MS.
	Analytical Chemist, *Science Applications International (SAIC), San Diego, California,* 1989-1990. Performed organic extraction and GC/FID analysis on sediment/rock samples for the Exxon Valdez oil spill.
	GC Chemist, *Analytical Technologies, San Diego, California,* 1987-1989. Responsible for analysis of volatile organics using purge and trap and GC/PID/ELCD.
Education	**Ph.D., Oceanography,** *Texas A&M University, College Station, Texas.* 1999 **MS, Oceanography,** *Texas A&M University, College Station, Texas.* 1993 **BA, Chemistry,** *University of California San Diego, Revelle College, La Jolla, California.* 1987
Publications/ Presentations	*Dr. Salata has a number of publications and published abstracts. For a list of these publications and published abstracts, please contact CAS.*
Affiliations	Society of Environmental Toxicology and Chemistry (SETAC)
	American Chemical Society

JEFFREY A. CORONADO

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER IV, INORGANICS DEPARTMENT MANAGER –** 2001 to Present
Responsibilities	Oversee the operation of the Metals Group. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Metals Department Manager,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-2001. Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence.. **Supervisor, GFAA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1989-1992. Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.
Education	**Field Immunoassay Training Course**, *EnSys Inc*., 1995. **Winter Conference on Plasma Spectrochemistry**, *San Diego, California,* 1994. **ICP-MS Training Course**, *VG-Elemental*, 1992. **BS, Chemistry,** *Western Washington University, Bellingham, Washington,* 1988. **BA, Business Administration,** *Western Washington University, Bellingham, Washington,* 1985.

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**CLIENT SERVICES MANAGER IV** – 1998 to Present
Responsibilities	Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Personally responsible for approximately 1.5 million dollars of client work annually performing technical project management and client service. Provides technical and regulatory interpretation assistance, as well as project organization of work received by the laboratory.
	Documentation of Demonstration of Capabilities is available for review.
Experience	**Project Chemist,** *Columbia Analytical Service, Inc., Kelso, Washington,* 1992-1998. Primary responsibilities included technical project management and client service in areas of pulp & paper, marine services, mining, and DOD. Also responsible for providing technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory
	Project Chemist and Department Manager, General Chemistry Laboratory, *Columbia Analytical Services, Inc.,* 1989-1992. Responsible for management of the General Chemistry laboratory for routine wastewater, bioassay, and microbiological analyses. Also responsible for supervision of staff, data review, and reporting.
	Analyst III, *Columbia Analytical Services, Inc., Kelso, Washington*, 1989. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and TKN, in addition to miscellaneous wet chemistry analyses.
	Microbiologist/Chemist, *Coffey Laboratories, Portland, Oregon,* 1983. Coliform analysis; water chemistry.
	Laboratory Assistant, *Oregon State University, Corvallis, Oregon,* 1983. Wheat spike dissection and tissue culture.
Education	**BS, Microbiology,** *Oregon State University, Corvallis, Oregon,* 1983.

HARVEY L. JACKY

1999TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER II** – 2008 to Present
Responsibilities	Oversee the operation of the General Chemistry and Microbiology groups. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Project Manager III,** *Columbia Analytical Services, Inc., Kelso, WA*, 1999-2008. Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies. **Director of Project Management,** *Coffey Laboratories, Portland, Oregon*, 1997-1999. Responsible for technical project management. Communicated with clients to determine needs and expectations. Monitored laboratory production and ensured the timely completion of analytical projects. Technical consultant for clients regarding environmental compliance. Supervised and managed other members of the project management team. Served as a member of the senior management team for oversight of general operations, strategic planning, finances, and policy. **Project Manager/Chemist,** *Coffey Laboratories, Portland, Oregon*, 1997-1999. Served as primary liaison between Coffey Laboratories and major clients. Ensured that work was completed in a timely manner and done to client specifications. Served as technical consultant regarding environmental chemistry, soil remediation, and waste water industrial compliance. Clients included the Oregon Department of Transportation, Hazmat Unit, Portland, Oregon; Raythion Demilitarization Co., Umatilla, Oregon; Hydroblast - Wastewater Evaporator Systems, Vancouver, Washington; and Union Pacific Railroad, Northwest Region, Klamath Falls, Oregon. **Technical Sales Representative,** *Coffey Laboratories, Portland, Oregon*, 1995-1997. Responsible for marketing and sales, including actively prospecting for new potential clients. Additional responsibilities included procurement and preparation of all major project bids; ensuring that client expectations were met; and maintaining customer satisfaction. Served as consultant regarding industrial compliance issues, environmental remediation projects, and hazardous waste management. **Senior Chemist/Laboratory Chemical Hygiene Officer,** *Coffey Laboratories, Portland, Oregon*, 1988-1995. Performed analytical tests including Anions by Ion Chromatography (EPA 300.0), PAHs by HPLC (EPA 8310), Cyanides (EPA 335), and other inorganic, wet chemistry, and organic analytical tests on a wide variety of sample matrices. Responsible for the initial quality assurance review of work performed, supervised and managed personnel. Developed and implemented Laboratory Chemical Hygiene Plan. Directed personnel in regards to safety issues and hazardous waste management. Served as consultant and teacher regarding analytical methodology, environmental compliance, and industrial hygiene.
Education	**40-Hour Hazmat Certification,** *PBS Environmental,* 1996. **Industrial Emergency Response,** *SFSP Seminar,* 1991 **BS, Zoology,** *Oregon State University, Corvallis, Oregon,* 1988. **BS, General Science,** *Oregon State University, Corvallis, Oregon,* 1988. **COURSEWORK, General Studies,** *Linfield College, McMinnville, Oregon,* 1981-1982.
Publications/ Presentations	*Biochemical and Physical Factors Involved in the Application and Measurement of a Soil Bioremediation System.* Biogeochemistry, Portland State University, 1996
Affiliations	American Chemical Society, Member since 1988

JEFFERY A. GRINDSTAFF

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER III, PHARMACEUTICAL, GC/MS VOA AND SEMI-VOA LABORATORIES, –** 1997 to Present
Responsibilities	Primary responsibilities include leadership of the Pharmaceutical, GC/MS VOA and Semi-VOA staff, management of method development, training, data review, tracking department workload, scheduling analyses. Responsible for ensuring data quality and timeliness. Also responsible for project management and coordination for pharmaceutical clients. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Manager, GC/MS VOA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1997. Responsible for supervision of GC/MS VOA staff, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems. **Scientist III, GC/MS VOA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1994. Responsibilities included scheduling workload, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and customer service. **Chemist,** *Enseco-CRL, Ventura, California,* 1990-1991. Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses. **GC/MS Chemist, VOA Laboratory** *Coast-to-Coast Analytical Service, San Luis Obispo, California,* 1990-1991. Responsible for standard preparation for VOA analyses, instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants.
Education	**Sampling and Testing of Raw Materials***, PTI International, 2004*. **Leadership Training,** *Richard Rogers Group, 1996* **Mass Selective Detector Maintenance,** *Hewlett Packard Education Center, 1993* **Interpretation of Mass Spectra I,** *Hewlett-Packard Analytical Education Center,* 1992. **B.S., Chemistry**, *California Polytechnic State University, San Luis Obispo, California,* 1989. **A.A., Liberal Arts,** *Allan Hancock College, Santa Maria, California.* 1986
Publications/ Presentations	*Low Level Analysis of 1,4-Dioxane by GC/MS SIM using Large Volume Injection,* with J. Peterson and R. Holden. SETAC National Meeting Poster Session, Portland, OR 2004. *Low Level Determination of N-nitrosodimethylamine by Chemical Ionization GC/MS with Large Volume Injection*, with C. Degner and J. Peterson. SETAC National Meeting Poster Session, Portland, OR 2004. *Analysis of Polybrominated Diphenyl Ethers by GC/MS with Large Volume Injection,* J. Peterson and M.Thompson SETAC National Meeting Poster Session, Portland, Oregon, 2004. *Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater*, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, FL 1996
Affiliations	American Chemical Society. 1989

NICOLAS BLOOM

2008 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position **Scientist VII** – 2008 to Present

Responsibilities **Senior Research Scientist**

Mr. Bloom has been involved in research on the biogeochemistry of trace metals in the environment for 30 years. After graduating from the University of Washington in 1979, he entered the graduate program in the Civil Engineering Department, where he worked as a full time researcher, investigating the sorption behavior of ultra-trace concentrations of cations and anions on ferric hydroxide suspensions. In 1980, Mr. Bloom was hired by the Battelle Marine Research Laboratory to develop sampling and analytical techniques to quantify a wide range of trace metals in sea water at ambient levels and apply those methods to the biogeochemical cycling of Hg, As, Ag, Pb, Cd, and Cu in Puget Sound. In 1984 Mr. Bloom returned to graduate school at the University of Connecticut, where he developed analytical techniques to allow the speciation of Hg at the sub-picogram level by GC-CVAFS. These methods have since been applied to investigate the cycling of Hg and its various compounds in lacustrine and marine systems throughout the world.

In 1991, Mr. Bloom founded Frontier Geosciences Inc., where he continued research into ultra-low level metals speciation in sediments, air, and fossil fuels, as well as mentored the development of IC-ICP/MS and IC-HG-AFS methods for most other trace metals and for Se, As, and Cr speciation. From 2001-2005, Mr. Bloom collaborated extensively with the Universita Ca'Foscari di Venezia in a study of Hg speciation and dynamics in the Venice Lagoon. In 2004, Mr. Bloom founded Studio Geochimica LLC, continuing his studies of the biogeochemistry of trace metals in the environment and industry. In 2008, Mr. Bloom joined Columbia Analytical Services, as Director of the Trace Metals Research and Development Department. In this position, Mr. Bloom is responsible for the development and validation of new trace metals speciation methodologies as well as working with clients and staff having biogeochemical questions or particularly perplexing analytical issues.

Experience

Research Scientist, *Battelle Pacific Northwest Laboratory, Marine Sciences Lab, Sequim, WA,* 1980-1989. As an analyst, developed and validated ultra-clean sampling methods and techniques for the analysis of all 13 EPA priority trace metals in water, sediment, and tissues with detection limits below the ambient background concentrations. As a researcher, emphasized biogeochemical processes of trace metals, particularly at the air/sea and sediment/water interfaces. Supervised two technicians.

Owner/Manager/Sr. Scientist, *Studio Geochimica LLC, Seattle, WA*, 2004 - 2008. Set up the scientific agenda, marketing, sales, inventing new analytical methods, mentoring, working in the lab as scientist and analyst, etc. Staff varied from 4 to 9 people.

Owner/Manager/Sr. Scientist, *Frontier Geosciences Inc., Seattle, WA*, 1991 – 2004. Set up the scientific agenda, marketing, sales, inventing new analytical methods, mentoring, working in the lab as scientist and analyst, etc. Staff varied from 3 in 1991 to 87 people in 2003.

Education

BS, Chemistry, *University of Washington, Seattle, WA*, 1979.

MS, Chemical Oceanography, *University of Connecticut, Storrs, CT* 1986.

Publications/ Presentations

Nicolas Bloom Mr. Bloom has approximately 120 publications on the biogeochemistry and analysis of trace metals in the environment (please inquire for publication list or copies of key papers), and has over 400 presentations at conferences and symposia world-wide.

Affiliations

ASTM, ACS (past member), ASLO (past member)

LOREN E. PORTWOOD

1992 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

Technical Manager I, DRINKING WATER LABORATORY – 2008 to Present

Responsibilities

Responsible for the overall operation and supervision of the Organic Drinking Water department. Also responsible for implementation and oversight of UCMR2 analyses. Perform method development. Project management of drinking water accounts. Development of Standard Operating Procedures for Drinking Water methods. Operation of Varian GC/MS, Agilent GC/ECD and Agilent HPLC.

Documentation of Demonstration of Capabilities is available for review.

Experience

Scientist IV, Drinking Water Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Plan, conduct, and, as lead analyst, supervise analyses using advanced instrumentation such as HPLC with post column derivatization, GC/MS, and GC/ECD. Responsible for data interpretation, quality control and data reporting. Additional responsibilities include preparation of SOPs and specifications for processes and tests; handling routine and advanced maintenance and troubleshooting of instrumentation; and assisting in the training of staff department analysts. Assists the department manager and/or other senior scientists in setting up more complex procedures. Serves as senior technical advisor for teams and projects.

Technical Manager I, Petroleum Hydrocarbon Laboratory Supervisor, *Columbia Analytical Services, Inc., Kelso, Washington,* 1998-2002. Primary responsibilities include organizing and prioritizing the workload for the petroleum hydrocarbon team, initiating new methods and process improvements, and staff development and training. Other duties include department wide compliance with CAS quality assurance guidelines, routine system checks, assist and encourage staff in troubleshooting equipment and procedural problems, and lead by example in a manner that is consistent with company, state and federal guidelines. Also responsible for duties listed below under Scientist II and Scientist III.

Scientist III, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1997-1998. Duties primarily as listed below.

Scientist II, **Petroleum Hydrocarbon Laboratory,** Columbia Analytical Services, Inc., Kelso, Washington, 1996-1997. Primary responsibilities included analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons and miscellaneous FID tests. Methods of analysis include EPA methods 8100, 8310, 8315, 8330, 8040, 8015 and various state modifications of 8015 (OR, WA, CA, AK). Additional analyses include solvent scans, alcohols, glycols, and EPA methods 413.2 and 418.1. Other responsibilities include sample preparation and instrument maintenance.

Scientist I, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1993-1996. Primary responsibilities included the analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons. Methods of analysis include EPA method 8015 and various state modifications thereof (OR, WA, CA, AK). Additional responsibilities include sample preparation, instrument maintenance, and assistance with other departmental analyses, including EPA methods 413.2 & 418.1.

Bench Chemist I, Organic Extractions Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1993. Primary responsibilities included the performance of a full range of semi-volatile sample preparations for water, soil, and oil to be analyzed in the GC, GC/MS, and Petroleum Hydrocarbon Laboratories. These extraction methods included hazardous waste, wastewater, and drinking water procedures. Other responsibilities included extract cleanup via Florisil®, GPC, and Hg.

Chemist, Treclen Laboratories, Spokane, Washington, 1990-1992. Primary responsibilities included inorganic water and soil testing by EPA methods. As Chemist, I developed the testing which was accredited by the EPA, which included everything from metal digestions, to phosphates, to TSS and TDS.

Education

Comprehensive HPLC Training, *Restek,* 2002.
Purge & Trap Theory and Troubleshooting, *Full Spectrum Analytics, Inc.,* 2001.
HP5890 GC Advanced Operations, *Hewlett Packard,* 1996.
HP6890 Fast GC, *Hewlett Packard,* 1996.
Quality Training, *Roger Tunks,* 1996.
Capillary Chromatography Training, *Restek,* 1993.
HP5890 GC Maintenance and Troubleshooting, *Hewlett Packard,* 1993.
BS, Chemistry, Emphasis in Biochemistry, *Whitworth College, Spokane, Washington*, 1990.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFTEY OFFICER –** 1994 to Present
Responsibilities	Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Project Chemist, Client Services Group, Kelso Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1994. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Scientist IV, Metals Laboratory, Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington, 1987-1992.* Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Chemist,** *Dow Corning Corporation, Springfield, Oregon,* 1986-1987. Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon. **Chemist,** *Ametek, Inc., Harleysville, Pennsylvania,* 1982-1985. Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques. **Chemist,** *Janbridge, Inc., Philadelphia, Pennsylvania*, 1978-1982. Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.
Education	**BA, Chemistry,** *Immaculata College, Immaculata, Pennsylvania,* 1977.
Affiliations	American Chemical Society, Member since 1987.

APPENDIX C

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (10):			
Precisa and Mettler models	1988-2008	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autotitrator – Thermo Orion 500	2007	LM	3
Calorimeters (2):			
Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	15
Colony Counter - Quebec Darkfield	1988	LM	4
Conductivity Meters (2):			
YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5):			
COD (4)	1987, 1989	LM	5
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	5
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	7
Drying Ovens (11):			
Shel-Lab and VWR models	1988 - 2003	LM	15
Flash Point Testers (2):			
ERDCO Setaflash Tester	1991	LM	4
Petroleum Systems Services	2005	LM	4
Flow-Injection Analyzers (2):			
Bran-Leubbe	2002	LM	4
Lachat 8500	2007	LM	4
Ion Chromatographs (4)			
Dionex 2000i with Peaknet Data Systems	1988	LM	3
Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Dionex ICS-2000 with Chromchem Data System	2006	LM	3
Ion Selective Electrode Meters (5)			
Fisher Scientific Accument Model 50	1997	LM	6
Fisher Scientific Accument Model 25	1993	LM	6
Fisher Scientific Accument Model 20	2000	LM	6
Orion Model 920A	1990	LM	6
Corning pH/ion Meter Model 135	1992	LM	6
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	15
pH Meters (2):			
Fisher Scientific Accument Model 20	1993	LM	6
Fisher Scientific Accument Model AR25	2005	LM	6

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Shatter Box - GP 1000	1989	LM	5
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	7
Total Organic Carbon (TOC) Analyzers (2)			
Coulemetrics Model 5012	1997	LM	3
O-I Corporation Model 1010	2002	LM	3
Total Organic Halogen (TOX) Analyzers (3):			
Mitsubishi TOX-Sigma	1995	LM	4
Mitsubishi TOX-100 (2)	2001	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	8
UV-Visible Spectrophotometers (3):			
Hitachi 100-40 Single Beam	1986	LM	5
Beckman-Coulter DU520	2005	LM	5
Perkin Elmer Lambda 25	2008	LM	5
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991	LM	13
Water Baths/Incubators (6):			
Hach Model 15320 Incubator	1986	LM	15
Precision Model L-6 (2)	1989, 1990	LM	15
VWR 1540	1991	LM	15
Fisher 11-680-626M Incubator	1992	LM	15
Fisher Isotemp Incubator	2001	LM	15

METALS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (6)			
Mettler AE 200 analytical balance	1990	MM	12
Various Mettler, Sartorius, and Ohaus models (5)	1988	MM	12
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA Zeeman/220 AA w/Data Systems (2)	2000	LM	3
CETAC Mercury Analyzer	2000	LM	2
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	2
Atomic Fluorescence Spectrophotometer			
Brooks-Rand Model III (2)	1996, 2005	LM	3
Leeman Mercury Analyzer (1)	2006	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (2) - Labconco	1992, 2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (3)			
Thermo Jarrell Ash Model 61E	1988	LM	4
Thermo Jarrell Ash, Model IRIS	2000	MM	4
Thermo Scientific Model iCAP 6500	2007	MM	3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	2
Muffle Furnace - Thermolyne Furnatrol Model 53600 (2)	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5

SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (4)			
Mettler PM480, AE166, BB300	1999 - 2005	MM	18
OHaus EP613	2006	MM	18
Centrifuge - Sorvall Model GLC-1	1988	LM	18
Drying Ovens (2)			
Fisher Model 655G	1991	LM	18
VWR Model 1305U	1999	LM	18
Evaporators (14):			
Organomation N-Evap (7)	1989-98, 2001, 2006	LM	18
Organomation S-Evap (7)	1989-1991, 2006	LM	18
Extractor Heaters: Lab-Line Multi-Unit Models for Continuous Liquid-Liquid and Soxhlet Extractions (102)	1987-1992, 2007	LM	12
Extractors (52):			
Branson Model 450 Sonifier (2)	1991	LM	6
Tekmar Sonicator	1994	LM	6
Fisher Scientific Sonicator	1994	LM	6
Soxhtherm (48)	2000, 2008	LM	8
Extractors, TCLP (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	2
TCLP Extractor - Tumbler (12 position)	1989	LM	2
Gel Permeation Chromatography (GPC) (5)			
ABC single column (3)	1998, 1999, 2007	LM	4
ABC Autoprep 1000	1995	LM	4
J2 Scientific	2005	LM	4
Muffle Furnace - 4	1994-2006	LM	4
Solid Phase Extractors (8) – Horizon SPE-Dex 4790	2003, 2006	LM	4
Ultrasonic Water Bath – VWR 550D	2007	LM	18
Vacuum Pump – Edwards	1992	LM	8

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler AT 250	1989	MM	7
Chromatography Data Systems (12)			7
HP Enviroquant (8)	1994-2002	LM	
Thruput Target (4)	1998-2000	LM	
Gas Chromatographs (11):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (4)	1990 – 1995	LM	7
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (5)	2001, 2005, 2007	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	LM	7
Agilent 7890A Dual ECD Detectors Agilent 7683B autosampler	2008	LM	7

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	5
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	5
Semivolatile GC/MS Systems (9):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	5
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	5
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler	1994	LM	5
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	LM	5
Agilent 6890/5973 with ATAS Optic3 LVI and 7683 Autosampler	2004	LM	
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler	2007	LM	4
Semivolatile GC/MS/MS – Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	1

PETROLEUM HYDROCARBONS GC/HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB240	1994	MM	6
Aspirator pump – GAST	2004	LM	6
Drying Oven - Fisher Model 630F	1991	LM	6
Evaporator - Organomation N-Evap	1990	LM	6
HP Enviroquant Chromatography Data Systems (8)	1994-2002	LM	6
Gas Chromatographs (6):			
Hewlett-Packard 5890 Series II with PID/PID/FID(2)	1991	LM	4
EST-ENCON Purge and Trap Concentrator	1991	LM	4
Dynatech Archon 5100 Autosampler	1992	LM	4
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector	1995	LM	4
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (3)	2001, 2005	LM	4
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	4
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	4
High-Performance Liquid Chromatograph/Mass(2) Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS and Autosampler	2005	MM	2
API 5000 LC/MS/MS and SIL-20AC Autosampler	2008	MM	2

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	5
Drying Ovens (2):			
Narco 420	1989	LM	5
VWR 1305 U	1991	LM	5
Sonic Water Bath - Branson Model 2200	1989	LM	5
Volatile GC/MS Systems (7):			
Agilent 5890/5970	1989	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 5890/5971	1991	LM	5
Tekmar 3000 Purge and Trap Concentrator	2001	LM	5
Dynatech ARCHON 5100 Autosampler	1995	LM	5
Agilent 5890/5972A	1993	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 6890/5973	2001	LM	5
Tekmar 3100 Purge and Trap Concentrator	2001	LM	5
Varian Archon Autosampler	2001	LM	5
Agilent 6890/5973	2005	LM	5
Tekmar Velocity Purge and Trap Concentrator	2005	LM	5
Tekmar Aquatech Autosampler	2005	LM	5
Agilent 6890/5973 (2)	2007	LM	5
Tekmar 3000 Purge and Trap Concentrator	2007	LM	5
Varian Archon 5100 Autosampler	2007	LM	5

DRINKING WATER ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB300	1991	MM	2
Extractors (10) – Horizon SPE-DEX Solid Phase Extractor	2003/2008	LM	2
Aglinet Enviroquant Chromatography Data Systems (2)	2003	LM	2
Varian Saturn Chromatography Data System	2003	LM	2
Evaporator - Organomation N-Evap	2003	LM	2
Agilent 1100 HPLC w/post-column derivitization: UV/Fluoescence detectors Pickering PCX-5200 Post-column derivitization unit	2003 2003 2003	LM LM LM	2 2 2
Agilent 6890N GC/Dual ECD system w/ autosamplers Agilent 7890 GC/Dual ECD w/autosamplers	2003 2008	LM LM	2 2
Varian Ion trap GC/MS: Varian 3800 GC w/CP8400 autosampler Varian Saturn 2100T mass spectrometer	2003 2006 2003	LM LM LM	2 2 2
Thremo Ion Trap GC/MS w/TriPlus autosampler	2008	LM	2

Metals Method Development Laboratory			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Perkin-Elmer ICP/MS Elan 9000 w/ Perkin-Elmer AS-93+ Autosampler	2008	LM	2
Perkin-Elmer Series 200 IC	2008	LM	2
Brooks Rand III Atomic Fluoresence Spectrophotometer - 2	2008	LM	2
Oriel Atomic Fluoresence Spectrophotometer – Lab Designed	2008	LM	2
Balances - 4	2008	LM	2
Ovens - 2	2008	LM	2
Buck AA Spectrophotometer Model 205	2008	LM	2
Forma Scientific Bio Freezer	2008	LM	2
Digital Shaker SK-71	2008	LM	2

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMS Sample Manager using Oracle 10g DBMS running on Redhat Advanced Server 3.0 (Linux) platform connected/linked on a frame relay WAN environment	1994-2004	LM	NA
1 - Network Server Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 Advanced Server, 1 for Applications running Windows 2003 Advanced Server. Data acquisition capacity at 65GB with redundant tape and disk arrays.	2004	LM	NA
Approximately 50+ HP and Dell Laserjet printers (various types including models III, 4, 5, 8150, 4000, 4050, 4250, 8150, 1720dn, W5300)	1991 - 2007	LM	NA
Approximately 180 Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2004	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97.	1996 - 2004	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Outlook Web Access interface. Microsoft Outlook 2003.	1994 - 2006	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Facsimile Machines - Brother 4750e (2); Brother SuperG3 (1); Canon CFX-L4000 (1)	1991 - 2007	LM	NA
Copiers/Scanners: Konica BizHub 420 (1), BizHub 600 (1), BizHub 920 (2), BizHub Pro 1050 (3). The 920s and 1050s are accessible via LAN for network scanning.	2000 - 2007	LM	NA
Dot Matrix Epson FX-880, LQ-1050, LX-300	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, StarLIMS reporting software systems.	1998 - 2004	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures	Daily
	Clean coils	Annually
	Check coolant	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured	Quarterly
	Sash operation	As needed
	Change filters	Annually
	Inspect fan belts	Annually
Ovens	Clean	As needed or if temperature outside lim.
	Record temperatures	Daily, when in use
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures	Daily, morning and evening
	Wash with disinfectant solution	When water is murky, dirty, or growth appears
Autoclave	Check sterility	Every month
	Check temperature	Every month
	Clean	When mold or growth appears
Analytical Balances	Check alignment	Before every use
	Check calibration	Daily
	Clean pans and compartment	After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride ISE	Store in storage solution	Between uses
Ammonia ISE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero	Weekly
	Check digestion/condensation vessels	Each use
	Clean digestion chamber	Every 2000 hours, or as needed
	Clean permeation tube	Every 2000 hours, or as needed
	Clean six-port valves	Every 200 - 2000 hours, or as needed
	Clean sample pump	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	Every 200 - 2000 hours, or as needed
	Clean IR cell	Every 2000 - 4000 hours, or as needed

Instrument	Activity	Frequency
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed
Flow Injection Analyzer	Check valve flares	Each use
	Check valve ports	Each use
	Check pump tubing	Each use
	Check light counts	Each use
	Check flow cell flares	Quarterly
	Change bulb	As needed
	Check manifold tubing	Each use
	Check T's and connectors	Each use
Ion Chromatographs	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro-photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency

Instrument	Activity	Frequency
ICP - MS	Check argon dewar	Daily
	Check water level in chiller	Daily
	Complete instrument log	Daily
	Replace peristaltic pump tubing	Daily
	Clean sample and skimmer cones	As needed
	Clean RF contact strip	As needed
	Inspect nebulizer, spray chamber, and torch	Clean as needed
	Clean lens stack/extraction lens	As needed
	Check rotary pump oil	Monthly
	Change rotary pump oil	Every six months
Gel-Permeation Chromatographs	Clean and repack column	As needed
	Backflush valves	As needed
High Pressure Liquid Chromatographs	Backflush guard column	As needed
	Backflush column	As needed
	Change guard column	As needed when back pressure too high
	Change column	Annually or as needed
	Change in-line filters	As needed
	Leak check	After column maintenance
	Change pump seals	As needed
	Change pump diaphragm	Annually
	Clean flow cell	As needed
	Fluorescence detector check	Daily
	Diode array absorbance check	Daily
Gas Chromatographs, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean FID	Weekly or as needed
	Clean ECD	Quarterly or as needed
	Leak test ECD	Annually

Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Annually or as needed
	Change septum	Daily, when in use
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications
Purge and Trap Concentrators	Change trap	Every four months or as needed
	Change transfer lines	Every six months or as needed
	Clean purge vessel	Daily
Gas Chromatographs, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean PID lamp	As needed
	Clean FID	As needed
	Change ion exchange resin	Every 60 days
	Replace nickel tubing	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Annually or as needed
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications

APPENDIX E

CORPORATE POLICY STATEMENTS

Columbia Analytical Services™

Policy for Data Review and Validation

May 2009
Effective July 1, 2009

The purpose of this policy is to identify the requirements for performing data review and validation prior to releasing data and reports to customers of Columbia Analytical Services. It is a requirement of NELAC (TNI) quality system standards and Department of Defense (DoD) agencies to have data review procedures established.

This policy is applicable to the review of raw and reported data generated in all laboratories. Specific data review and validation processes or logistics may vary somewhat from facility to facility, or vary for data generated using different methodologies however; the policies described here are to be followed. The documentation practices should be consistent within the facility. Automated validation processes are encouraged, but must be sufficiently described in an SOP.

In general, the data review and validation practices used at each facility will meet the requirements of NELAP quality system standards, the DoD Quality System Manual (QSM), and ISO 17025. Specific data review and validation policies are as follows:

1. Each laboratory facility will have a written and approved standard operating procedure (SOP) for conducting data review/validation that meets the standard CAS requirements for administrative SOPs. The SOP will list details of data review practices for the facility. The SOP will also give a detailed explanation of the review documentation procedures for each type of data.

2. Data review will be performed by qualified personnel who have documented training on either the analysis itself or training specific to the data review SOP. Personnel preparing reports who may do some level of clerical review or proofreading do not need technical knowledge of the test, but must be knowledgeable of reporting systems and requirements.

3. All data will be reviewed by a minimum of two persons. Data generated or reported by one person may not be released without another person's review.

4. However defined, one review (typically a "primary" technical review) must focus on the validity of the analysis and raw data generated, the technical accuracy and correctness of the analysis (the analytical procedure is in control), use of valid and approved procedures and methods, and interpretation of sample results.

5. The secondary review will be performed by someone other than the technical reviewer. The secondary review will make the same assessments as the primary reviewer, and check the interpretations, data manipulations, and decisions made by the primary reviewer. Additionally, the secondary reviewer will review the outputs from the initial review to the raw data. This includes such things as data processing results/outputs, calculations, runlogs, bench sheets, QC analyses, etc. The secondary review verifies the completeness and validity of the data to be reported.

6. All client-ready final reports will be reviewed in the format, and as presented to, the client; either by analysis fraction or in their entirety. This review will include verification of the accurate and correct reporting of sample and QC results; including accurate translation of results from data to report forms, report format, use of qualifiers and flags, and method citations. This review will also include verification of the correct project information; such as client name, project name, sample I.D.s, etc. The report review should ensure that the report is error-free and contains no inconsistencies. For upper tier deliverables, this review will verify that all deliverables are included in the report package.

7. The Project Manager will review all complete reports prior to signing the report and submitting to the client. The review of the reported data will focus on the following items:
    a. Consistency with client, contract, and/or project specifications.
    b. Acceptability of any data qualifiers or footnotes.
    c. Accuracy and completeness of explanations or discussion in the report cover letter or case narrative.
    d. As needed depending on the scope of testing, an additional level of technical review of all data generated.
    e. A general overview of the completed service request file with respect to overall reasonableness, and if available, with historical project information.

8. Data review must be documented. Persons performing data and report review must sign (or initial) and date the applicable data reviewed. Checklists or review summaries should be used for guidance and documentation. Documentation processes must be described in the laboratory SOP.

Lee Wolf, Corporate Director of Quality Assurance — Date: 5-5-09

Steve Vincent, President — Date: 5-5-09

Columbia Analytical Services™

Policy for Conducting Research, Method Development, and Method Investigations

December 2009

Columbia Analytical Services (CAS) often develops test procedures internally by conducting research and development or method development based on published procedures. This type of testing may not fall under common laboratory regulations which describe benchmarks, or minimum requirements, for procedure development and implementation. Also, it may be necessary at certain times to conduct investigations into the quality of existing methods. Therefore, a policy is necessary to identify and establish those minimum requirements.

The purpose of this Policy is to identify the CAS requirements for performing internal research and subsequent method development, performing method development from published references, and performing investigations into method performance.

For the purpose of this policy, the following Definitions are provided:

> *Research and development (R&D)* – The practice of independently evaluating analytical options and procedures and applying them to a sample analysis challenge; resulting in an internally developed analysis method. For this policy, R&D is limited to that performed by CAS personnel.
>
> *Method development* – The practice of implementing a CAS analysis procedure based on published references.
>
> *Method investigation* – For the purpose of this policy, this is defined as the evaluation of major changes in methodology outside the scope of published methods or SOPs. This is generally done to improve method performance or troubleshoot a significant analytical problem; and done outside of the routine maintenance, troubleshooting, and nonconformance/corrective action process.

The intent of this policy is to ensure that CAS R&D, method development, and method investigations are performed in an unbiased manner, ensure data integrity, use common scientific practices; and ensure that these activities are peer reviewed.

General Provisions

- When conducting any of the activities covered by this policy, employees will follow standard CAS procedures for maintaining documentation and analysis records.
- Initial and final review of statements, plans, and summaries will be done by two persons; the applicable Technical Director (TD) and the Laboratory Director (LD). If the TD is the LD, then a Peer will conduct the second review.
- Once development is concluded, the adoption of Standard Operating Procedures (SOPs), conducting personnel training, etc., will be done following routine CAS QA protocols.

Research and Development

When conducting research on new analyses and developing in-house procedures not based on reference methods or published methods, the research and development effort will include the following components:

1) There will be a written *Development Statement* detailing the intent of the research and development effort. This will state the purpose of the work, the resources and references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the statement:
   a) Equipment to be used.
   b) Quality Control measures to be incorporated into the analysis.
   c) Method Performance (validation) measures to be taken and expectations.
2) There will be an initial internal review and acceptance of the statement by the Technical Director and the Laboratory Director.
3) The person leading a R&D effort will gather information, references, and resources as described in the Development Statement and document those resources.
4) The experimentation will be performed and documented.
5) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
6) The developer will draw conclusions, and if successful, summarize the results in a brief R&D summary.
7) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
8) Following approval, an SOP will be written for subsequent implementation.

Method Development

When developing and implementing new methods based on reference or published methods, the method development effort will include the following components:

Non-certified (nor certifiable) methods

1) There will be a written *Development Statement* detailing the method development effort. This will state the purpose of the work, the reference method, references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the statement:
   a) The reference method being implemented and the application(s).
   b) Equipment to be used.
   c) Quality Control measures to be incorporated into the analysis.
   d) Method Performance (validation) measures to be taken and expectations.
   e) Modifications to the reference method.
2) There will be an initial internal review and acceptance of the statement by the applicable Technical Director and the Laboratory Director.

3) The experimentation will be performed and documented.
4) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
5) The developer will draw conclusions, and if successful, summarize the results in a brief method development summary.
6) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
7) Following approval, an SOP will be written for subsequent implementation on the stated applications.

Certified (certifiable) methods

1) There will be a written *Experimental Plan* detailing the method development effort. This will state the method being implemented, references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the Plan:
   a) The reference method being implemented.
   b) Equipment to be used.
   c) Quality Control measures to be incorporated into the analysis.
   d) Method Performance (validation) measures to be taken and expectations. This will include method and certification requirements for accuracy and precision, sensitivity, selectivity, calibration/linear range, etc. For methods where NELAC accreditation is being pursued, the requirements of the NELAC Standard (2003 Standard, Quality Systems section 5, Appendix C.3) will be met.
   e) Modifications to the reference method.
2) There will be an initial internal review and acceptance of the Plan by the applicable Technical Director and the Laboratory Director.
3) The method will be set up and run following the procedural steps of the method and the Plan; and will be documented.
4) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
5) The developer will draw conclusions, and if the results meet the method performance criteria in the method and/or Experimental Plan, the results will be summarized in a brief method development summary.
6) The summary report will include a documented approval the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
7) Following approval, an SOP will be written for subsequent implementation.

Method Investigations

1) There will be a written *Investigation Statement* detailing the method investigation effort. This will state the purpose of the investigation, the CAS procedure, the targeted problem, the experimentation that will be performed, and the desired improvement result. The following items will be included in the statement:
   a) The CAS procedure being investigated and the equipment used.
   b) A brief discussion of the problem, the solutions being investigated, and the impact on method compliance and data quality.
   c) The experimentation used to perform the investigation.
   d) The Method Performance (validation) measures that will be taken to re-establish conformity to QA/QC criteria.
2) There will be an initial internal review and acceptance of the statement by the applicable Technical Director and the Laboratory Director.
3) Once data is collected, it will be interpreted objectively using the assessments applicable to that analysis and CAS SOP.
4) The investigator will draw conclusions, and if the results meet the method performance criteria in the method and SOP, the results will be summarized in a brief method investigation summary.
5) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
6) Following approval, the CAS SOP will be revised to implement the changes to procedure.

Documentation

The developer or investigator will generate the written Development or Investigation statements, or Experimental Plan, and provide them for initial review prior to beginning experimentation and data collection. The initial review and acceptance of the Statement will be documented. The laboratory QA PM will keep this documentation on file.

The developer or investigator will generate the written summary report and validation package, and will submit supporting data for review. The approval of the development or investigation (and SOP changes) will be documented and the laboratory QA PM will keep this documentation on file.

Steve Vincent

Steve Vincent, President/CEO

12-15-09

Date

Lee Wolf

Lee Wolf, Chief Quality/Ethics Officer

12-15-09

Date

Columbia Analytical Services™

Policy for Standards and Reagents Expiration Dates

September 2009
Effective September 28, 2009

The purpose of this policy is to state the standardized requirements for assigning expiration dates to standards and reagents used in the laboratories of Columbia Analytical Services. It is a requirement of NELAP Quality System standards, the DoD Quality System Manual (QSM), and ISO 17025 to have written protocols to ensure the use of standards and reagents of appropriate quality. Additionally, documentation of the expiration date of reagents and standards is required. This policy is intended to meet the requirements of NELAC, DOD, and ISO 17025.

This policy is applicable to all purchased and prepared standards and reagents used by the laboratory to generate reported data. This includes raw (neat) materials, stock, intermediate, working, and calibration standards and/or reagents. This does not include solvents and acids.

In general, the expiration date is the date after which a standard or reagent shall not be used. It is either the date assigned by the manufacturer, the date (duration) specified by the applicable reference method, or it is a date assigned by the laboratory under this policy.

General Policies:

1. All standard and reagent expiration dates/periods shall be listed in the applicable laboratory SOP.

2. When establishing an expiration date, the following hierarchy will be used:

   - If the cited analytical method specifies the expiration date/period, that date shall be used.
   - If the cited analytical method does not specify the expiration date/period, then the date assigned by the manufacturer will be used.
   - If the cited analytical method does not specify the expiration date/period, and an expiration date is not assigned by the manufacturer, then the laboratory will assign the expiration date according to the CAS Standardized Expiration Dates tables below.

CAS Expiration Dates for Reagents	
Chemical	Expiration Date
Purchased neat reagents	5 years after receipt
Inorganic reagent solutions	1 year from preparation or receipt
Organic reagent solutions	6 months from preparation or receipt

CAS Expiration Dates for Standards	
Chemical	Expiration Date
Purchased neat standards	5 years after receipt
Inorganic stock standard solutions	1 year from preparation or receipt
Inorganic secondary, intermediate, or working standard solutions	6 months from preparation or receipt
Purchased semivolatile organic stock standard solutions	1 year from receipt
Prepared semivolatile organics stock standards	1 year from preparation
Semivolatile organic secondary, intermediate, or working standard solutions	6 months from preparation or receipt
Purchased volatile organics stock standards – **unopened** ampules	1 year from receipt
Purchased volatile organics stock standards – **opened** ampules	≤2000 mg/L 1 month after opening >2000 mg/L 3 months after opening
Prepared volatile organics stock standards	1 year from preparation
All volatile organics secondary, intermediate, or working standards* * note: common 'gases' standards and standards used for calibration should not be older than 7 days	≤20 mg/L 7 day expiration date >20 and ≤200 mg/L 1 month expiration date >200 mg/L 3 month expiration date
Dioxin/Furan and PCB stock standards	5 years from receipt
Dioxin/Furan and PCB working standards	1 year from preparation or receipt
Derivatized (prepared) semivolatile organics standard solutions	1 year from date of derivatization

3. The expiration date of a prepared reagent or standard cannot exceed the expiration date of the starting material, with the exception of standards prepared via in-lab derivatization to yield a different compound. The expiration date of a reagent or standard cannot be extended by preparing a dilution of it. For example, a purchased standard has an expiration date of July 15, 2009. A standard prepared on February 20, 2009 from this purchased standard would ordinarily have an expiration date of six months (namely, 8/20/2009), but since the purchased standard expires before six months, the prepared standard would be assigned an expiration date of July 15, 2009.

4. A multicomponent prepared reagent or standard will be assigned an expiration date not to exceed the expiration date of any of the components' expiration date. For example, a prepared standard is made from purchased standard A (with an expiration date of August 5, 2009) and from purchased standard B (with an expiration date of December 15, 2009). Consequently, the prepared standard will have an expiration date of August 5, 2009.

5. The stability and concentration of the reagent or standard are to be taken into account when assigning the expiration date. Certain solutions, depending on use and storage, may have shorter usable life time than defined by the method, manufacturer, or this policy; and should be assigned expiration dates accordingly. Reagents and standards must be stored under conditions specified by the test method and outlined in the analytical SOP.

6. Expiration dates can be extended under the following conditions:
   - A new, replacement reagent or standard is not readily available from vendors and,
   - The cited analytical method does not specify the expiration date/period and,
   - The material has been stored under conditions specified by the analysis method and outlined in the analytical SOP and,
   - The material is not reactive, volatile, or prone to degradation under the specified storage conditions and,
   - The suitability of the material is verified by the laboratory as follows, under the same valid analysis conditions used for sample analysis, and meet the following criteria:

     a. For reagents:
        i. Perform a blank and LCS pair of analysis three times using three different subaliquots of the reagent.
        ii. Each LCS result must be within the specified control limits for the test.
        iii. The %RSD for the three LCS's must be <10%.
        iv. Each blank result must be < 1/2MRL for every compound to be reported from subsequent analysis.
     b. For standards:
        i. Analyze three separate dilutions of the standard at a concentration near the midpoint of the calibration range. (Note that standards below this concentration cannot be re-verified).
        ii. The average result must be within ± 5% of the original true value.
        iii. The %RSD for the three results must be <10%.

     If these conditions and criteria are met and documented, the material may be assigned a new expiration period the same as newly prepared material.

Lee Wolf, Corporate Director of Quality Assurance — Date: 9-10-09

Steve Vincent, President — Date: 9-15-09

Policy for the Use of Accreditation Organization Names, Symbols, and Logos

September 2009

Effective October 1, 2009

The purpose of this policy is to state Columbia Analytical Services' (CAS) requirements and restrictions for the company use of the name, symbols, and logos of accreditation organizations. In general, the names, symbols, and logos used by these organizations are the property of the organization. Therefore, it is a policy that CAS will comply with the requirements and policies of the organizations that accredit our laboratories.

The NELAC Institute (TNI): The TNI Board of Directors approves and oversees the use of TNI logos and marks (TNI, NELAC, NELAP) by programs, members, and other entities. In consideration that CAS is a member of TNI, CAS will abide by the following TNI policy and be subject to the TNI Consequences of Misuse.

All persons and entities that use or reproduce TNI logos and marks:

1. *Shall restrict access to them by unauthorized parties.*
2. *Shall use them only for purposes and activities authorized by the TNI Board of Directors.*[a]
3. *Shall endeavor to avoid statements in relation to their use that the TNI Board of Directors may consider misleading or unauthorized.*
4. *May not imply endorsement or approval by TNI in communication media such as the Internet, documents, brochures, or advertising without the expressed consent of the TNI Board of Directors.*
5. *May not imply an association or partnership with TNI when such an arrangement has not been authorized by the TNI Board of Directors.*

[a] Authorized uses and activities are listed in the 2003 NELAC Standard, Section 6.8

American Association for Laboratory Accreditation (A2LA): CAS will comply with A2LA policy *P101 – Reference to A2LA Accredited Status – A2LA Advertising Policy*[b].

- CAS will only use the A2LA logo and symbol/phrase "A2LA Accredited" at individual CAS laboratory locations which have demonstrated to be in compliance with A2LA quality system requirements for the applicable A2LA accreditation program (e.g. Testing Laboratory).
- The "A2LA Accredited" symbol will not be used by a CAS laboratory that is not A2LA accredited and the symbol will not be used by a CAS laboratory that has only applied for accreditation.

- When promoting A2LA accreditation, CAS will follow the requirements of the A2LA policy.
- Where the "A2LA Accredited" symbol is used to endorse results on reports, it will always be accompanied by the A2LA certificate number and an indication of the type of laboratory (i.e., testing laboratory).

[b] The A2LA policy can be found at http://www.a2la.org/policies/A2LA_P101.pdf

International Organization for Standardization (ISO): ISO does not perform assessments and therefore is not a certification or accreditation organization. ISO is a standards development organization and compliance with an ISO standard does not imply ISO endorsement. ISO's statement on the use of the name and logo is listed below, and can be found at the following URL: http://www.iso.org/iso/support/name_and_logo.htm ISO has also provided a guide for how to publicize certification to an ISO standard: http://www.iso.org/iso/publicizing2005-en.pdf

Use of ISO's name®

Within the context of international standardization or related activities (such as consultancy, training or conformity assessment including certification) "ISO" (or "iso") is the short name of the International Organization for Standardization. The name is registered within this context as the sole property of ISO and the Organization will protect its name on behalf of all ISO's members - the national standards institutes of some 150 countries. In particular, ISO will not authorize the use of the name "ISO" (or "iso") by any organization other than its members in Internet domain names, names of Web sites, trademarks, companies / organizations, products, etc. Such use could mislead third parties into believing that the domain name / Web site / trademark / company / organization / product concerned represents ISO, or has been approved or authorized to act on behalf of ISO or belongs to ISO.

Therefore, ISO will take whatever actions it considers necessary to prevent the misuse of its name.

Use of ISO's logo®

The ISO logo is a registered trademark. Unless authorized by ISO, use of its logo is prohibited. Notably, ISO will not allow its logo to be used in connection with conformity assessment activities. These include the certification of management systems, products, services, materials or personnel, even when these certifications attest conformity to an ISO standard, such as one of the ISO 9000 or ISO 14000 series. Examples of unacceptable use of the ISO logo would include use on products, in publications, on Internet sites, in marketing materials, advertisements and company letterheads.

Allowing the ISO logo to be used would give the false impression that ISO carries out certification activities, or has approved or authorized the organization using its logo. These activities are not business functions of ISO.

ISO is not an auditor, assessor, registrar, or certifier of management systems, products, services, materials or personnel, nor does ISO endorse any such activities performed by other parties. ISO develops International Standards but does not operate any schemes for assessing conformance with them.

Therefore, ISO will take whatever actions it considers necessary to prevent the misuse of its logo.

The organizations specifically discussed in this policy do not comprise a complete list of organizations to which the policy applies. It is reiterated that, with regards to the use of names, symbols, and logos; it is a policy that CAS will comply with the policies of the organizations that accredit our laboratories.

Lee Wolf, Corporate Director of Quality Assurance — Date: 9-21-09

Steve Vincent, President — Date: 9-21-09

Columbia Analytical Services™

Policy for Internal Quality Assurance Audits

May 2009
Effective July 1, 2009

The purpose of this policy is to identify the requirements for performing internal systems audits and data audits in the laboratories of Columbia Analytical Services. Internal audits are necessary to ensure that laboratory operations work within the quality systems and that these systems yield data of high quality. Internal audits are also necessary in order to meet certification and accreditation requirements. The internal auditing practices used at each facility will meet the requirements of NELAC quality system standards, the Department of Defense (DoD) Quality System Manual (QSM), and ISO 17025.

For systems audits, the concept of this policy is that corporate quality assurance audits will evaluate the laboratory QA systems and operation horizontally, or as an overall 'umbrella' assessment, whereas local QA audits will be 'drill down' audits focused on technical correctness and data validity. It is practical to verify related systems implementation as these audits are conducted.

For electronic data auditing, the concept is to assess critical data from high liability steps of procedures, from all applicable instruments, in a frequent manner (quarterly) so as to identify any potential problems relatively quickly. This is in contrast to performing 100% data assessment from a subset of instruments quarterly and taking a long period of time to assess all instruments.

Definitions

- System audits are audits used to evaluate quality system implementation, policies, procedures, laboratory practices, and testing activities of the laboratory.

- Data audits are used to assess reported laboratory data. This includes all data used to generate the reported results and the final report itself. These are performed as 'desk audits' of reported data packages, and supporting data if not included in the reported data.

- Electronic data audits are used to assess laboratory data that is processed, interpreted, used by the analyst in electronic format. This is generally limited to electronic chromatographic data.

    - "Critical" and "high liability" data – Data related to the tuning, calibration, calibration verification, and QC analyses for an analysis; as well as data vulnerable to improper manipulation (improper processing/reprocessing of files, clock changes, poor interpretation of control data, peak integrations, etc., as described in CAS Ethics policies).

Specific internal auditing policies are as follows:

1. A comprehensive internal audit will be conducted annually (approximately every 12 months) at each laboratory. The audit will address all elements of the quality system and will include environmental testing activities, and used to meet the annual internal audit requirements of NELAC, DoD, and ISO 17025. In general, the comprehensive audit will be conducted and lead by the Quality Assurance Director (QAD), with assistance from the laboratory Quality Assurance Program Manager (QA PM).

   The laboratory QA PM will not be required to conduct an additional comprehensive audit. While performing the system and data audits described below, the QA PM will verify ongoing implementation of many QA systems.

2. Each laboratory QA PM will conduct three technical systems audits per calendar quarter. These audits will be technically-focused audits of three different test procedures and technologies.
   a. The three procedures will be varied throughout the year such that analytical disciplines (e.g. digestion, extraction, ICP, ICP/MS, titrimetric, colorimetric, GC, GC/MS, HPLC, microbiology, etc) from all sections of the laboratory are assessed in a year (for laboratories with fewer than 12 tests performed, the same tests will be audited more than once).
   b. The audits will assess SOP and method compliance.
   c. The audits will assess the use of sound analytical techniques and practices.
   d. The audits will assess the analyst(s) training and documentation of /proficiency.
   e. The audit will assess all aspects of the test being evaluated, including sample handling/preparation, calibration, sample batching/run sequences, standards, quality control, instrument operation/maintenance, data interpretation, data review/reporting, and applicable quality assurance.

3. Each laboratory will conduct two complete hardcopy data audits per quarter. These audits will focus on data validity, accuracy, and completeness. Data audits will be performed on hardcopy raw and reported data (or electronic version of) and on a 'Service Request basis'.
   a. The audits will be performed on data generated no earlier than three months prior to the audit.
   b. Service requests are to be chosen at random to encompass various analytical disciplines of the laboratory over the course of a year.
   c. The audit will assess the validity of the laboratory procedures used to generate the results reported, from sample receipt to analysis to data reporting, and the accuracy and completeness of the final report.
   d. The audit may be used as a convenient way to assess training documentation for the analysts who performed the analyses.

4. DoD report reviews will be conducted quarterly at the frequency required by the DoD QSM.

5. Electronic data auditing

    a. Each laboratory will conduct random screening of chromatographic data using Mint Miner software (where analytical software is compatible) every quarter on every instrument on data generated that quarter.
    b. Mint Miner software will be adequately configured in order to make screening effective.
    c. Using the screening results, data files will be selected for auditing from each instrument each quarter. Two sequences will be audited, one an initial calibration and one a typical sample analysis sequence. Test methods are to be chosen at random to encompass various methods performed.
    d. The audits will focus on calibration and QC data, including the evaluation of proper processing of files, interpretation of data, peak integrations, and comparison of raw electronic data to 'interpreted' and approved data.
    e. If screening results indicate significant potential problems, additional files should be inspected. The QA PM will conduct these added audits as needed.
    f. If Mint Miner software is not compatible with instrument software, auditing will be performed manually by the QA PM by auditing the data from two sequences per quarter, including one initial calibration sequence, per instrument.

6. As with any audit, additional auditing and investigation may be necessary based on the audits performed and magnitude of findings.

7. Each laboratory facility will have a written and approved standard operating procedure (SOP) for conducting their internal audits. The SOP will include detailed procedures for technical system audits, data audits, and electronic data audits as defined in this policy. In addition to meeting the standard CAS requirements for administrative SOPs, the SOP will include details of the audit processes, use of checklists, documentation, audit reporting, corrective action, and resolution of audit findings.

Lee Wolf, Corporate Director of Quality Assurance — Date: 5-5-09

Steve Vincent, President — Date: 5-5-09

Columbia Analytical Services

CAS Quality and Ethics Policy Statement

March 2009

Columbia Analytical Services (CAS) vision is simple. We strive to be the best in everything we do. This includes ethics and professional practice where CAS is committed to the highest standards of ethical behavior and quality of its analytical testing.

Unethical behavior carries a heavy price - one that we do not want to bear. This includes loss of reputation, loss of business, civil and criminal penalties, and government and customer sanctions.

CAS is committed to excellence and superior performance in everything we do. We will not sacrifice our ethical principles in order to achieve business success. This means we will always strive to conduct business honestly and with integrity. We will always follow and obey the law of the land in which we are operating our business. We will always follow, to the best of our ability, standard operating procedures, rules and regulations that apply to our industry and specifically to our laboratory operations. Our customers, employees, suppliers and communities that we serve expect and deserve nothing less than the highest standards of conduct and compliance.

The following are the critical elements of the Quality and Ethics program at CAS.

- The Executive Management and Board of Directors of CAS sponsor and support the Quality and Ethics program through their personal commitment and by providing the necessary resources to promote this program throughout the organization.
- Chief Quality and Ethics Officer. The position is responsible for the quality and ethics program, ensures that appropriate resources are provided, reviews and recommends changes in the program, and resolves ethical and quality issues brought to management attention. This Officer reports directly to the Board of Directors Audit Committee on quality and ethics.
- Core Values. The CAS Statement of Core Values was developed internally with input from the entire company. We are committed to ensuring the integrity and quality of data, and meeting the needs of our clients, while conducting business with high ethical standards. We hold strong to the core values of Honor, Truth, and Fairness. We are committed to these values and rely on them when confronted by difficult choices.
- Ethical Code of Conduct. As a member of the American Council of Independent Laboratories (ACIL) and part of the laboratory industry, CAS subscribes to and supports the core values and ethical codes established by this industry organization.

- CAS Code of Conduct. CAS requires its employees to be introduced to and to sign the "CAS Commitment to Excellence in Data Quality" statement and to comply with standards outlined in Section 6, Employee Conduct, of our Employee Handbook. All personnel concerned with analytical testing activities within the laboratory are required to acquaint themselves with the quality documentation and to implement these policies and procedures in their work.
- Open Door Policy. Employees have the right and obligation for open communications to ask questions, seek guidance, and report incorrect practices and wrong doing without fear of retribution. As described in the CAS Open Door Policy; CAS believes in using the chain-of-command channels for this dialogue. However, if there is fear or a concern that using this approach is not appropriate, employees are free to take their concerns to the President, the Director of Human Resources, the Chief Administrative Officer, the Chief Quality Officer, or the company Ombudsman. Employees may do so without fear of retribution.
- Ombudsman Program. CAS has implemented an external ombudsman/hotline program through EthicsPoint, a phone and internet-based reporting system, to enhance communication and empower employees to promote safety, security, and ethical behavior. Employees can file a report anonymously to address issues in the workplace and to cultivate a positive work environment.
- Internal Audits. Policies are established to ensure that internal systems and data audits are conducted periodically in addition to external agency and client audits. The data audits include a detailed in-depth review of hardcopy data and electronic data to ensure compliance with the CAS Quality program and on-going data integrity.
- NELAP Accreditation. CAS management is committed to compliance with the NELAP standards. CAS maintains NELAP accreditation and as such includes quality systems documented in QA Manuals, documented procedures in Standard Operating Procedures (SOPS) and policies, and documented training for demonstration of capabilities.
- Ethics Training. CAS has the obligation to provide training to its employees with respect to company policies concerning business conduct. This includes introductory training on this, and related policies, at the time of hire; in-depth "core" training within one year of hire, and on-going refresher training on a semi-annual basis.

The CAS Quality and Ethics Program has been in place for several years. However, this is a "living" program that will change and improve as the company grows and changes.

Steve Vincent, President/CEO — Date: 3-19-09

Lee Wolf, Chief Quality/Ethics Officer — Date: 3-19-09

ROUTINE PREVENTIVE MAINTENANCE

The GC is equipped with silicone septa (Supelco, Thermogreen, 10 mm) which eventually core and leak. Replace as necessary to avoid contaminating the injection port and to minimize column bleed.

Inlet sleeves can become contaminated and restrict flow after accumulating sample debris and septum particles. Replace the sleeve periodically to minimize loss in resolution.

In addition to sleeve replacement and when resolution degrades, clip about 6 inches off the front end of the column. Replace a column that becomes too short for adequate resolution after clipping.

SPARE PARTS

The main spare parts are detailed in the section above. However, here are the parts we keep on hand:

Septa – 10mm (Supelco Thermogreen)
Packed Injection Port Liners – straight (Supelco)
Ferrules – 0.8mm graphite-vespel (Restek)
Column – DB-5 30m x 0.53mm id (Agilent)
FID Jet
Gas-tight Autosampler Syringes – 10ul (Hamilton 701N)

SOP Change in Progress Attachment (CIPA)

SOP Number	SOP Title	SOP Revision	SOP Effective Date	CIPA Effective Date
BR-EX-001	Extraction Procedure for Organotins	8	05/14/10	08/05/10

The following revisions were made to this standard operating procedure (SOP). These changes are effective as of the CIPA Effective Date. Changes to this document will be incorporated into the document with the next revision. This document change is authorized and issued by the laboratory's QA Department.

Remove the following text from the SOP:

Page 8 of 18:

Section 10.3.1 Solid Extraction:

~~NOTE: For USACE work, extract the samples following the procedure given in laboratory SOP BR-EX-007 *Soxhlet Extraction, SW-846 3540C* using 0.05% tropolone/hexane as the extraction solvent.~~

Title: Extraction Procedure for Organotins

Approval Signatures:

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Approval Date: May 14, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the extraction procedure for the determination of organotins in water, soil, sediment, waste and tissue samples.

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used for the following matrices: non-potable water, soil/sediment waste and tissue.

The analyte list and reporting limits are provided in laboratory SOPs for instrument analysis.

2.0 Summary of Method

Soil, sediment and waste samples are extracted in hexane/tropolone using ultrasonic or soxhlet extraction. Tissue samples are homogenized and extracted in hexane/tropolone using a Tissuemizer. Water samples are extracted in hexane/tropolone by separatory funnel. The extracts are concentrated, exchanged into hexane and reacted with hexyl magnesium bromide to form the hexyl derivatives. The concentrated extracts are fractionated using Silica Gel/Florisil.

This procedure was developed in-house and is based on procedures described in NOAA Status and Trends Program Document: Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Vol. IV, NOAA Technical Memorandum, NOS ORCA 71.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference and glassware must be cleaned prior to use following laboratory SOP BR-EX-017 Glassware Cleaning Procedure.

Each batch of hexyl-magnesium bromide used for derivitization should be tested for contamination and effectiveness before use on field sample extracts.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Soil Extraction: Sonicators can generate high levels of noise and must be used in an appropriate noise reduction area.

Water Extraction: Hexane may create excessive pressure inside the separatory funnel. Initial venting of separatory funnels must be done immediately after the sample has been sealed and inverted. Vent into a fume hood away from your person or other analysts.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

6.1 Extraction Equipment

- High Intensity Ultrasonic Processor - Tekmar 600-watt Model Dual Output with pulsing capability, 3/4" standard disrupter horn.
- Tissumizer Fisher Scientific, Power-gen 700 with 20mm rotor shaft.
- Separatory Funnel shaker – Glas-Col.
- Soxhlet Water Bath
- Soxhlet Glassware: Top, bottom, and soxhlet thimble.
- Glass Funnel, 100 mm diameter. Fisher Scientific or equivalent
- Fiberglass Wool, 8 um. Fisher Scientific or equivalent
- Filter paper, Whatman No. 541 or equivalent.
- Beakers - 400 mL
- Spatula, Teflon or stainless steel Fisher Scientific or equivalent
- Teflon or Glass Separatory Funnel, 2 L with stopcock and stopper

6.2 Extract Concentration (KD Apparatus)

- Concentrator tube, 10 mL graduated.

- Snyder Column, Three ball macro
- Snyder Column, Two ball micro
- Evaporation Flask, 500 mL attached to concentrator tube with clip.
- Boiling Chips, silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride.
- Teflon Ultra Pure PTFE Boiling Stones. Chemware Catalog Number 0919120
- Heating mantle rheostat controlled for water bath capable of temperature control (± 5°C).
- Water Bath, capable of temperature control to ±5°C.
- Solvent Vapor Recovery System, Kontes K-54000-1006, K-547300-000, Ace Glass 6614-30 or equivalent.

6.3 Miscellaneous

- 75ul, 1mL & 10mL adjustable pipettes, Fisher Scientific or equivalent
- Balance, top loading, capable of accurate weight measurements to the nearest 0.01 g.
- pH Paper/Stripes: Range 1-14.
- pH Meter.
- Pasteur glass pipettes, 1 mL, disposable. Fisher Scientific or equivalent
- 0.5 mL – 2.0 mL Hamilton Gastight® syringes or equivalent.
- Vials and caps: 1.8, 4, 8, 16, and 40 mL with Teflon lined septa and screw caps. Fisher Scientific or equivalent
- Vacuum Box with Teflon inserts.
- Wrist Shaker.

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate, granular, anhydrous, (Na_2SO_4): J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours.
- Methylene Chloride (CH_2C_{12}): Pesticide quality, J.T. Baker or equivalent.
- Hexane, (C_6H_{14}): Pesticide quality, J.T. Baker or equivalent.

- Acetone, ($(CH_3)_2CO$): Pesticide quality, J.T. Baker or equivalent.
- Silica Gel/ Florisil Cartridges: 21 gram, Restek or equivalent.
- Tropolone: 98% neat Material, Aldrich Chemical or equivalent.
- Triethylamine, ($(C_2H_5)_3N$): Pesticide quality, Aldrich Chemical or equivalent.
- Hydrochloric Acid (HCl): J.T. Baker or equivalent.
- Magnesium Metal: Mallinkrodt or equivalent.
- 1-Bromohexane: 98% neat Material, Aldrich Chemical or equivalent.
- Diethyl Ether: Anhydrous, Pesticide quality, J.T. Baker or equivalent.

7.1.1 Prepared Reagents

- HCl Solution (1:1 v/v): Add 500 mL of reagent water to a 1 L volumetric flask. Slowly add 500 mL of HCl to the flask to dilute to volume. Store the solution in reagent bottle at room temperature. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.
- Tropolone/Hexane Solution (0.05%): Add 2.00 g of tropolone to a 4 L bottle that contains 4 L hexane. Tumble for one hour. Prepare fresh each day of use.
- Grignard Reagent (Hexylmagnesium Bromide) Preparation (2M):

  Dry all glassware, stir bars and other equipment used to prepare grignard reagent in an oven maintained at a minimum temperature of 105°C for at least 2 hours.

  While the equipment is still warm, fit a 1.0 L two or three-neck round bottom flask with a Liebig condenser and addition funnel sidearm to the glassware. Do not begin water flow at this time.

  Add gas inlet tubes to the top of the condenser and the addition funnel.

  Connect the gas lines from a regulator attached to the dry nitrogen cylinder to the gas inlet adapter on the sidearm addition funnel and from the gas inlet adapter on the condenser to the inlet side of the nitrogen bubbler.

  Remove the condenser and add 30 g of magnesium turnings to the flask. Add a stir bar and replace the condenser. Begin the stirring motor to agitate the turnings.

  Start the flow of nitrogen through the apparatus so that the flow approximates 20-30 mL/min. The nitrogen should sweep the entire apparatus in order to eliminate all trace of air and water vapor through the system. Allow the flow to continue for at least 30 minutes prior to the addition of any other reagent.

Remove the gas inlet adapter from the top of the addition funnel, open the stopcock on the addition funnel and add 500 mL of anhydrous diethyl ether to the round bottom flask through the addition funnel. Close the stopcock and add 140.37 mL or 165 grams of 1-bromohexane to the addition funnel. Replace the gas inlet adapter onto the addition funnel and continue the nitrogen flow.

Maintain the flow of nitrogen throughout the reaction. Start the water flow through the condenser at this point and continue until the reaction is complete. Reaction is complete when all of the 1-bromohexane is added.

Add ~10 mL of 1-bromohexane to the flask and continue stirring. Formation of bubbles around the magnesium turnings indicates that the reaction has started. After 5-10 minutes of continuous stirring, resume drop wise addition of reagent at a rate that prevents the diethyl ether from refluxing higher than 1/3 of the way up the condenser. These additions should take 30 minutes to 1 hour and the magnesium turnings will be mostly consumed in the reaction.

When the addition of 1-bromohexane is complete, stir the reaction for another 10 minutes.

Stop the stir motor, remove the condenser and the sidearm flask and quickly pour the reagent into dry 40 mL vials. Immediately cap the vials, and then label each vial with the reagent name, molarity (2 M), date made, expiration date and your initials. An expiration date of 1 year may be assigned to the prepared reagent unless the parent components expire sooner, in which case, use the earliest expiration date.

7.2 Standards

Purchase stock standard solutions from commercial vendors. From these, prepare surrogate and spiking solutions by diluting known volumes of the stock standard solutions in an appropriate solvent using the formulations provided in Appendix B. Record the preparation of the standard in the LIMS. Unless otherwise specified in Appendix B, store the prepared solutions in glass containers at a temperature of 4°C (±2) and assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Listed below are the recommended minimum sample size needed for analysis and the requirements for preservation and holding times.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]
Water	Glass	1L	4°C (±2°C)	Extraction: 7 days
Solid	Glass	50g	4°C (±2°C)	Extraction: 14 days
Tissue	Glass	50g	-15°C (±5°C)	Extraction: 14 days

[1]Extraction holding time is determined from sampling date; analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	With every batch if sufficient volume is available	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

10.0 Procedure

10.1 Instrument Calibration

Calibrate the pH meter on each day of use, prior to use using pH 4, 7 and 10 buffer solutions. Check the calibration of the balance each day of use prior to use with at least 3 Class S weights that bracket the range of use. Check the calibration of the pipettes per the frequency specified in laboratory SOP BR-QA-008.

10.2 Calibration Standard Preparation

Prepare calibration standards on request from the GC department.

To prepare each calibration standard and the calibration verification standard (ICV) prepare the parent standards using the formulation(s) given in Appendix B. Add the volume of parent standard(s) as specified in the following table to a 16 mL clear labeled vial that contains 5 mL of hexane.

Formulations for the Preparation of the Calibration Standards

Parent Standard	Level 1 (uL)	Level 2 (uL)	Level 3 (uL)	Level 4 (uL)	Level 5 (uL)	ICV (uL)
Tin Surrogate	50	100	1250	500	1000	1250
Tin Spike	50	100	1250	500	1000	1250

Derivatize each standard using the procedure given in Section 10.5. Concentrate calibration levels 1, 2, 4, and 5 to 1.0 mL in hexane. Concentrate level 3 and the ICV to 5.0 mL in hexane. Give he standards to the GC department.

The final concentration of each calibration level is provided in the following table.

Final Concentration of Prepared Calibration Standards

Component	Level 1	Level 2	Level 3	Level 4	Level 5	ICV

	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)
Tripentyltin Chloride	50	100	250	500	1000	250
Tetra-n-butyltin	50	100	250	500	1000	250
Tri-n-butyltin chloride	50	100	250	500	1000	250
Di-n-butyltin dichloride	50	100	250	500	1000	250
n-Butyltin trichloride	50	100	250	500	1000	250

10.3 Sample Preparation

10.3.1 Solid Extraction

Mix sediment samples thoroughly and discard any foreign objects such as sticks, leaves and rocks. Homogenize the sample following the procedures given in laboratory SOP BR-QA-020.

Label and place a 400 mL beaker on the balance and tare the balance. Weigh approximately 30 g ± 1 gram of sample into the beaker and upload the weight measurement into the LIMS worksheet. Use 30 g of purified sodium sulfate for the MB and LCS

Add 10 mL of 1:1 HCl solution to each beaker then add a sufficient amount of anhydrous granular sodium sulfate to each sample beaker and stir to create a free-flowing sample.

Add the 0.5 mL of surrogate solution to each field and QC sample. Add 0.5 mL of spike solution to the LCS and each MS/MSD.

Add 100 mL of 0.05% tropolone/hexane solution to each beaker. Extract the samples by ultrasonic extraction using the procedure specified in laboratory SOP BR-EX-008.

NOTE: For USACE work, extract the samples following the procedure given in laboratory SOP BR-EX-007 *Soxhlet Extraction, SW-846 3540C* using 0.05% tropolone/hexane as the extraction solvent.

Concentrate the sample using the techniques described in Section 10.4 to a volume of 5 mL in hexane.

Derivatize the sample following the steps in Section 10.5.

10.3.2 Tissue Extraction

Homogenize the tissue following the procedure given in laboratory SOP BR-EX-009.

Label and place a 400 mL beaker on the balance and tare the balance. Weigh approximately 30 g ± 1 gram of sample into the beaker and upload the weight measurement into the LIMS worksheet. Use 30 g of purified sodium sulfate for the MB and LCS

Add 10 mL of 1:1 HCl solution to each beaker then add a sufficient amount of anhydrous granular sodium sulfate to each sample beaker and stir to create a free-flowing sample.

Add the 0.5 mL of surrogate solution to each field and QC sample. Add 0.5 mL of spike solution to the LCS and each MS/MSD.

Add 100 mL of 0.05% tropolone/hexane solution to each beaker. Extract the samples by ultrasonic extraction using the procedure specified in laboratory SOP BR-EX-008.

Concentrate the sample using the techniques described in Section 10.4 to a volume of 5 mL in hexane.

Derivatize the sample following the steps in Section 10.5.

10.3.3 Water Extraction

Measure 1 L of sample into a graduated cylinder then quantitatively transfer the sample to the separatory funnel. Alternatively, if samples were received in 1 L containers, mark the meniscus of the aqueous volume on the sample container with a permanent marker. Pour the entire sample into the 2 L separatory funnel. Rinse the sample container with ~60 mL of troplone/hexane mixture and pour the rinsate into the separatory funnel extractor. To measure the actual sample volume, fill the sample container with tap water to the mark of the meniscus and pour the water into a graduated cylinder for volume measurement. Use 1000 mL of reagent water for the method blank and LCS.

Acidify each sample to pH < 2 with 10 mL of 1:1 HCl solution.

Add the 0.5 mL of surrogate solution to each field and QC sample. Add 0.5 mL of spike solution to the LCS and each MS/MSD.

Extract the samples using the procedure given in laboratory SOP BR-EX-005.

Concentrate the sample using the techniques described in Section 10.4 to a volume of 5 mL in hexane.

Derivatize the sample following the steps in Section 10.5.

1.2 Extract Concentration Techniques

Macro Snyder Column (K-D)

Add one or two clean boiling chips to the K-D evaporation flask and attach a three-ball Snyder column to the flask. Add ~1 mL of methylene chloride to the top of the column then place the K-D apparatus in a hot water bath (60-70°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 water vapor.

Attach the solvent vapor recovery glassware to the Snyder column. Adjust the vertical position of the apparatus and check the water bath temperature. The water bath temperature should be between 54.8 – 74.8°C when methylene chloride is the extraction solvent and 84-89°C when hexane is the extraction solvent. Higher water bath temperatures may be used so long as the recovery of target analytes is not impacted. The boiling point of each solvent is provided in the following table:

Solvent	Boiling Point	Water Bath Temperature
Hexane	69°C	84 – 89°C
Methylene Chloride	39.8°C	54.8 – 74.8°C

Monitor the concentration and do not let the extract evaporate to dryness. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with solvent.

When the apparent volume of the extract reaches desired amount remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

Micro Synder Column (K-D)

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column to the tube. Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and check the temperature of the water bath to ensure the proper temperature for the extract solvent.

Continuously monitor the distillation process to ensure sample extracts do not evaporate to dryness. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. Remove setup when desired sample volume is reached.

Nitrogen Blowdown

Nitrogen blow down may be used to concentrate extracts as needed.

Place the concentrator tube in a warm water bath maintained at a temperature of 35°C. Apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

10.4 Derivatization and Extract Cleanup

Perform sulfur cleanup on all soil and sediment extracts prior to derivitization. Refer to laboratory SOP BR-EX-002 for the sulfur cleanup procedure

Add 150 uL of triethylamine and 0.80 mL hexyl magnesium bromide to each extract then shake the extracts for 60 minutes using the wrist shaker.

Working in a fume hood, slowly add 1:1 HCl to each extract to dissolve the precipitate, and vortex the extract. Centrifuge the acid/extract mixture to separate the extract from the acid. Transfer the solvent phase to a concentrator tube. Rinse the acid layer with 1-2 mLs of hexane and transfer the acid rinse to the same concentrator tube.

Attach a 21g silica/florisil cartridge to the vacuum box using a Teflon insert. Elute 100 mL of methylene chloride through the cartridge followed by 100 mL of hexane at gravity flow rate. Remove the cartridge and Teflon insert and place it on top of a K-D flask.

Elute the extract through the cartridge using 100 mL of hexane. Collect extract in a K-D flask. Concentrate the extract to a final volume of 1.0 mL.

Transfer the extract to a labeled Teflon lined screw cap vial. Complete the extraction batch information in the LIMS batch.

11.0 Calculations / Data Reduction

11.1 Calculations

Not applicable.

11.2 Data Review

1.3 Data Review

Primary Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Enter the batch information into LIMS and complete the batch editor and worksheet for each extraction and cleanup performed. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

12.0 Method Performance

12.1 Limit of Detection (LOD) & Limit of Quantitation (LOQ)

A limit of detection (LOD) must be determined for the method if the laboratory reports results below the limit of quantitation (LOQ). If results are not reported below the LOQ, a LOD is not required but may be performed at the laboratory's discretion. The laboratory's procedures for LOD and LOQ are further described in laboratory SOP BR-QA-005.

12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

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

14.0 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 BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 gallon covered and vented drum.
- Extracted water samples - Satellite container: 55 gallon covered and vented drum.
- Vials containing extracts - Satellite container: 5 gallon covered bucket in fume hood.
- Hydrochloric Acid Waste-Satellite Container: 2.5L Waste Bottle Labeled with appropriate acid type (Hydrochloric).
- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)

15.0 References / Cross-References

- *Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992,* Vol. IV, NOAA Technical Memorandum, NOS ORCA 71.

16.0 Method Modifications

Not applicable.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Formulas

18.0 Revision History

Revision 7:
- Title Page: Updated approval signatures.
- All sections: Updated practice for LIMS implementation.
- Section 6.0: Added equipment

- Section 10.0: Added spike information
- Section 10.5: Added sulfur cleanup for soils and sediments

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methylene chloride	Cancer causing	25 ppm (TWA)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of with six months.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Magnesium Metal	Flammable Reactive w/ water Irritant Eye/vision damage		Flammable solid. Dangerous when wet. Highly reactive. May ignite spontaneously on contact with water or damp materials. May cause irritation to skin, eyes, and respiratory tract. Keep away from heat, sparks, and flame. Avoid breathing dust. Keep container closed. Use with adequate ventilation. Wash thoroughly after handling.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above-mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect, or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

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.

Appendix B: Standard Preparation Formulations

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock or parent standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in methylene chloride using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance.

Appendix B: Standard Preparation Formulas

Primary Source Tin Surrogate Solution

Stock Standard	Vendor	Component	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Tripentyltin Chloride Mixture	Restek #31477	Tripentyltin Chloride	2000	250	500	1.0

Solvent: Methylene Chloride

Primary Source Tin Spike Solution

Stock Standard	Vendor	Component	Stock Standard Concentration (ug/mL)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Butyltin Chlorides Calibration Mixture	Restek #31472	Tetra-n-butyltin	2000	250	500	1.0
		Tri-n-butyltin chloride				
		Di-n-butyltin dichloride				
		n-Butyltin trichloride				

Solvent: Methylene Chloride

Using a different lot of the Butyltin Chlorides Calibration Mixture (Restek #31472) prepare a Second Source Tin Spike Solution following instruction for the Primary Source Tin Spike Solution.

SOP Change in Progress Attachment (CIPA)

SOP Number	SOP Title	SOP Revision	SOP Effective Date	CIPA Effective Date
BR-GC-008	Organotins by Gas Chromatography (GC)	9	03/18/10	08/05/10

The following revisions were made to this standard operating procedure (SOP). These changes are effective as of the CIPA Effective Date. Changes to this document will be incorporated into the document with the next revision. This document change is authorized and issued by the laboratory's QA Department.

Change acceptance criteria for preservation for water and solids:

Page 4 of 22: Section 8.0 Sample Collection, Preservation, Shipment and Storage

Listed below are the recommended sample amounts needed for analysis and size, preservation and holding time requirements:

Matrix	Sample Container	Recommended Sample Size	Preservation	Extract Holding Time[1]	Reference
Water	Glass	1 L	Chilled to 4°C (~~± 5~~ 2°C)	40 Days	Lab
Solid	Glass	50 g	Chilled to 4°C (~~± 5~~ 2°C)	40 Days	Lab
Tissue	Glass	50 g	-15°C (±5°C)	40 Days	Lab

[1]Analytical holding time is determined from date of initiation of extraction.

Page 17of 22: Appendix B: Standard Preparation Tables

Final Concentration of Prepared Calibration Standards (as un-substituted alkyltin compounds)

Component	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	ICV (ug/L)
Tripentyltin	45.0	90.0	225	450	900	225
Tetrabutyltin	50.0	100	250	500	1000	250
Tributyltin	44.5	89.0	222.5	445	~~895~~ 890	222.5
Dibutyltin	38.5	77.0	192.5	385	770	~~19.25~~ 192.5
Monobutyltin	31.0	62.0	155	310	620	155

Title: Organotins by Gas Chromatography (GC)

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Approval Date: March 18, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure used to determine the concentration of organotins in environmental samples using dual column gas chromatography with flame photometric detectors (GC/FPD).

1.1 Analytes, Matrices, and Reporting Limits

This procedure may be used for a variety of matrices including: water, soil, sediment, waste and tissue.

The list of target compounds that can be determined from this method along with the associated reporting limits (RL) is provided in Table 1.

2.0 Summary of Method

3 uL of extract is injected into a dual capillary column gas chromatograph equipped with flame photometric detectors (GC/FPD). Organotins are quantified using external standard technique.

This procedure is a laboratory developed method derived from the NOAA Status and Trends Program Document: Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Vol. IV, NOAA Technical Memorandum, NOS ORCA 71.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

- Method interference may be caused by contaminants in the extraction solvent. Solvents should be stored away from possible sources of contamination.

- Matrix interferences may be caused by contaminants co-extracted from the sample. The extent of the interferences will vary depending on the nature and diversity of the samples.

- Each lot of hexyl-magnesium bromide used during the extraction procedure for derivitization should be tested by the GC department prior to its use to ensure that it is free of contamination.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst must be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

Table 2 lists 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.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

- Autosampler Vials, National Scientific or equivalent.
- Hydrogen Generator: Parker Balston.
- Volumetric Syringes, Class “A” (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

6.2 Analytical System

- Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5 or higher.
- GC/FPD: with dual columns, dual FPDs, and auto-sampler capable of a 3-µl injection split onto two columns: HP 7673A, HP 5890 with Leap Technology CTC A200SE and A200S Fisons autosamplers, Agilent Technologies 6890N with 7683 Series injector, or equivalent.
- GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses:
    - RTX-35, (30m x 0.32 mmID x 0.25um)
    - RTX-5, (30m x 0.32 mmID x 0.25um)

Equivalent columns may be used, provided the elution orders are documented and compound separations are maintained.

7.0 Reagents and Standards

7.1 Reagents

- Hexane, Ultra-Resi Analyzed, JT Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The extractions laboratory prepares all calibration standards, CCVs and ICVs. The recommended formulation for the standards used in this procedure is provided in the extraction SOP. The final concentration of each calibration level is provided in Appendix B of this SOP.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are the recommended sample amounts needed for analysis and size, preservation and holding time requirements:

Matrix	Sample Container	Recommended Sample Size	Preservation	Extract Holding Time[1]	Reference
Water	Glass	1 L	Chilled to 4°C (±5°C)	40 Days	Lab
Solid	Glass	50 g	Chilled to 4°C (±5°C)	40 Days	Lab
Tissue	Glass	50 g	-15°C (±5°C)	40 Days	Lab

[1]Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3
Matrix Spike(s) MS/MSD	1 pair per extraction batch when sufficient sample volume is provided or per client request	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Table 3
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 3
Continuing Calibration Verification (CCV)	Daily, every 10 samples, end of sequence	See Table 3
Retention Time Windows	As Needed	See Table 3

10.0 Procedure

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column into the injection port and connect the guard column to the separate analytical columns using a glass "Y". The analytical columns are installed into independent FPD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature:	100°C for 3 minutes
Temperature Program:	20°C per minute to 290°C Hold for 1 minute.
Detector Temperature	290°C
Injector Temperature:	225°C
Injection volume:	3µL
Carrier Gas:	Helium flow set at 6.0-10.0 mL/min
Makeup Gas:	Hydrogen and zero air (each supplied by gas generators) should be optimized for sensitivity, but is generally set at approximately 175 mL/min total flow

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

10.2 Retention Time Window Establishment

Whenever a new GC column is installed, establish RT windows for each analyte by analyzing three standards over a 72-hour period and calculating the mean RT and Standard Deviation (SD). Calculate the RT window as the mean RT $\pm$ 3SD. If the SD is <0.01 minutes, a default SD of 0.01 minutes may be used.

If this procedure results in a RT window that is too tight, favoring false negatives, the laboratory may opt to use an alternate method to determine the RT windows. An alternate method consists of using a RT window of $\pm$ 0.05 minutes. The center of the RT window is set at the midpoint calibration level in the initial calibration sequence. RT windows are then updated daily (minimum frequency), re-centering the windows on the retention times established in a CCV.

10.3 Instrument Calibration

10.3.1 Initial Calibration (ICAL)

Before initial or daily calibration, inject an instrument blank (IBLK) consisting of hexane to bring the GC/FPD system online.

The instrument is calibrated using a minimum of five different concentration levels for each target analyte. The calibration standards are prepared and derivatized following the procedures given in the extraction SOP. Prepare calibration standards for analysis by adding 10 uL of internal standard (tetra-n-propyltin) to 100 uL of standard in an autosampler vial insert. Cap the vials and place on the autosampler tray. Enter the standard names into the acquisition software. Set the autosampler to inject 3-µl of each calibration standard. Start the acquisition process and autosampler.

The data processing system calculates the Response Factor (RF), mean RF and Percent Relative Standard Deviation (%RSD) for each analyte on both columns. The %RSD for each target analyte must be less than or equal to 20% in order to use the mean RF for quantification. If this criterion is not met, use another suitable quantification method for that analyte or correct the problem and repeat the calibration. Once a method of quantification is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is performed.

Alternate Quantification Option:

Linear Regression & Weighted Linear Regression: Generate a curve of concentration vs. response for each analyte and calculate the correlation coefficient. The calibration must have a correlation coefficient (r) $\geq$ 0.995 (or $r^2 \geq$ 0.990). If this criterion is not met, correct the problem and repeat the calibration. The use of linear regression requires a minimum of 5 calibration points.

10.3.2 Second Source Calibration Verification (ICV)

Immediately after each calibration and prior to the analysis of QC or field samples, verify the accuracy of the initial calibration by analyzing a second source ICV.

The ICV is prepared and derivatized following the procedures given in the extraction SOP. Prepare the ICV for analysis by adding 10 uL of internal standard (tetra-n-propyltin) to 100 uL of ICV in an autosampler vial insert. Inject 3 µl of the ICV standard onto the instrument in the same manner as performed for the initial calibration standards.

The percent recovery of each analyte must be within $\pm$ 25% of the expected value (%R: 75-125). If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards or ICV standard and/or perform instrument maintenance and recalibrate. The QC acceptance criteria must be met on both columns.

10.3.3 Continuing Calibration Verification (CCV)

CCVs are prepared and derivatized following the procedures given in the extraction SOP. Prepare CCVs for analysis by adding 10 uL of internal standard (tetra-n-propyltin) to 100 uL of CCV in an autosampler vial insert. Inject 3 µl of the CCV standard onto the instrument in the same manner as performed for the initial calibration standards.

Analyze a CCV at or below mid-calibration level each day before sample analysis, after every ten injections and at the end of each analytical batch to monitor instrument drift. Calculate the RF and percent difference or drift (Appendix C) for each analyte on both columns. The percent difference or drift must be within ±25% for each analyte. Compare the RT of each analyte in the CCV with the established RT windows; the RT must be within the established window (refer to section 10.2). The acceptance criteria must be met on both columns.

If the CCV fails, it may be repeated once. If repeat analysis fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples must be bracketed by passing CCVs. Samples analyzed before and after CCV failure must be reanalyzed, unless the CCV is high and there are no detects in the associated samples.

10.4 Troubleshooting

Check the following items in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if the chromatography has degraded as evidenced by peak shapes.

- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column and remove one or more loops from each analytical column.

- Needle crushed during injection - Replace the needle and check the injection port for obstructions and check the autosampler for misalignment.

- Auto-sampler failure - Reset the auto-sampler.

- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

10.5 Sample Preparation

Remove the sample extract from refrigerated storage and warm to room temperature.

Prepare samples for analysis by adding 10 uL of internal standard (tetra-n-propyltin) to 100 uL of sample in an autosampler vial insert. Cap the vials and place on the autosampler tray. Enter the sample ID's into the data acquisition program.

10.6 Sample Analysis

Arrange the samples in a sequence that begins with the calibration standards and ICV followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs).

Enter the standard and sample names into the data acquisition program in the order the samples were placed in the autosampler tray and initiate the analytical sequence. Set the autosampler to inject 3-µl of each standard and sample onto the instrument.

An example analytical sequence that includes initial calibration (ICAL) and subsequent sample analysis is provided below.

Injection Number	Lab Description
1	Instrument Blank
2	50ppb Tin Standard
3	100ppb Tin Standard
4	250ppb Tin Standard
5	500ppb Tin Standard
6	1000ppb Tin Standard
7	Instrument Blank
8	ICV
9 - 18	10 injections
19	CCV (250ppb Tin Standard)
	Repeat steps 9 -19

Cleaning blanks (IBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention times of the peaks to the established retention time windows (refer to section 10.2).

Review and accept or reject the qualitative identifications made by the data processing software using the following guidelines:

Compare the retention times of the peaks to the established RT windows (refer to section 10.2), taking into account the shift of the surrogate peak. If the surrogate peak has shifted, open the retention time window in the direction of the shift. The processing software identifies the peak in the retention time window that is closest to the expected retention time set in the Target method, so the peak may need to be re-identified if a shift has occurred.

Look for shoulders on large peaks that may be the peaks of interest. The processing software does not always automatically integrate the shoulder off of the larger peak, so manual integration (split) of the shoulder may be necessary.

Each target analyte must be detected above the reporting limit on each column for qualitative identification to be made.

11.2 Quantitative Identification

The data system calculates the corrected concentration for each target analyte from the calibration curve using the equations given in Appendix C. If sample interference is suspected, the laboratory may choose to report the value from the result that is not affected by interference. The lower value between the two columns is reported unless otherwise specified for the project.

11.3 Calculations

See Appendix C.

11.4 Data Review

11.4.1 Primary Review

Review project documents such as the environmental test request (ETR) analytical worksheets, Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Confirm qualitative and quantitative identification criteria using the criteria provided in Section 11.1 and Section 11.2. If the data system does not properly integrate a peak, perform manual integration in accordance with laboratory SOP BR-QA-006.

Review the instrument QC against the acceptance criteria given in Section 10.0 and summarized in Table 3. If the results do not fall within acceptance criteria, perform the recommended corrective action. If corrective action is not taken or is not successful, document the situation with a nonconformance memo (NCM).

Dilute and reanalyze samples whose results exceed the calibration range. The dilution analysis should result in a determination within the calibration range, preferably in the upper half of the calibration range. A more concentrated analysis is not necessary unless the project requires it. Dilution analyses may also be performed to minimize matrix interference.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carry over. If carry over is suspected, reanalyze the sample.

Upload the data into the LIMS. Enter worksheet information and verify batch information is complete. Set results to primary, secondary, acceptable or rejected as necessary. Verify QC and calibration associations then set the batch to first level review.

11.4.2 Secondary Data Review

Spot-check quantitative and qualitative identifications using the criteria provided in Section 11.1 and Section 11.2.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-005. If a problem is found, immediately consult with the primary analyst or notify the Technical Director or QA Manager.

  Reintegration (by secondary data reviewers) should not be performed except in limited circumstances such as when the primary analyst who performed the initial integration is not available to correct any errors found during secondary review. If reintegration is performed, each integration performed by the secondary reviewer must be reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-005.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

- Verify a "before" and "after" chromatogram for every manual integration performed on an instrument performance check standard (Tune, ICAL, ICV, CCV), QC sample (MB, LCS) and for any manual integration performed on any surrogate or internal standard in any field sample was created.

- Document your review of manual integrations on the manual integration summary report and obtain any review signatures of integrations performed during secondary review as required.

Verify that the performance criteria for the QC items listed in Table 1 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. Verify an NCM was initiated for any QC that does not meet established criteria and verify data is qualified accordingly. Set samples to 2nd level review.

Run the QC Checker, investigate and correct any problems found. Run and review the deliverable. Fix any problems found then set the method chain to lab complete.

11.5 Data Reporting

Data reporting and creation of the data deliverable is performed by the LIMS using the formatters set by the project manager during project initiation.

The following sections describe the default reporting scheme for this method:

Analytical results above the reporting limit (RL) are reported as the value found. Analytical results less than the RL are reported as non-detect to the adjusted RL. The RL is adjusted for sample dilution/concentration. The unadjusted RL for each target analyte is provided in Section 1. If estimated values are requested, results between the LOD and the RL are reported and flagged as estimated.

Further guidance on the application and use of method detection limits (MDLs), reporting limits (RLs) and quantitation limits (QL) for the reporting analytical data is provided in laboratory SOP LP-QA-005.

Electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014 Laboratory Records.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

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

14.0 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 BR-EH-001. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.

- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

15.0 References / Cross-References

- NOAA Status and Trends Program Document: Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Vol. IV, NOAA Technical Memorandum, NOS ORCA 71.
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-QA-011
- Laboratory SOP BR-EH-001
- Laboratory SOP BR-QA-014
- Laboratory SOP BR-QA-006
- Laboratory SOP BR-QA-005

16.0 Method Modifications

Not applicable

17.0 Attachments

- Table 1: Target Compound List and Reporting Limit
- Table 1A: Accuracy and Precision Limits
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

18.0 Revision History

BR-GC-008, Rev. 9

- SOP updated to the new TestAmerica format.
- Standard concentration tables were added to Appendix B.
- Formulas in Appendix C were revised to use Response Factors instead of Calibration Factors.
- Language was added to section 10.2 to allow for updating RT windows on CCVs.
- Language was added to section 11.4.1 to allow for dilution to minimize matrix interference.
- Added statement after table 1A indicating Monobutyltin as a poor performer.

Table 1: Routine Target Analyte List & Reporting Limit (RL)

ANALYTE	Routine Reporting Limit (RL)[1,2]	
	Water (ug/L)	Solid (ug/Kg)
Tetrabutyltin	0.050	1.7
Tributyltin	0.045	1.5
Dibutyltin	0.039	1.3
Monobutyltin[1]	0.50	5.0
Tripentyltin (Surrogate)	N/A	N/A

[1]*The routine RL is the unadjusted value that can be achieved in a blank matrix.*
[2]*The RL for tissue matrix is project defined.*

Table 1A: Routine Accuracy and Precision Limits[1]

Analyte	In-House Limits[2] (%R)		Precision (RPD) (≤)
	Water	Solid	
Tetrabutyltin	30-150	30-160	30
Tributyltin	30-150	30-160	30
Dibutyltin	30-150	30-160	30
Monobutyltin[1]	10-48	10-48	30
Tripentyltin (Surrogate)	15-150	30-120	N/A

[1] *The control limits for Monobutyltin are advisory because this analyte is a poor performer.*

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	Option 1: RSD for each analyte ≤ 20% Option 2: Linear Regression: r ≥ 0.995	Correct problem, reanalyze, and repeat calibration.
ICV	After each initial calibration	(% R) ± 25% from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±25%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate	All field and QC samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Internal Standard: a known amount of standard added to a test portion of a sample as a

reference for evaluating and controlling the precision and bias of the applied analytical method.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to them.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in hexane using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 6 months from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance.

Internal standard (tetra-n-propyltin) is added to each calibration, ICV, CCV, and sample aliquot before analysis. 10 uL of internal standard is added to 100 uL of standard or sample aliquot for analysis.

Internal Standard Solution (5 mg/L)

Parent Standard	Vendor	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Tetra-n-propyltin	Retek #31474	2000	0.250	100	5.0

All working calibration standards, ICVs and CCVs for this method are prepared and derivitized by the organic prep department following the procedures given in the extraction SOP.

Final Concentration of Prepared Calibration Standards (as alkyltin chloride compounds)

Component	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	ICV (ug/L)
Tripentyltin Chloride	50	100	250	500	1000	250
Tetrabutyltin	50	100	250	500	1000	250
Tributyltin chloride	50	100	250	500	1000	250
Dibutyltin dichloride	50	100	250	500	1000	250
Monobutyltin trichloride	50	100	250	500	1000	250

Final Concentration of Prepared Calibration Standards (as un-substituted alkyltin compounds)

Component	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	ICV (ug/L)
Tripentyltin	45.0	90.0	225	450	900	225
Tetrabutyltin	50.0	100	250	500	1000	250
Tributyltin	44.5	89.0	222.5	445	895	222.5
Dibutyltin	38.5	77.0	192.5	385	770	19.25
Monobutyltin	31.0	62.0	155	310	620	155

The alkyltin chloride compounds are reported as un-substituted alkyltin compounds. The factors used to convert from the alkyl tin chloride to the alkyl tin are listed below.

Analyte	Conversion Factor	Report as
Tetrabutyltin	---	Tetrabutyltin

Analyte	Conversion Factor	Report as
Tributyltin chloride	0.89	Tributyltin
Dibutyltin dichloride	0.77	Dibutyltin
Monobutyltin trichloride	0.62	Monobutyltin
Tripentyltin chloride (SS)	0.90	Tripentyltin
Tetrapropyltin (ISTD)	--	Tetrapropyltin

Conversion Factors are determined from the following formula:

$$Conversion\ Factor = \frac{MWT - MWC}{MWT}$$

MWT= Total molecular weight of the analyte

MWC= Number of chlorides * molecular weight of chloride (molecular weight of chloride = 35.5)

Appendix C: Equations

Response Factor (RFx) = $\frac{\text{Peak area or height (x) x Concentration (is)}}{\text{Peak area or height (is) x Concentration (x)}}$

Where: x=compound, is = Internal Standard

Mean Response Factor ($\overline{RF}$) = $\frac{\sum_{i=1}^{n} RF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Response Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Response Factor =

$7\frac{SD}{\overline{RF}} \times 100\%$

Percent Difference (%D) = $\frac{RF_v - \overline{RF}}{\overline{RF}} \times 100\%$

where: RF_v = Response Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ x 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Measured concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Measured concentration of the Spiked Sample

C_u = Measured concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (RPD) = $\dfrac{|C_1 - C_2|}{\left(\dfrac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$$C_{extract}\,(ug/L) = \frac{\text{Peak response (x)}}{\text{Peak response (is)}} \times \frac{\text{Concentration (is)}}{\text{Average RF (x)}}$$

Where: x=compound, is = Internal Standard

Water

$$C_{sample}(ug/L) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solid

$$C_{sample}(ug/Kg) = C_{extract}(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\,\text{solids}} \times DF$$
