

TABLE OF CONTENTS

List of Figures	v
List of Tables	vi
Flagging Conventions	xi
Standard Operating Procedures.....	xii
List of Acronyms	xv
Terms and Definitions.....	xviii

QUALITY ASSURANCE PROJECT PLAN

1.0	PROGRAM MANAGEMENT.....	1-1
1.1	Program/Project Organization	1-1
	1.1.1 Program Manager.....	1-1
	1.1.2 Quality Assurance Officer	1-1
	1.1.3 Technical Manager.....	1-2
	1.1.4 Project Manager	1-2
	1.1.5 Project Chemist.....	1-3
	1.1.6 Program Geologist	1-3
	1.1.7 Health and Safety Officer	1-3
	1.1.5 Sampling Team Leader.....	1-3
	1.1.6 Field Personnel.....	1-3
1.2	Problem Definition / Background.....	1-4
	1.2.1 Location and History.....	1-4
	1.2.2 Geology.....	1-4
	1.2.3 Hydrogeology	1-5
	1.2.4 Project Specific Information	1-5
1.3	Project Description.....	1-6
	1.3.1 Site and Project Background.....	1-6
1.4	Data Quality Objectives For Environmental Data	1-6
	1.4.1 Data Categories.....	1-8
1.5	Documentation and Records	1-10
	1.5.1 Field Documentation.....	1-10
	1.5.2 Laboratory Documentation and Records	1-13
	1.5.3 Calculations.....	1-20
	1.5.4 Data Integrity and Treatment of Outliers.....	1-20
	1.5.5 Data Management	1-21
	1.5.6 Data Archive	1-21
2.0	MEASUREMENTS / DATA ACQUISITION.....	2-1
2.1	Sampling Process Design.....	2-1

2.2	Sampling Methods Requirements	2-1
2.3	Sampling Handling Procedures and Custody Requirements	2-2
2.3.1	Packing.....	2-2
2.3.2	Shipping	2-3
2.3.3	Sample Preservation and Holding Times.....	2-3
2.3.4	Laboratory Receipt and Entry of Samples	2-4
2.3.5	Pre-analysis Storage.....	2-5
2.3.6	Post-analysis Storage	2-5
2.4	Analytical Methods Requirements.....	2-5
2.4.1	Overview of Analytical Methods.....	2-5
2.4.2	Method Descriptions	2-9
2.4.3	Preventive Maintenance Program	2-22
2.4.4	Laboratory Data Reduction and Review.....	2-23
2.5.	Quality Control Requirements	2-26
2.5.1	Analytical Quality Control Requirements	2-26
2.5.2	Definition of Terms.....	2-27
2.5.3	Laboratory Batch Quality Control Logic.....	2-31
2.5.4	Laboratory Data Completeness.....	2-36
2.6.	Instrumentation Calibration and Frequency.....	2-37
2.6.1	Standards.....	2-37
2.6.2	Instrument Calibration	2-38
2.7.	Data Acquisition Requirements (Non-direct Measurement)	2-47
3.0	ASSESSMENTS / OVERSIGHT	3-1
3.1	Quality Control	3-1
3.1.1	Definable Features of Work.....	3-1
3.1.2	Preparatory Phase.....	3-2
3.1.3	Initial Phase.....	3-2
3.1.4	Follow-Up Phase.....	3-3
3.1.5	Completion Inspection	3-3
3.2.	Assessment and Response Actions	3-3
3.2.1	Readiness Reviews.....	3-3
3.2.2	System Audits	3-3
3.2.3	Surveillances	3-4
3.2.4	Performance Evaluation Samples / Data Tracking Audits	3-4
3.2.5	Nonconformance / Corrective Actions	3-5
3.3.	Reports to Management	3-5
3.3.1	Field Activities.....	3-5
3.3.2	Drilling Subcontractors.....	3-5
3.3.3	Subcontract Laboratories	3-5
4.0	DATA VALIDATION AND USABILITY	4-1
4.1	Data Review, Verification and Validation Requirements.....	4-1
4.1.1	Field Sampling / Non-analytical Data.....	4-1
4.1.2	Screening / Non-definitive Data	4-1
4.2.	Data Validation and Verification Methods	4-1

4.2.1	Data Verification.....	4-2
4.2.2	Data Validation	4-4
4.2.3	Data Usability	4-5
4.3.	Reconciliation with Data Quality Objectives	4-5
4.3.1	Analytical/Statistical Control Parameters	4-6

FIELD SAMPLING PLAN

1.0	INTRODUCTION	1-1
1.1	Sample Types.....	1-1
1.2.1	Trip Blanks.....	1-1
1.2.2	Quality Control (QC) Samples.....	1-1
1.2.3	Quality Assurance (QA) Samples	1-2
1.2.4	Rinsate Samples	1-2
1.2.5	Field Blanks	1-2
2.0	FIELD DOCUMENTATION	
2.1	Sample Information Documentation.....	2-1
2.2	Preparation of Field Logbooks.....	2-1
2.3	Photographs.....	2-1
3.0	SAMPLING EQUIPMENT AND PROCEDURES	3-1
3.1	Standard Operating Procedures.....	3-1
3.2	Drilling and Sampling Activities	3-1
3.2.3	Mud Rotary Drilling	3-2
3.2.4	Air Rotary Drilling.....	3-2
3.2.5	Dual Tube Percussion Drilling.....	3-3
3.2.6	Drilling and Development Equipment Decontamination	3-3
3.2.7	Lithologic Logging	3-4
3.2.8	Cone Penetrometer Test (CPT).....	3-4
3.2.9	Soil Organic Vapor Sampling.....	3-4
3.2.10	Hydropunch Sampling	3-4
3.3	Monitoring Well Installation and Development Procedures	3-5
3.3.1	Monitoring Well Installation.....	3-5
3.3.2	Filter Pack and Well Screen Slot Size Determinations.....	3-5
3.3.3	Monitoring Well Development.....	3-5
3.4	Borehole and Well Abandonment Procedures	3-5
3.4.1	Borehole Abandonment	3-5
3.4.2	Well Abandonment	3-6
3.5	Split-Spoon Sampling	3-6
3.6	Shallow Subsurface Sampling	3-6
3.7	Grab Sampling	3-6
3.8	Stockpile Soil Sampling.....	3-7
3.8.1	Engineering Controls For Stockpiled Soil	3-7
3.8.2	Stockpiled Soil Characterization.....	3-8
3.9	Groundwater Sampling	3-9

3.9.1	Definition of Terms.....	3-9
3.9.2	Well Sampling Procedure	3-10
3.10	Surface Water Sampling	3-12
3.10.1	Sampling for VOC Analysis	3-12
3.10.2	Sampling for Other Analyses.....	3-13
3.10.3	Deep Surface-water Sampling	3-13
3.11	Field Measurements	3-14
3.11.1	Water-Level Measurements	3-14
3.11.2	Analytical Measurements.....	3-15
3.11.3	Soil Organic Vapor Analyses.....	3-15
3.12	Decontamination Procedures	3-16
4.0	SAMPLE HANDLING PROCEDURES.....	4-1
4.1	Sample Containers	4-1
4.2	Sample Preservation.....	4-1
4.3	Sample Transportation	4-1
4.4	Chain of Custody Procedures.....	4-1

Appendix A

List of Figures

<u>Figure</u>	<u>Title</u>
1-1	Map of Tooele Army Depot

Appendix B

List of Tables

<u>Table</u>	<u>Title</u>
2-1	UFP-QAPP Worksheet Organization
2-2	Sample Containers, Preservation and Holding Time Requirements

Appendix C

Flagging Conventions

<u>Table</u>	<u>Title</u>
4-1	Flagging Conventions, Data Evaluation and Validation (Organic Methods)
4-2	Flagging Conventions, Data Evaluation and Validation (Inorganic Methods)

Appendix D

Standard Operating Procedures

This appendix includes standard operating procedures for use by field and administrative personnel represent and supplement the information presented in the CDQMP in a procedural format.

<u>SOP No.</u>	<u>Title</u>	<u>Rev.</u>
SOP 1.0	Quality Control Program	0
SOP 1.1	Chain of Custody	0
SOP 1.2	Field Activity Documentation	0
SOP 2.0	Sample Handling, Packaging and Shipping	0
SOP 2.1	Sample Labeling	0
SOP 2.2	Sample Numbering	0
SOP 2.3	On-Site Sample Storage	0
SOP 3.0	Surface and Shallow Subsurface Soil Sampling	0
SOP 3.1	Subsurface Soil Sampling While Drilling	0
SOP 3.2	Composite Sample Preparation	0
SOP 3.3	Duplicate and Split Sample Preparation	0
SOP 3.4	Surface Wipe Sampling	0
SOP 3.5	Chip/Core Sampling	0
SOP 4.0	Calibration and Maintenance of Measuring and Test Equipment	0
SOP 4.1	Field Instrument QA/QC	0
SOP 5.0	Water Level Measurements in Monitoring Wells	0
SOP 5.1	Nonaqueous Phase Liquid Measurement in Monitoring Wells	0
SOP 6.0	Sampling Equipment and Well Material Decontamination	0
SOP 6.1	Drilling and Heavy Equipment Decontamination	0
SOP 7.0	Compaction of Fill Material	0
SOP 7.1	Surface and Subsurface Geophysics	0
SOP 8.1	Monitoring Well Installation	0
SOP 8.2	Monitoring Well Development	0
SOP 9.0	Groundwater Sampling	0
SOP 9.2	Cone Penetration Testing and Hydropunch Groundwater Sampling	0
SOP 10.0	Lithologic Logging	0
SOP 11.0	Aquifer Testing	0
SOP 12.0	Soil Stockpiling	0
SOP 14.0	Hollow Stem Auger Drilling	0
SOP 15.0	Field QC Sampling	0
SOP 16.0	Management of Investigation-Derived Waste (IDW)	0
SOP 17.0	Preparation, Revision and Approval of Plans and Procedures	0
SOP 18.0	Quality Inspection and Inspection Report	0

Example Forms

Daily Quality Control Report

Chain of Custody

Cooler Receipt Form

Field Test Equipment Calibration Log

Field Boring Log

Monitoring Well Depth Measurement Log

Monitoring Well Purge and Sample Log

Appendix E

Electronic Data Deliverable Specification

Electronic Data Specification - This specification provides for a deliverable consistent with the latest Environmental Restoration Program Information Management System (ERPIMS) format. The *ERPIMS '98 Data Loading Handbook, Version 4.0* (October 1997) is incorporated by reference.

Appendix F

DoD QSM Appendix F and G SW846 Analytical Requirements

List of Acronyms

<i>ACS</i>	<i>American Chemical Society</i>
<i>ANSI</i>	<i>American National Standards Institute</i>
<i>AR/COC</i>	<i>Analysis Request/Chain of Custody Record</i>
<i>ARAR</i>	<i>Appropriate, Relevant, and Applicable Requirements</i>
<i>ASQC</i>	<i>American Society for Quality Control</i>
<i>ASTM</i>	<i>American Society of Testing and Materials</i>
<i>ATL</i>	<i>Audit Team Leader</i>
<i>BRAC</i>	<i>Base Realignment and Closure</i>
<i>BS/BSD</i>	<i>Blank Spike/Blank Spike Duplicate</i>
<i>BTEX</i>	<i>Benzene, Toluene, Ethylbenzene, and Xylene</i>
<i>CAE</i>	<i>Contractor Acquired Equipment</i>
<i>CAR</i>	<i>Corrective Action Requests</i>
<i>CDQAR</i>	<i>Chemical Data Quality Assessment Report</i>
<i>CDQMP</i>	<i>Chemical Data Quality Management Plan</i>
<i>CERCLA</i>	<i>Comprehensive Environmental Response Compensation and Liability Act</i>
<i>CIH</i>	<i>Certified Industrial Hygienist</i>
<i>CLP</i>	<i>EPA Contract Laboratory Program</i>
<i>CMD</i>	<i>Corrective Measures Design</i>
<i>CMS</i>	<i>Corrective Measures Study</i>
<i>COC</i>	<i>Chain-of-Custody</i>
<i>DERP</i>	<i>Defense Environmental Restoration Program</i>
<i>DOD</i>	<i>Department of Defense</i>
<i>DOE</i>	<i>U.S. Department of Energy</i>
<i>DOT</i>	<i>U.S. Department of Transportation</i>
<i>DQCR</i>	<i>Daily Quality Control Report</i>
<i>DQO</i>	<i>Data Quality Objective</i>
<i>DRO</i>	<i>Diesel Range Organics</i>
<i>EB</i>	<i>Equipment Blank</i>
<i>EE/CA</i>	<i>Engineering Evaluation/Cost Analysis</i>
<i>ELAP</i>	<i>Environmental Laboratory Accreditation Program</i>
<i>EM</i>	<i>Engineer Manual</i>
<i>EPA</i>	<i>United States Environmental Protection Agency</i>
<i>ER</i>	<i>Engineer Regulation</i>
<i>FADL</i>	<i>Field Activity Daily Log</i>
<i>FS</i>	<i>Feasibility Study</i>
<i>FSP</i>	<i>Field Sampling Plan</i>
<i>FUDS</i>	<i>Formerly Used Defense Sites</i>
<i>FWV</i>	<i>Field Work Variance</i>
<i>GFE</i>	<i>Government Furnished Equipment</i>
<i>gm</i>	<i>Gram</i>
<i>GRO</i>	<i>Gasoline Range Organics</i>
<i>H&S</i>	<i>Health and Safety</i>
<i>HTRW</i>	<i>Hazardous, Toxic, and Radioactive Waste</i>
<i>IATA</i>	<i>International Air Transportation Association</i>

<i>ID</i>	<i>Identification</i>
<i>IFB</i>	<i>Invitation for Bid</i>
<i>IRP</i>	<i>Installation Restoration Program</i>
<i>ISO</i>	<i>International Standards Organization</i>
<i>ISO</i>	<i>International Standards Organization</i>
<i>Kg</i>	<i>Kilogram(s)</i>
<i>L</i>	<i>Liter(s)</i>
<i>LCS/LCSD</i>	<i>Laboratory Control Sample / Laboratory Control Sample Duplicate</i>
<i>LQMM</i>	<i>Laboratory Quality Management Manual</i>
<i>LRL</i>	<i>Laboratory Reporting Limit</i>
<i>LUFT</i>	<i>Leaking Underground Fuel Tank</i>
<i>MB</i>	<i>Method Blank</i>
<i>MDL</i>	<i>Method Detection Limit</i>
<i>MFR</i>	<i>Memorandum for Record</i>
μg	<i>Microgram(s)</i>
μl	<i>Microliter(s)</i>
<i>MIPR</i>	<i>Military Interdepartmental Purchase Request</i>
<i>ml</i>	<i>Milliliter</i>
<i>MQL</i>	<i>Method Quantitation Limit</i>
<i>MS/MSD</i>	<i>Matrix Spike / Matrix Spike Duplicate</i>
<i>NCR</i>	<i>Nonconformance Report</i>
<i>NELAC</i>	<i>National Environmental Laboratory Accreditation Conference</i>
<i>NELAP</i>	<i>National Environmental Laboratory Accreditation Program</i>
<i>NEPA</i>	<i>National Environmental Policy Act</i>
<i>NPL</i>	<i>National Priorities List</i>
<i>NPL</i>	<i>Superfund National Priority List</i>
<i>O&M</i>	<i>Operation and Maintenance</i>
<i>OE</i>	<i>Ordnance and Explosives</i>
<i>OSHA</i>	<i>Occupational Safety and Health Administration</i>
<i>PA</i>	<i>Preliminary Assessment</i>
<i>PARCC</i>	<i>Precision, Accuracy, Representativeness, Completeness, and Comparability</i>
<i>PCBs</i>	<i>Polychlorinated biphenyls</i>
<i>PE</i>	<i>Performance Evaluation</i>
<i>PHSP</i>	<i>Program Health and Safety Plan</i>
<i>PM</i>	<i>Program Manager</i>
<i>PO</i>	<i>Purchase Order</i>
<i>POC</i>	<i>Point of Contact</i>
<i>ppb</i>	<i>Part(s) per Billion</i>
<i>ppm</i>	<i>Part(s) per Million</i>
<i>PRP</i>	<i>Principle Responsible Party</i>
<i>QA</i>	<i>Quality Assurance</i>
<i>QA/QCM</i>	<i>QA/QC Manager</i>
<i>QAP</i>	<i>Quality Assurance Plan</i>
<i>QAPP</i>	<i>Quality Assurance Project Plan</i>
<i>QC</i>	<i>Quality Control</i>
<i>QSM</i>	<i>Quality Systems Manual</i>

<i>RAC</i>	<i>Remedial Action Contract</i>
<i>RCRA</i>	<i>Resource Conservation and Recovery Act</i>
<i>RD</i>	<i>Remedial Design</i>
<i>RFA</i>	<i>RCRA Facility Assessment</i>
<i>RFI</i>	<i>RCRA Facility Investigation</i>
<i>RFP</i>	<i>Request for Proposal</i>
<i>RI</i>	<i>Remedial Investigation</i>
<i>ROD</i>	<i>Record of Decision</i>
<i>RPD</i>	<i>Relative Percent Difference</i>
<i>SAP</i>	<i>Sampling and Analysis Plan</i>
<i>SARA</i>	<i>Superfund Amendments and Reauthorization Act</i>
<i>SI</i>	<i>Site Inspection</i>
<i>SOP</i>	<i>Standard Operating Procedure</i>
<i>SOV</i>	<i>Soil Organic Vapor</i>
<i>SQP</i>	<i>Standard Quality Procedure</i>
<i>SSHP</i>	<i>Site Safety and Health Plan</i>
<i>SVOA</i>	<i>Semivolatile Organic Analysis</i>
<i>TDS</i>	<i>Total Dissolved Solids</i>
<i>TERC</i>	<i>Total Environmental Restoration Contract</i>
<i>TIC</i>	<i>Tentatively Identified Compound</i>
<i>TM</i>	<i>Technical Manager</i>
<i>TNI</i>	<i>The NELAC Institute</i>
<i>TPH</i>	<i>Total Petroleum Hydrocarbons</i>
<i>TRPH</i>	<i>Total Recoverable Petroleum Hydrocarbons</i>
<i>TSS</i>	<i>Total Suspended Solids</i>
<i>UFP-QAPP</i>	<i>Uniform Federal Policy for Quality Assurance Project Plans</i>
<i>U.S. Army</i>	<i>U.S. Department of the Army</i>
<i>USACE</i>	<i>U.S. Army Corps of Engineers</i>
<i>UST</i>	<i>Underground Storage Tank</i>
<i>VECP</i>	<i>Value Engineering Change Proposals</i>
<i>VOA</i>	<i>Volatile Organic Analysis</i>
<i>VOC</i>	<i>Volatile Organic Compound</i>
<i>°C</i>	<i>Degrees Celsius</i>

Terms and Definitions

Acceptance Criteria - Specified limits placed on characteristics of an item, process, or service defined in codes, standards, or other requirement documents.

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one.

Accrediting Authority: The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation. (NELAC).

Accuracy - The closeness of agreement between the measured value and the true value. Calculated as percent recovery.

Activities that Affect Quality - Activities that, if not performed properly, could compromise the validity of information or data reported, which could result in an unacceptable risk to the environment, health, or safety of the public or the workers involved, or could have a detrimental effect on the achievement of the project objectives.

Activity - An all-inclusive term describing a specific set of operations or related tasks to be performed, either serially or in parallel, that in total result in a product or service.

Assessment - An all-inclusive term used to denote any of the following: audit, performance evaluation, management systems review, peer review, or surveillance performed by or for management.

Audit - A planned and documented activity performed to determine by investigation, examination, or evaluation of objective evidence the adequacy of and compliance with established procedures, instructions, drawings, and other applicable documents, the effectiveness of implementation and whether the results are suitable to achieve objectives. An audit should not be confused with surveillance or inspection activities performed for the sole purpose of process control or product acceptance.

Audit Team - One or more persons who are responsible for audit performance and reporting. The team may consist of, or is headed by, an individual designated as the Audit Team Leader.

Audit Team Leader - The individual responsible who organizes and directs the audit, coordinates the preparation and issuance of the Audit Report, and evaluates the responses.

Bias - The systemic or persistent distortion of a measurement process which causes errors in one direction.

CDQMP - A document that describes the management system for planning, performing, and assessing work to ensure that the results demonstrate stated quality, technical, and performance objectives. The CDQMP will describe the organizational structure, QC policies and procedures,

functional responsibilities, levels of accountability and authority, and necessary interfaces for organizations performing activities in support of the program management office.

Chain-of-custody - *An unbroken trail of accountability that ensures the physical security of samples, data, and records.*

Characteristic - *Any property or attribute of a datum, item, process, or service that is distinct, describable and/or measurable.*

Comparability - *A qualitative characteristic which defines the extent to which a chemical parameter measurement is consistent with, and may be compared to, values from other sampling events.*

Completeness - *A quantitative evaluation of what percent of the chemical measurements met the project data quality objectives.*

Conformance - *An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation.*

Controlled Documents - *Documents which have been assigned a unique identifier and issued to a specific person, discipline, or facility. These documents are maintained current by accounting for their initial issue and revisions.*

Corrective Action - *Measures taken to rectify conditions adverse to quality and, where possible, to preclude their recurrence.*

Data Quality Objectives - *Qualitative and quantitative statements that clarify technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed for support decisions.*

Data Quality Assessment - *A statistical and scientific evaluation of the data set to determine the validity and performance of the data collection design and statistical test, and the adequacy of the data set for its intended use.*

Data Useability Review - *The process of ensuring or determining whether the quality of the data produced meets the intended use of the data.*

Data of Known Quality - *Data that have the qualitative and quantitative components associated with their derivation documented appropriately for their intended use, and such documentation is verifiable and defensible.*

Data Verification - *The process for evaluating the completeness, correctness, consistency, and compliance of a data package against a standard or contract.*

Data Validation - *The process of data assessment in accordance with EPA regional or national functional guidelines or project-specific guidelines. Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)*

Data Assessment - The all-inclusive process used to measure the effectiveness of a particular data gathering activity. This process may be comprised of data verification, data review, data evaluation, and data validation.

Data Evaluation - The process of data assessment done by the district project chemist to produce a chemical data quality assessment report.

Deficiency - An unauthorized deviation from approved procedures or practices, or a defect in an item.

Definitive Data - Data that are generated using rigorous, analyte-specific analytical methods where analyte identifications and quantitations are confirmed and QA/QC requirements are satisfied.

Design Review - A documented evaluation by a team, including personnel such as the responsible designers, the client for the work or product being designed, and a QA representative, but other than the original designers, to determine if a proposed design will meet the established design criteria and perform as expected when implemented.

Detection Limit (DL) Per the DOD QSM, the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

Document - Any written or pictorial information describing; defining; specifying; reporting; or certifying activities, requirements, procedures, or results.

Duplicate Sample - A sample replicate collected as near as possible at an identical time and place as an original sample. Sometimes used in place of a split sample for volatile analytes, or to assess overall sample matrix homogeneity (see also split sample).

Entity - Something which can be individually described and considered, such as a process, product, item, organization, or combination thereof.

External Audit - An audit of those portions of another organization's QA program not under the direct control or within the organizational structure of the auditing organization.

Field Work Variance - Documented authorization from the Contracting Officer to depart from specified requirements.

Finding - A document statement of fact concerning a noncompliance or deviation from established requirements.

HTRW Activities - Activities undertaken for the U.S. EPA's Superfund Program, the Defense Environmental Restoration Program (DERP), including Formerly Used Defense Sites (FUDS) and Installation Restoration Program (IRP) sites at active DOD facilities, HTRW actions associated with Civil Works projects, and any other mission or non-mission work performed for others at HTRW sites. Such activities include, but are not limited to, Preliminary Assessments/Site Inspections (PA/SI), Remedial Investigations (RI), Feasibility Studies (FS),

Engineering Evaluation/Cost Analyses (EE/CA), RCRA Facility Investigations/Corrective Measures Studies/Corrective Measures Implementation/Closure Plans/Part B Permits, or any other investigations, design activities, or remedial construction at known, suspected, or potential HTRW sites. HTRW activities also include those conducted at petroleum tank sites and construction sites containing HTRW.

Independent Assessment - *An assessment performed by a qualified individual, group, or organization that is not a part of the organization directly performing and accountable for the work being assessed.*

Inspection - *Examination or measurement of an item or activity to verify conformance to specific requirements.*

Inspector - *A person who performs inspection activities to verify conformance to specific requirements.*

Internal Audit - *An audit of those portions of an organization's QA/QC program retained under its direct control and within its organizational structure.*

Item - *An all-inclusive term used in place of any of the following: appurtenance, facility, sample, assembly, component, equipment, material, module, part, structure, subassembly, subsystem, system, unit, documented concepts, or data.*

Limit of Detection (LOD) - *Is the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a 99% confidence level. In other words, if a sample has a true concentration at the LOD, there is a minimum probability of 99% of reporting a "detection" (a measured value $\geq DL$) and a 1% chance of reporting a non-detect (a false negative).*

The Limit of Quantitation (LOQ) - *Is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ is typically larger than the LOD (but may be equal to the LOD, depending upon the acceptance limits for precision and bias); therefore, the following is true:*

$$DL < LOD \leq LOQ$$

Quantitative results can only be achieved at or above the LOQ. Measurements between the DL and the LOQ assure the presence of the analyte with confidence, but their numeric values are estimates.

Management System - *A structured non-technical system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for conducting work and for producing items and services.*

Management - *Those individuals directly responsible and accountable for planning, implementing, and assessing work.*

Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured within a given matrix and reported with 99% confidence that the analyte concentration is greater than zero (40 CFR 136 App. B). The MDL is obtained by seven replicate analyses of the matrix for the analyte under investigation at a concentration level which is two to five times the estimated MDL. The MDL is defined as three times the standard deviation of the replicate sample results.

Method - A body of procedures and techniques for performing an activity systematically presented in the order in which they are to be executed.

Nonconformance (NCR) - A deficiency in characteristic documentation or procedure which renders the quality of an item unacceptable or indeterminate with respect to project criteria. Examples of nonconformances include, but are not limited to test failures, physical defects, incorrect or inadequate documentation, data losses, or deviation from prescribed processing, inspection, or procedure.

Objective Evidence - Any documented statement of fact, other information, or record, either quantitative or qualitative, pertaining to the quality of an item or activity that is based on observations, measurements, or tests that can be verified.

Observation - A statement of fact regarding the potential for a noncompliance which could lead to a more serious problem if not identified and/or corrected, but which does not constitute a lack of compliance with established requirements.

Ordinance and Explosives (OE) Activities. - All work undertaken to manage or eliminate the immediate risks associated with OE related material. OE activities are usually response activities undertaken for DERP, FUDS, or Base Realignment and Closure (BRAC) projects. OE responses include site inventories, preliminary assessments, site investigations, public involvement, engineering estimates, cost analyses, action memoranda, removal designs, removals (both time critical & non-time critical), and clean-up of residual OE.

Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions, expressed generally in terms of standard deviation.

Preparatory Inspection - A systematic, documented review of the readiness for startup or continued extended use of a facility, process, or activity. Preparatory inspections are typically conducted before proceeding beyond project milestones and prior to institution of a major phase of work activities.

Primary Laboratory - Laboratory that analyzes the majority of the project samples.

Procedure - A document that specifies or describes how an activity is to be performed.

Process - A set of interrelated resources and activities which transforms inputs into outputs.

Procurement Document - Purchase requisitions, purchase orders, drawings, contracts, specifications, or instructions used to define requirements for purchase.

Program Manager - The organizational manager having direct responsibility for administration and direction of the Contract.

Project Manager - The leader of the project team, responsible for managing the project parameters (budget, cost, safety, schedule, scope and quality), as well as interfacing with those involved in the project process (customers, functional elements, government, and non-government entities).

Project - An organized set of activities within a program.

Qualification (Personnel) - The characteristics or abilities gained through education, training, and/or experience, as measured against established requirements, such as standards, tests and/or evaluation that qualify a person to perform a required function.

Quality Assurance - An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement that measures the degree of excellence of environmental data and communicates the information to a data generator or data user in a convincing manner.

Quality Assurance Laboratory - The USACE HTRW chemistry laboratory, or its subcontracted agent that is responsible for analysis of the project QA samples.

Quality - The degree to which an item or process meets or exceeds the user's requirements and expectations.

Quality Indicators - Measurable attributes of the attainment of the necessary quality for a particular environmental decision. Indicators of data quality include precision, bias, completeness, representativeness, reproducibility, comparability, sensitivity, and statistical confidence.

Quality Assurance Sample - A sample collected to monitor the quality of sampling operations. This type of sample is analyzed by the quality assurance laboratory and typically includes split samples, duplicate samples, and various types of blank samples.

Quality Assurance (QA) - All of those planned and systematic actions necessary to provide confidence that a structure, system, or component will perform satisfactorily in service. When the product is a report of a significant study or investigation, QA also comprises those planned and systematic actions necessary to provide adequate confidence in the validity and integrity of the reported data, methods, and procedures and in the protection, retrievability, and replicability of the data. The quality management system includes a multidisciplinary system of management controls backed by quality verification and overview activities that demonstrate completeness and appropriateness of achieved quality.

Quality Assurance Documents - Those documents which establish the requirements and methods to implement the client activities. These documents are identified as the Work Plan, Sampling and Analysis Plan, Contractor Quality Control Plan, Standard Quality Procedures, Standard Operating Procedures, and Field Work Variances.

Quality Control Program - The overall program established by an organization to implement the requirements of the contract document. The program assigns responsibilities and authorities, defines policies and requirements, and provides for the performance and assessment of work. The QC program is described in the CDQMP.

Quality Control Record - A completed document that furnishes evidence of the quality of items and/or activities affecting quality.

Quality Control - The overall system of technical activities that monitors the degree of excellence of environmental data so that the stated requirements of defined standards are achieved.

Quality Control Sample - A sample collected to monitor and control the quality of sampling operations. This type of sample is analyzed by the primary laboratory and typically includes split samples, duplicate samples, and various types of blank samples.

Reporting Limit (RL) - The RL is a project-specific reporting limit based on a regulatory action level, a risk-based screening level, or documented data quality objective. As defined in the DOD QSM, the RL is the lowest concentration value that meets project requirements for reporting quantitative data with known precision and bias for a specific analyte in a specific matrix. The project specific RLs are established to support the DQOs for collection of the data.
Representativeness - A measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process, or an environmental condition.

Reproducibility - The precision, usually expressed as variance, that measures the variability among the results of measurements of a sample at different laboratories.

Sample Reporting Limit (SRL) - The Sample Reporting Limit (SRL) is the RL adjusted for the size of the sample aliquot analyzed, any dilution/concentration factors unique to the analysis of a particular sample, and any allowances made for the sample matrix which might elevate the normal RL (i.e., moisture content of a soil or sediment).

Screening Level Data - Data that are generated by less precise methods of analysis, less rigorous sample preparation, and less stringent QA/QC procedures. The data generated provide analyte identification and quantification, although the quantification may be relatively imprecise.

Significant Deficiency - Any state, status, incident, or situation of an environmental process or condition, or environmental technology in which the work being performed will be adversely affected sufficiently to require corrective action to satisfy quality objectives or specifications and safety requirements.

Significant Condition Adverse to Quality - A condition that, if left uncorrected, could have a serious effect on safety or operability. This term includes environmental and program compliance.

Split sample - A sample which has been collected, homogenized, and divided into two or more

portions for analysis by multiple laboratories. Applicable for all test parameters except those involving volatile analytes where homogenization might affect the concentration of volatile substances (see also duplicate sample).

Standard Operating Procedure (SOP) - A written document that details the process for an operation, analysis, or action, with thoroughly prescribed techniques and steps, and that is officially approved as the method for performing certain routine or repetitive tasks.

Stop Work Order - The order to stop further processing, delivery, installation, or operation until proper disposition of a nonconformance, deficiency, or unsatisfactory condition has occurred.

Supplier - Any individual or organization that furnishes items or services in accordance with a procurement document. An all-inclusive term used in place of any of the following: vendor, seller, contractor, subcontractor, fabricator, consultant, and their subtier levels.

Surveillance - The act of monitoring or observing to verify whether an item or activity conforms to specified requirements.

Technical Systems Audit - A thorough, systematic, on-site, qualitative audit of facilities, equipment, personnel, training, procedures, record keeping, data verification/ validation, data management, and reporting aspects of a system.

Technical Review - A documented critical review of work that has been performed within the state of the art. The review is accomplished by one or more qualified reviewers who are independent of those who performed the work, but are collectively equivalent in technical expertise to those who performed the original work. The review is an in-depth analysis and evaluation of documents, activities, material, data, or items that require technical verification or validation for applicability, correctness, adequacy, completeness, and assurance that established requirements are satisfied.

Technical Manager - The leader of the technical process, responsible for the content and quality of technical products.

Technical Specialist - One or more persons who are assigned to the audit team due to the specialized or technical aspects of the areas to be audited. Technical Specialists are selected based on their special abilities, specialized technical training, and/or prior experience in the specialized or technical aspects of the area to be audited.

Traceability - The ability to trace the history, application, or location of an entity by means of recorded identifications. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for quality for the project.

Training - To impart specific information with regard to job functions which will achieve initial proficiency, maintain proficiency and adapt to changes in technology, methods or job functions.

Uncontrolled Document - A document which is issued current but which is not maintained current with revisions.

Use-As-Is - A disposition permitted for a nonconforming item when it can be established that the item is satisfactory for its intended use.

**CHEMICAL DATA QUALITY MANAGEMENT
PLAN
TOOELE, UTAH**

**Submitted to:
Tooele Army Depot
Environmental Management Office
Tooele, UT**

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Approved by: _____

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CONTENTS

SECTION 1: QUALITY ASSURANCE PROJECT PLAN

SECTION 2: FIELD SAMPLING PLAN

**SECTION 3: GUIDANCE FOR THE PREPARATION OF SITE
SPECIFIC SAMPLING AND ANALYSIS PLANS
UFP-QAPP FORMAT**

QUALITY ASSURANCE PROJECT PLAN

EXECUTIVE SUMMARY

This Chemical Data Quality Management Plan (CDQMP) delineates the procedures that will be used to accomplish the chemical quality control items to assure accurate, precise, representative, complete, legally defensible and comparable data. The CDQMP presents functions, procedures, and specific quality assurance (QA) and quality control (QC) activities designed to achieve the data quality goals set for investigations at Tooele Army Depot. The CDQMP is composed of the Quality Assurance Project (QAPP) Plan, the Field Sampling Plan (FSP), and the Sampling and Analysis Plan (SAP). This CDQMP incorporates by reference the requirements of the following publications:

Environmental Protection Agency (EPA):

- EPA SW-846 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, Third Edition, as updated by Updates I, II, IIA, IIB, III, IIIA, IIIB, IVA and IVB, Revision 6, February 2007;
<http://www.epa.gov/epaoswer/hazwaste/test/main.htm>
- EPA QA/G-4 *Guidance on Systematic Planning Using the Data Quality Objectives Process*, EPA/240/B-06/001, February 2006

Department of Defense (DoD):

- DoD: DTIC ADA 427785, EPA-505-B-04-900A
Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual, Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs, Final, Version 1, March 2005;
<http://www.epa.gov/fedfac/documents/qualityassurance.htm>
- DoD: DTIC ADA 426957, EPA-505-B-04-900B
Uniform Federal Policy for Quality Assurance Project Plans, Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities, Final, Version 1, March 2005;
<http://www.epa.gov/fedfac/documents/qualityassurance.htm>
- DoD QSM *Department of Defense Quality Systems Manual For Environmental Laboratories*, Version 4.1 Final, June 2009;
<http://chppm-www.apgea.army.mil/dls/DoDV3.pdf>

U.S. Army Corps of Engineers (USACE):

EM 200-1-10

Guidance for Evaluating Performance-Based Chemical Data, 30
June 2005; [http://www.usace.army.mil/inet/usace-docs/eng-
manuals/em200-1-10/toc.htm](http://www.usace.army.mil/inet/usace-docs/eng-manuals/em200-1-10/toc.htm)

1.0 PROGRAM MANAGEMENT

1.1 Program and Project Organization

This section details the program and project organizations of personnel expected to perform work under this CDQMP. Program personnel and their respective responsibilities are clearly delineated. Project personnel will be identified in the project specific Sampling and Analysis Plans / UFP-QAPP which will clearly identify the specific personnel that are managing or performing tasks on each project. The lines of authority and communication will be clearly delineated on a project specific organizational chart and responsibilities of key personnel will be clearly defined.

1.1.1 Program Manager

The Program Manager (PM) will be identified. The PM will be fully responsible and accountable for all program and contractual activities. He will serve as the focal point and main channel of communication between the TEAD and the contractor's team. Using the Program staff, he will establish and interpret program policies, monitor schedule and cost, coordinate all reporting, ensure that necessary resources are made available, prepare long-range program plans, identify and resolve potential problems or conflicts, and provide for safe performance and quality of the work. He will also be responsible for leading the public relations effort in support of TEAD's public outreach program. Other duties, as appropriate, will include:

- Procurement, along with procurement personnel, and supervision of Program subcontractors
- Receive, negotiate, and track the performance of projects
- Assign Technical Managers and Project Leaders to direct specific projects and provide the necessary resources to these managers
- Approve and consistently implement the program planning documents (e.g., this document, Program Management Plan, Health and Safety Plan, etc.)
- Assess the overall Program for compliance with federal, state, and local regulations/laws and with specific delivery orders and directives
- Interact with regulatory/public agency clients at the request of the client
- Disseminate Program-related information from the client and others

- Provide Program change order control
- Report any significant conditions adverse to quality and obtain concurrence on proposed resolution(s)
- Provide overall Program technical, quality, and performance consistency
- Attend meetings and conferences between USACE and TEAD as appropriate
- Review Program quality assurance audit reports and any resulting corrective action disposition.

1.1.2 Quality Assurance Officer

The Quality Assurance Officer (QAO) will be identified and will be responsible for overseeing that Quality Control (QC) operations are executed for all field and laboratory activities. Day to day monitoring of QC functions will be designated to the appropriate staff personnel (i.e., Technical Manager, Project Manager). The Quality Assurance Officer will verify compliance with work plans and procedures by providing for periodic field audits, laboratory audits, and review of work plans, reports, and laboratory data. The Quality Assurance Officer will report to the Project Manager.

1.1.3 Technical Manager

The Technical Manager (TM) will be identified. The TM will be responsible for reviewing the sampling program and associated field activities, ensuring that all sampling activities conform to the SAP. The TM will provide technical support throughout the program and will provide review of all technical documents submitted to the client. The TM reports to the PM.

1.1.4 Project Manager

Quality assurance of field activities will be overseen by the Project Manager, who will be in the field to supervise and perform initial inspections of field activities. Prior to the start of field activities, preparatory meetings will be held with the field crew. Checklists will be used during field activities. If field conditions require modifications to protocol outlined in the CDQMP or if questions arise, the field crew will contact the Project Manager for direction. The Project Manager will be also be responsible for overseeing review of the project CDQMP program as it relates to the compilation of data. The Project Manager reports to the TM.

1.1.5 Project Chemist

The Project Chemist will be identified. The Project Chemist will have a “hands on” role in management of project tasks associated with sampling and analysis including instruction of field personnel in sampling and preservation requirements and general oversight of field personnel involved in sampling activities. The Project Chemist will assist the project team in selecting the analytical laboratory and developing the project specific sampling and analysis plan (SAP). The Project Chemist will provide coordination with the analytical laboratory to insure readiness to implement project specific requirements, review of analytical data as it becomes available to insure conformance with quality standards, implementation of corrective actions in accordance with CDQMP and SAP specifications when review of data uncovers deficiencies, and serve as a point of contact for the Army appointed Chemist for issues related to environmental chemistry. The Project Chemist will oversee on-site analytical testing including field screening analyses. The Chemist will also prepare all data validation reports or review for accuracy all data validation reports prepared by subcontractors. The Project Chemist will report to the Project Manager.

1.1.6 Program Geologist

The Program Geologist will be identified. The Program Geologist will be responsible for design and internal review of all aspects of work related to Geology such as drilling program design and execution, monitoring well design and installation, preparation of boring logs, and groundwater modeling as directed by the Project Manager.

1.1.7 Health and Safety Officer

The Health and Safety Officer (HSO) will be identified. The HSO will be an experienced Industrial Hygienist. The HSO is responsible for the general health and safety plan development and training for field personnel. This individual is also responsible for ensuring that health and safety procedures are understood and followed by all field personnel, and for reporting and correcting any violations of policy or regulation.

1.1.8 Sampling Team Leader

The Sampling Team Leader will be responsible for implementing and overseeing field activities, data compilation, review of the project QA/QC program, and preparation of all technical documents. The Sampling Team Leader will also be responsible for quality assurance of field activities as described above and for executing all work elements related to the sampling program, including documenting field activities, maintaining field notes and photographs, maintaining a record of onsite personnel and visitors, and implementing the sampling plan. The Sampling Team Leader will be identified in the FSP and SAP. The Sampling Team Leader reports to the Project Manager.

1.1.9 Field Personnel

Field personnel will be responsible for performance of project mobilization, demobilization, sample collection and oversight. Field personnel will be identified in the FSP and SAP. Field personnel will report to the Sampling Team Leader.

1.2 Problem Definition/Background

This section provides a general background discussion of site history, geology, and hydrogeology for the Tooele Army Depot area. A detailed discussion of site specific information will be included in project specific SAP's and FSP's.

1.2.1 Location and History

TEAD is 7 miles south of the Great Salt Lake and 35 miles southwest of Salt Lake City. It is separated from Salt Lake City by the Oquirrh Mountains. TEAD is located in Tooele Valley, in the central portion of northern Utah, west of the town of Tooele and south of Grantsville and Erda. The valley is a northward plunging structural basin flanked by coalescing alluvial fans that slope generally to the north. TEAD began operating in 1942 as one of the major ammunition storage and equipment maintenance installations in the continental United States. The primary missions included administration of the TEAD complex; repair and maintenance of tactical wheeled vehicles and power generation equipment; and storage, maintenance, issuance, and disposal of conventional munitions. Upholding TEAD's mission necessitated that TEAD be engaged in a wide variety of operations which involved the use of materials with toxic and hazardous properties. Hazardous wastes were generated as a result of these activities. Materials associated with the industrial waste lagoon (IWL) and other solid waste management units (SWMU) activities at TEAD include the following general categories of compounds:

- petroleum wastes
- organic solvents
- metal dusts and fumes
- plating wastes
- pesticides (herbicides and insecticides)
- explosives
- paint wastes
- strong acids and bases
- coolants
- rubber wastes

1.2.2 Geology

Tooele Valley is typical of basin and range physiography in which fault block mountains rise above flat, intermountain valleys filled with unconsolidated sediments of Tertiary and

Quaternary age. The unconsolidated sediments beneath TEAD consist of alluvial outwash materials and lacustrine deposits whose thickness ranges from zero at bedrock outcrops out to over 1,500 ft north of the IWL. The bedrock outcrop is a surface expression of a large bedrock block that dips to the south in a series of terraces. The northern most terrace is estimated to exist at a depth of over 200 feet below ground surface (bgs) in the vicinity of the IWL.(Geomatrix,1997)

1.2.3 Hydrogeology

Groundwater in the unconsolidated sediments and bedrock at TEAD is generally unconfined. The alluvial and bedrock aquifers are recharged by subsurface seepage along the Oquirrh Mountains east of TEAD, by upward flow from deeper confined aquifers, percolating precipitation, and minor subsurface flow from adjacent areas. Depth to groundwater at TEAD ranges from about 200 feet to 400 feet bgs. Groundwater flows from south to north and toward the center of Tooele Valley.

Subsurface information collected at TEAD indicates that the alluvial aquifer consists of poorly sorted, poorly rounded, silty sand, gravel, and cobbles with occasional layers of clay, sand and gravel to approximately 5 feet thick. The sand grains, gravels, and cobbles are composed of limestone and quartzite eroded from the Oquirrh Mountains. The alluvial aquifer is relatively uniform throughout TEAD. Hydraulic conductivity values of the alluvial aquifer range from approximately 10 ft/day (ft/d) to 100 f/d in shallow wells at TEAD, indicating a relatively uniform coarse-grained aquifer. Results from other alluvium wells indicate that the properties of the alluvial aquifer vary with depth, with the hydraulic conductivity values ranging from approximately 0.1 ft/d to greater than 140 ft/d. This variation could be due in part to the presence of the bedrock occlusion located approximately 1,000 ft north of the IWL (Geomatrix, 1997 / Kleinfelder, 1997).

1.2.4 Project Specific Information

A detailed description of the problem definition from the DQO process and pertinent background information will be included in project-specific SAPs, as described below.

A narrative describing the project and specific problems to be solved or decisions to be made will be included in this section of the SAP. The goal of the environmental remedial activities will be clearly stated. A description of the work site including an area map, location map, and site map, site history as it relates to the current work, and any unusual conditions will be included, as applicable. The text will include diagrams detailing areas to be sampled as relevant to the definition of the project goals. These sections will also contain a summary of site geology/hydrogeology, as known based on previous site activities. The discussion will include enough information about the problem, the past history, any previous work or data, the regulatory or legal context, and any relevant ARARs to present a clear description of the project

objectives.

1.3 Project Description

A detailed narrative of the project description will be included in the project-specific SAP's, using text and applicable UFP-QAPP work sheets (WSs) as described below.

1.3.1 Site and Project Background

This section provides the description of the project to be performed in response to the preceding problem definition. A detailed description of the project sampling strategy will be discussed, including anticipated project start and completion dates in the SAP. As a minimum, this section of the SAP will include a brief discussion of the following:

- Expected measurements and anticipated approaches
- Applicable requirements, standards, or specifications to meet Program technical, regulatory, or quality objectives
- Special project requirements for items or services
- Assessment activity to be used to evaluate Program compliance
- Project schedule with milestones.

1.4 Data Quality Objectives

The SAP will describe the general scope of work and background information as it relates to the acquisition of geological, geophysical, hydrogeological and chemical data. The text will explicitly describe the data that are needed to meet the objectives of the project, how that data will be used, and discuss implementation of control mechanisms and standards that will be used to obtain data of sufficient quality to meet or exceed project objectives. The discussion of Data Quality Objectives (DQO's) will follow the guidance contained in the EPA document Guidance for the Data Quality Objectives Process, EPA QA/G-4, Final, February 2006 and the requirements of this document are included by reference. Work performed by an on-site laboratory will be required to meet the same standards as a fixed site laboratory as described in this scope of work. The SAP will also describe in quantitative terms the sensitivity, precision, accuracy, and completeness goals for each major measurement parameter and for each matrix to be sampled. The SAP may need to define different types of sensitivity (e.g. quantitative, qualitative, screening) for each major measurement parameter. A qualitative discussion will be presented regarding representativeness and comparability. The section on DQO's will address the following topics in the specified order.

- ***Statement of the Problem***
Summarize the problem that requires environmental data acquisition and identify the resources available to resolve the problem. The type of information obtained for each

site in step number one of the DQO process includes:

- a. types of contaminants that were suspected at each site;
- b. types of pathways and receptors present;
- c. types of disposal sites present; and
- d. types of contaminated media.

▪ ***Identification of Decisions***

- a. Identify the decision that requires acquisition of environmental data to address the problem. Identify the intended uses of data projected to be acquired. Data uses will be prioritized. The output for this DQO step includes:
 - b. expected decisions based on the data collected; and
 - c. types of actions that will be taken to determine these decisions.

▪ ***Identify Inputs to Decisions***

- a. Identify the information needed to support the decision and specify the inputs requiring environmental measurements. The output for this DQO step may include:
 - b. lists of all the data need to accomplish the objectives, including data that already exists and data that must be collected; and
 - c. identification of methods for establishing the action levels (e.g. regulatory threshold, risk or exposure assessment, technological limits etc.)

▪ ***Definition of Study Boundaries***

Specify the spatial and temporal aspects of the environmental media that the data must represent to support the decision. The output for this DQO step may include:

- a. definition of site boundaries;
- b. definition of boundaries for individual suspected contaminant source areas within a site;
- c. density of sampling;
- d. types of sampling or investigation constraints; and
- e. actions that will be taken if investigation constraints are encountered.

▪ ***Development of Decision Rules***

Develop a logical statement that defines the conditions that would cause the decision maker to choose among alternative actions.

▪ ***Specification of Limits on Decision Errors***

Specify the decision maker's acceptable limits on decision errors, which are used to establish appropriate performance goals for limiting uncertainty in environmental data.

- ***Optimization of Investigation Design for Obtaining Data***

Identify the most resource effective sampling and analysis design for generating data that are expected to satisfy project DQO's.

Project specific DQOs will be defined quantitatively as applicable. Identification of decisions and descriptions of data use will be described with text and supported with tables and lists that describe the following:

- Data needed. Measurement parameters, compounds and sample matrices;
- The action level or standards upon which decisions will be made, including the method detection limits and practical quantitation limits for relevant parameters;
- The summary statistics which specify the form the data will be in when compared against action levels or standards; and
- The acceptable level of confidence in the data needed for the stated purpose; or the acceptable limits of uncertainty.

The text will describe in quantitative terms the sensitivity, precision, accuracy, and completeness goals for each major measurement parameter and for each matrix to be sampled. The SAP may need to define different types of sensitivity (e.g. quantitative, qualitative, screening) for each major measurement parameter as applicable. A qualitative discussion will be presented regarding representativeness and comparability.

1.4.1 Data Categories

To assist in the interpretation of data for TEAD the following descriptive data categories will be implemented:

- Screening data
- Screening data with definitive confirmation
- Definitive data.

These three data categories are associated with specific QA/QC elements, and may be generated using a wide range of analytical methods. The particular type of data to be generated depends on the qualitative and quantitative DQOs developed during application of the DQO Process.

1.4.1.1 Screening Data

Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and clean-up. Screening data provide

analyte identification and quantification, although the quantification may be relatively imprecise. Screening data without associated confirmation data are not considered to be of known quality.

Screening Data QA/QC Elements

- Sample documentation (location, date and time collected, batch etc.);
- Chain of Custody (when appropriate);
- Sampling design approach (systematic, simple or stratified random, judgmental, etc.);
- Initial and continuing calibration
- Determination and documentation of detection limits;
- Analyte(s) identification;
- Analyte(s) quantification;
- Analytical error determination: An appropriate number of replicate aliquots as specified in the QAPP, are taken from at least one thoroughly homogenized sample, the replicate aliquots are analyzed and the standard laboratory QC parameters (such as variance, mean and coefficient of variation) are compared to method-specific performance requirements specified in Section 2.4.

1.4.1.2 Screening Data with Definitive Confirmation

Definitive confirmation of screening data provide for data of known quality and reduces the level of uncertainty of the data set. At least 10% of the screening data are confirmed by using EPA approved analytical methods and QA/QC procedures consistent with the requirements for definitive data described below.

Definitive confirmation

As a minimum, at least three screening samples reported above the action level (if any) and three screening samples reported below the action level (or as non-detects) should be randomly selected from the appropriate group and confirmed. At least ten percent of the screening data must be confirmed with definitive data as described below.

1.4.1.3 Definitive Data

Definitive data are generated using rigorous analytical methods, such as EPA approved reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total error must be determined. Definitive data may be obtained from laboratory data packages which incorporate the following QA/QC elements.

Definitive Data QA/QC Elements

- Sample documentation (location, date and time collected, batch etc..)
- Chain of Custody (when appropriate)
- Sampling design approach (systematic, simple or stratified random, judgmental, etc.);

- Initial and continuing calibration
- Determination and documentation of detection limits
- Analyte(s) identification
- Analyte(s) quantification
- QC blanks (trip, method, rinsate)
- Matrix spike recoveries
- Performance Evaluation (PE) Samples (when specified)
- Analytical error determination: An appropriate number of replicate aliquots as specified in the QAPP, are taken from at least one thoroughly homogenized sample, the replicate aliquots are analyzed and the standard laboratory QC parameters (such as variance, mean and coefficient of variation) are compared to method-specific performance requirements specified in Section 2.4.
- Total measurement error determination (measures overall precision of measurement system, from sample acquisition through analysis): An appropriate number of co-located samples as determined by the SAP are independently collected from the same location and analyzed following standard operating procedures. Based on these analytical results, standard laboratory parameters such as variance, mean, and coefficient of variation should be calculated and compared to established measurement error goals. This procedure may be required for each matrix under investigation, and may be repeated at more than one location at the site.

1.5 Documentation and Records

The following sections itemize the information and records which will be maintained for all projects covered by this CDQMP at TEAD.

1.5.1 Field Documentation

1.5.1.1 Field Log Books

A field notebook bound with serially-numbered pages will be used to record sample identification numbers, chain-of-custody numbers, and any significant observations or events. The project name, project number, site location, sampling event, project manager, telephone number and address of contractor office (should the book be misplaced or lost) will be listed in ink. The field notebook is intended to record events during sampling activities in sufficient detail to allow field personnel to reconstruct events that transpired during the project. The field notebook will be maintained by the Project Leader, who will sign and date the notebook prior to initiation of fieldwork. Detailed procedures for Field Activity and Documentation are presented in SOP 1.2.

If it is necessary to transfer the logbook to alternative personnel during the course of field work, the person relinquishing the logbook will sign and date the logbook at the time the logbook is transferred and the person receiving the logbook will do likewise. Corrections to erroneous data will be made by crossing a line through the entry and entering the correct information. The correction will be initialed and dated by the person making the entry. Unused portions of

logbook pages will be crossed out, signed, and dated at the end of each workday. Logbook entries must be dated, legible, in ink, and contain accurate documentation. Language used will be objective, factual, and free of personal opinions. Hypotheses for observed phenomena may be recorded, however, they must be clearly indicated as such and only relate to the subject observation.

The date and time of sampling preparation and collection, and personnel who conducted sampling are recorded with the sample identification number in the field log book and on the chain-of-custody form. The names of visitors and any other persons on site are also recorded in the field log book. Sampling personnel will also record the ambient weather conditions and other conditions at the sampling location that may affect sample collection, the apparent representativeness of the sample, or sample analysis in the field log book.

1.5.1.2 Photographs

Photographs will be used to supplement written descriptions of field activities, such as sampling. Photographs will be completely documented to include the project name and number, date of the photograph, weather conditions, the photographer, subject and a brief description of the purpose of the photograph. Photographs should be uniquely identified by photo number and traceable to negatives.

1.5.1.3 Chain of Custody Records

The specific sampling location of each sample is recorded with each sample identification number in the field log book and on the sample Chain-Of-Custody (COC) record. The type of sample media is recorded with the sample identification number in the field log book and on the COC record. Laboratory analyses to be conducted on the sample are recorded with the sample identification number in the field log book and on the chain-of-custody record.

Custody of samples must be maintained and documented from the time of sample collection to completion of the analyses. Each sample will be considered to be in the sampler's custody, and the sampler will be personally responsible for the care and custody of the samples until they are delivered to the courier service for delivery to the laboratory. A sample is considered to be under a person's custody if:

- The sample is in the person's physical possession;
- The sample is in view of the person after that person has taken possession;
- The sample is secured by that person so that no one can tamper with the sample;
- The sample is secured by that person in an area that is restricted to authorized personnel;

All samples will be accompanied to the laboratory by a chain-of-custody record. The chain-of-custody form contains the following information:

- Project name;
- Sample numbers;
- Sample collection point;
- Sampling date;
- Time of collection of samples (must match the time recorded on the sample label);
- Sample matrix description;
- Analyses requested for each sample;
- Preservation method;
- Number and type of containers used;
- Any special handling or analysis requirements.
- Signature of person collecting the samples;
- Signature of persons involved in the chain of possession.

The chain-of-custody record forms will be filled out with indelible ink. When the samples are transferred from one party to another, the individuals will sign, date, and note the time on the form. A separate form will accompany each delivery of samples to the laboratory. The chain-of-custody form will be included in the cooler used for preservation and transport of the samples. The sampling personnel will retain a copy of the form. Detailed procedure for completion of the COC record is presented in SOP 1.1.

1.5.1.4 Sample Identification

A unique identification number will be assigned to each sample. This number is typically an alphanumeric sequence or integer that serves as an acronym to identify the sample. Specific sample identification procedures will follow a strategy as outlined in the site specific SAP. All information pertaining to a particular sample is referenced by its identification number. The sample identification number is recorded on the sample container, in the field log book, and on the sample COC record. Following sample collection, the sample label is completed in waterproof ink and secured to the sample container with clear, tape which is wider than the label itself.

Each sample collected at the site will be labeled with the following information:

- Sample identification number;
- Sample location;
- Date and time of collection;
- Initials of person collecting the sample;
- Analysis requested;
- Preservation;
- Any other information pertinent to the sample.

1.5.2 Laboratory Documentation and Records

The laboratory will have all standard operating procedures (SOPs) formalized in writing and readily available for all staff. At a minimum, SOPs will be written for the following areas to include all their associated procedures and methods: sample receipt/control, sample preparation/extraction, sample analysis, result calculation, database management, health and safety, and the QA/QC program. In general, all steps of sample preparation/extraction, sample analysis, and result calculation will be documented in bound laboratory notebooks. Alternatively, computer-generated forms may be used if each page contains the date printed and is sequentially numbered. Such forms will be bound for long-term storage.

1.5.2.1 Sample Receipt/Laboratory Custody

All samples received at the laboratory will be carefully checked for label identification, and complete, accurate chain-of-custody documentation. The condition of the samples will be checked and the ambient temperature in the cooler and the temperature blank will be measured immediately after the cooler is opened. These results will be recorded on the Cooler Receipt Form. Photographs are recommended to document the condition of samples if significant out-of-control conditions are noted at the time of sample receipt. The laboratory will determine pH of samples for metals analysis upon receipt of sample coolers and will record measurements on the cooler receipt form. The pH of VOA samples will be measured at the time of analysis and recorded in laboratory injection logbooks.

Within one working day of sample receipt by the laboratory, an acknowledgment and cooler receipt form will be faxed to the Project Chemist at the fax number provided in the site specific SAP.

A unique laboratory identification number will be assigned through a computerized Laboratory Information Management System (LIMS) that stores all identification and essential information. The LIMS system tracks the sample from storage through each step in the laboratory until the analytical process is complete and the sample is returned to the custody of Sample Control for disposal. Access to the laboratory will be restricted to prevent any unauthorized contact with samples, extracts, or documentation.

1.5.2.2 Data Reporting / Comprehensive Certificates of Analysis

This section provides a detail of the requirements for each type of data reporting format which may be provided by the laboratory. The type of report will be determined on a project-specific basis. Preliminary certificates of analysis will be provided within 10 business days of sample receipt. The preliminary certificate of analysis will contain analytical results and basic QC

information including MS/MSD, LCS, and method blank results, and chain-of-custody and cooler receipt forms. Comprehensive certificates of analysis will be submitted to TEAD within 21 calendar days of sample receipt. Project SAPs may include other turnaround times which will replace these for that project only. Preliminary certificates of analysis will be shipped to TEAD as soon as they are available. Final comprehensive certificates of analysis will be submitted to TEAD within 21 calendar days after the last sample is collected for a delivery order. Each comprehensive certificate of analysis will contain the following items:

- Original copies of cooler receipt forms documenting sample conditions upon arrival at the laboratory and chain of custody/request for analysis (COC) forms for the samples included in the certificate
- Results for each sample and analytical method as a detected concentration or as less than the limit of quantitation (LOQ) for each analyte with appropriate data qualifiers, as needed. All samples with out of control spike recoveries being attributed to matrix interference will be designated as such. Soil sample results and LOQs will be reported on a dry weight basis with the percent moisture reported for each sample. Dilution factors and rationale for dilution, date of extraction, date of analysis, and analytical method will be reported for each analyte.
- Method blank results for all analytes and each analytical method. Sample results must be associated with a particular method blank. Any concentration above one half the LOQ detected in the method blank should be reported.
- Surrogate spike recoveries and control limits for all applicable methods (organic analyses), with any out-of-control recoveries flagged.
- Matrix spike/matrix spike duplicate (MS/MSD) results for all analyses, with recoveries, relative percent differences (RPD), and control limits for each spiked analyte. Sample results must be associated with a particular project-specific MS/MSD set. If a MS/MSD set is reanalyzed because of out of control results and the reanalysis is also out of control, both results will be reported and the data flagged. (MS/MSD sets with results not meeting specified acceptance criteria will be re-analyzed once. If re-analysis results are out of control both sets will be reported and the data flagged as appropriate.).
- Laboratory duplicate results with RPD and control limits for each analyte.
- Laboratory control samples (LCS) results with control limits. Sample results must be associated with a particular LCS.
- Initial and continuing calibration summaries and injection logs
- A summary of all samples with detected concentrations of target compounds indexed by method and by sample ID (to be provided when database is implemented)
- A summary of all surrogate recoveries for organic analyses for each applicable method

with the acceptable recovery range clearly indicated. This summary will be performed for all samples for each analytical method involving surrogate spikes

- A summary of all matrix spike/matrix spike duplicate analyses for each applicable method indicating acceptable recovery ranges and QC acceptance criteria for RPD
- A summary of all laboratory duplicates with QC acceptance criteria for RPD clearly indicated
- A summary (prepared by the contractor) of all field duplicates with QC acceptance criteria for RPD clearly indicated
- A table (prepared by the contractor) identifying all QA samples and the corresponding primary samples.
- A narrative section identifying all out of control conditions, corrective actions taken, and affected samples. A detailed discussion of all relevant quality control data will be included for out of control recoveries attributed to matrix effects.
- All data for analyses during the period covered by the comprehensive certificate of analysis will be included as an appendix to the comprehensive report. This data will be presented on numbered pages with an index or table of contents describing the contents of the appendix.

1.5.2.3 Raw Data Packages

Raw data packages will be requested for 10 percent of all samples submitted to the Laboratory. Raw data packages will be delivered within 21 days of a request for the data (or within 28 days of the last sample that is submitted for a project). The raw data package for organic/inorganic analyses will consist of a case narrative, chain-of-custody documentation, summary of results for environmental samples, summary of QA/QC results, and the raw data. Detailed descriptions of the requirements for each component of an organics/inorganics raw data package are provided in the following sections.

1.5.2.3.1 Case Narrative

A case narrative will be written on laboratory letterhead and the release of data will be authorized by the laboratory manager or his/her designee. Items to be included in the case narrative are the field sample ID with the corresponding laboratory ID, parameters analyzed for in each sample and the methodology used (EPA method numbers or other citation, a statement on the status of samples analyzed with respect to holding times (met or exceeded), detailed description of all problems encountered, discussion of possible reasons for out of control QA/QC criteria, and observations regarding any occurrences which may effect sample integrity or data quality.

1.5.2.3.2 Chain-of-Custody Documentation

Legible copies of COC forms for each sample will be maintained in the data package. Cooler log-in sheets will be associated with the corresponding COC form. Any internal laboratory tracking document will be included.

1.5.2.3.3 Summary of Environmental Results

For each environmental sample analysis, this summary will include field ID and corresponding laboratory ID, sample matrix, date of sample preparation (if applicable), date and time of analysis, identification of the instrument used for analysis, instrument specifications, GC column and detector specifications (if applicable), weight or volume of sample used for analysis/preparation, dilution or concentration factor used for sample preparation, percentage of moisture in the sample, method detection limit or sample quantitation limit, definitions of any data qualifiers used, and analytical results.

1.5.2.3.4 Summary of QA/QC Results

The following QA/QC results will be presented in summary form. Details specified in section 1.5.4.3.3 Summary of Environmental Results (Organic or Inorganic Analysis) will be included in the summary of QA/QC results. Acceptance limits for all categories of QC criteria will be provided with the data. All summaries will be presented on standard forms. Standard instrument output alone will not be submitted to satisfy the requirements of raw data packages.

1.5.2.3.5 Instrument Calibration

The order of reporting of calibrations for each analyte must follow the temporal order in which standards were analyzed.

1.5.2.3.6 Initial Calibration

The source of calibration standards true values and found values of concentrations and percent recovery will be noted. In addition, the concentrations of the standards used for analysis and the date and time of analysis, the correlation coefficient (r), coefficient of determination (r^2), calibration factor, relative response factor, percent relative standard deviation (%RSD), and retention time for each analyte (as applicable, GC and GC/MS analyses) will be included in initial calibration summaries. A statement should also be made regarding the samples or dates for which a single initial calibration applies.

1.5.2.3.7 Continuing Calibration

The concentration and source of the calibration standard used for daily calibration and/or mid-level calibration check will be reported. The response factor, percent difference, and retention

time for each analyte will be reported (GC and GC/MS) as well as percent recovery for each element analyzed. Daily calibration information will be linked to sample analyses by summary or by daily injection or analysis logs.

1.5.2.3.8 Method Blank Analyses

The concentrations of any analytes found in method blanks will be reported. The environmental samples and QA/QC analyses associated with each method blank will be stated. The date and time will also be reported.

1.5.2.3.9 Interference Check Sample

The concentrations and source of the interference check sample will be reported, as well as the percent recovery for each element analyzed, and the date and time of analysis.

1.5.2.3.10 Surrogate Standard Recovery

The name and concentration of each surrogate compound added will be reported. The percent recovery of each surrogate compound in the samples, method blanks, matrix spike/matrix spike duplicates, and other QA/QC analyses will be summarized with sample Ids such that the information can be linked to sample and QA/QC analyses.

1.5.2.3.11 Precision and Accuracy

For matrix spike/matrix spike duplicate analyses and LCS/LCS duplicate analyses, the sample results, spiked sample results, percent recovery, and RPD with the associated control limits will be reported. For laboratory duplicates, the original concentrations, RPD, and acceptable control limits for each analyte will also be reported. All batch QC information will be linked to the corresponding sample groups. For post digestion spikes, the concentration of the spiked sample, the sample results, the spiking solution added, percent recovery, and control limits will be detailed. Date and time for all analyses will be recorded.

1.5.2.3.12 Retention Time Windows (GC, GC/MS, HPLC)

The retention time window for each analyte for both primary and confirmation analyses will be reported. Retention time windows will be updated daily per EPA SW-846.

1.5.2.3.13 Compound Identification (GC, GC/MS, HPLC)

The retention times, mass spectra and the concentrations for each analyte detected in environmental and QA/QC samples will be reported for both primary and confirmation analyses (when applicable).

1.5.2.3.14 Method Detection Limits

Results of the most current detection limit study will be provided in the raw data package.

1.5.2.3.15 Injection Record

Injection logs for all instruments used for analysis of project samples will be provided indicating the date and time of analysis of project samples and the associated laboratory QA/QC samples (initial calibration, continuing calibration check, method blank, matrix spikes, etc.).

1.5.2.3.16 Method of Standard Additions (MSA)

This summary will be included when MSA analyses are required. The absorbance values and the corresponding concentration values, the final analyte concentrations, and correlation coefficients will be reported for all analyses. Date and time of analysis will be recorded for all analyses.

1.5.2.3.17 Inductively Coupled Plasma (ICP) Serial Dilution

The initial and serial dilution results with percent difference will be reported.

1.5.2.3.18 ICP Linear Ranges

For each instrument and wavelength used, the date on which the linear range was established, the integration time, and the upper limit concentration will be reported.

1.5.2.3.19 ICP Interelement Correction Factors

For each instrument and wavelength used, the date on which correction factors were determined will be detailed. Specific correction factors for Al, Ca, Fe, Mg, and any other element and the analytes to which they are applied will be detailed.

1.5.2.3.20 Method Detection Limits

Results of the most current method detection limit (MDL) study will be provided in the raw data package.

1.5.2.3.21 Analysis Record

Analysis logs for all instruments used for analysis of project samples will be provided indicating the date and time of analysis of project samples and the associated laboratory QA/QC samples (initial calibration, continuing calibration check, method blank, matrix spikes, etc.).

1.5.2.3.22 Raw Data

Raw data will be organized systematically on numbered pages. The data package will include

legible copies of the raw data for environmental samples (arranged in increasing order of field ID), instrument calibrations, QA/QC analyses, sample extraction and cleanup logs, instrument analysis logs for each instrument used. Instrument analysis logs will be provided for all days on which analysis was performed. Measurement printouts and quantitation reports for each instrument used will also be submitted. Records of absorbance, titrimetric or other measurements for wet chemical analysis will be recorded. All raw data will be presented on standard forms and accompanied by the instrument output.

1.5.2.3.23 HPLC/GC Analyses

This section of the data package will include legible copies of the raw data for environmental samples (arranged in increasing order of field ID, Primary and confirmation analyses), instrument calibrations, QA/QC analyses, sample extraction and cleanup logs, instrument analysis logs (injection record) for each instrument used, and GC/MS confirmation if applicable. The raw data for each analysis will include chromatograms (preferably with target compound, internal standard, and surrogate compounds labeled by name) with a quantitation report and/or area print out.

1.5.2.3.24 GC/MS Analyses

This section of the data package will include legible copies of the raw data for environmental samples (arranged in increasing order of field ID, spectrometer tuning and mass calibration reports, initial and continuing instrument calibrations, QC analyses, sample extraction logs, and instrument analysis logs (injection record) for each instrument used. The raw data for each analysis will include chromatograms (preferably with target compound, internal standard, and surrogate compounds labelled by name) and enhanced spectra of target compounds and/or tentatively identified compounds with the associated best matched spectra. Quantitation reports for all analyses will be included in the data package.

1.5.2.4 Electronic Data Deliverables

The contract laboratory shall provide sample data and all associated quality control data in electronic format as described in the *Electronic Data Specification* contained in Appendix E. This specification provides for a deliverable consistent with the latest Environmental Restoration Program Information Management System (ERPIMS) format. The *ERPIMS '98 Data Loading Handbook, Version 4.0* (October 1997) is incorporated by reference.

All electronic data submitted by the contract laboratory is required to be error-free, and in complete agreement with the hardcopy data. Data files are to be delivered both by email and on disks accompanying the hardcopy data reports. A software application will be supplied to the laboratory that will format the deliverable for email transmission. The disk must be submitted with a transmittal letter from the laboratory that certifies that the file is in agreement with

hardcopy data reports and has been found to be free of errors using the latest version of the evaluation software provided to the laboratory. The contract laboratory, at their cost, will correct any errors identified by the USACE, Sacramento District.

It is desired that analytical results be transferred electronically from instrument data systems to the laboratory's information management system (LIMS), at which point the electronic deliverable is generated in an automated fashion. In some analytical procedures where results are not captured by the analytical system, such as certain wet chemistry analyses, hand entry of results into the LIMS is necessary. In general, however, hand entry of any results is strongly discouraged.

1.5.3 Calculations

Data reduction calculations are typically included on the standard reporting forms developed by the laboratories and associated with each individual method or groups of methods. Calculations not present on standard reporting forms include computer-based data reduction programs. The laboratory is responsible for maintaining a list of these data reduction programs and for being able to demonstrate their validity. The complete calculation procedures used in computer-based data reduction programs (e.g., GC/MS and GC analyses) are based on the calculation procedures specified in each method.

Some instruments are configured to operate independently, without computer down-load of data. For these, the signal is recorded as a strip chart trace, numerical output on a printer strip, or direct reading from a digital or analog dial. In such cases, additional work is required by the analyst to reduce the data to a reportable format. The original signal must be multiplied by a calibration factor or compared with a standard curve. The aliquot result must be divided by the mass or volume of sample to produce a concentration-based final result. Most calculations are carried out on hand-held scientific calculators; simple programs (e.g., spreadsheets) are used for some. All of these data are recorded in a dedicated laboratory notebook or bench sheet for the particular determination in question. Results for single or multiple component tests are hand entered by the analyst in the assigned book.

Some laboratory tests, such as titrations or sensory evaluations, do not have instrument raw data. For these, the quantitative result or observation is recorded directly in a bound laboratory notebook or bench sheet by the assigned analyst. Calculations like those described above may be needed; these are recorded in the same laboratory notebook.

1.5.4 Data Integrity and Treatment of Outliers

All QC information will be recorded in the laboratory notebooks and printouts in the same format used for sample results. It is the analyst's responsibility to check the QC information against limits for the analysis. When an analysis of a QC sample (blank, spike, check standard,

replicate, or similar sample) shows that the analysis of that batch of samples is not in control, the analyst will immediately bring the matter to the attention of the group leader. The group leader will, if necessary, consult with the laboratory QC manager and/or the laboratory project manager to determine whether the analysis can proceed, or if selected samples should be rerun, or specific corrective action needs to be taken before analyzing additional samples. Out-of-control analyses and any corrective actions associated with TEAD project work must be documented and the records maintained by the laboratory. The analyst or group leader will file a Nonconformance Report with the laboratory QC manager for laboratory analysis out of control events that require documentation. The SAP will identify potential matrix interferences for laboratory analyses attributed to site characteristics. The associated methods for compensating for expected or unexpected interferences will be identified.

1.5.5 Data Management

The management of data takes place at varied levels within the full range of environmental services encompassing the scope of work. Program procedures, plans, and project-specific documents provide specific details of the individual positions responsible for data management, activities involved with data management, and minimum requisite credentials associated with these tasks. In general, the qualifications of individuals associated with data management activities will be commensurate with level of expertise necessary to ensure the intended level of evaluation.

1.5.6 Data Archive

Records management, including data archive, is specified in Section 4.0 of this document. Industry-standard hardware and software may be used for the development, processing, retrieval, and reporting of data stored on magnetic media. Contract laboratories will maintain all data records associated with a project for a minimum of five years following submission of the certificates of analysis (laboratory reports). As necessary, specific controls will be detailed in project-specific documents that require archiving protocols beyond that as specified in Section 4.0 of this document.

2.0 MEASUREMENTS / DATA ACQUISITION

This section describes the sample method requirements, analytical methods and quality control requirements, instrument calibration and data acquisition requirements.

2.1 Sampling Process Design

Project-specific SAPs will provide reference to applicable requirements that are to be followed from program level requirements (i.e. CDQMP and SOPs) and any project-specific details that may differ from this predefined guidance. In addition, the SAP will provide project-specific details of the experimental design to include the following:

- Sampling network design
- Types of samples required
- Sampling frequencies
- Sample matrices
- Measurement parameters of interest.

The rationale for the sampling design will be described for all sites where samples will be collected. Sample locations will be clearly identified on figures or other suitable means. Applicable measurement parameters will include, but are not limited to, geological, geophysical, hydrogeological, and chemical parameters. If field locations and sites are to be determined in the field based on observation (e.g., cone penetrometer, hydropunch, monitoring well), the criteria and guidelines to be used for this assessment will be specified. Similarly, the design for monitoring well installation, to include filter packs and well screens will be defined.

2.2 Sampling Methods Requirements

Samples will be collected in accordance with approved plans and SOP's which include qualitative and quantitative requirements for the specific collection methods to be utilized. These procedures will consider the mitigation of collection errors which may affect the representativeness of the sample and impact the established data quality objectives for the project. Soil sampling procedures will include split spoon sampling, shallow hand auger sampling, grab sampling, EnCore™ sampling, and stockpile soil sampling. Water sampling procedures will include groundwater sampling, surface water sampling, and drum (waste) sampling. The Field Sampling Plan component of this CDQMP provides a detailed discussion for each of the above mentioned procedures. The SAP will provide a detailed project specific discussion of the requirements and reference applicable procedures as they pertain to that project.

Table 2-1 outlines the required sample containers, preservative, and holding times for each analytical method and matrix.

To uniquely identify and track each sample, a unique sample number will be affixed to the sample container in accordance with SOP 2.1 and 2.2. A duplicate sample number, identical to the sample number on the sample label, will be placed in the field sample logbook along with all pertinent sample identification information.

Routinely, the selection of samples to be batched for extraction and the samples to be used for QC analysis purposes (i.e. matrix spikes and duplicates) in the laboratory will be designated by field personnel. This information will be communicated to the laboratory via COC. However, the laboratory will be responsible for ensuring that QC analysis is performed for each batch of samples/extracts for each parameter.

2.3 Sampling Handling Procedures

Samples will be collected in accordance with approved Field Sampling Plans and SOP's which include qualitative and quantitative requirements for the specific collection methods to be utilized. These procedures will consider the mitigation of collection errors which affect the representativeness of the sample and the established data quality objectives for the project.

Samples will be collected in containers appropriately labeled to uniquely identify each sample. The sample label information will include sample type, date, time, and sample number. Whenever possible labels will be placed on all sample containers prior to sample collection in accordance with SOP 2.1.

To uniquely identify and track each sample, a unique sample number will be affixed to the sample container in accordance with SOP 2.2. A duplicate sample number, identical to the sample number on the sample label, will be placed in the field sample logbook along with all pertinent sample identification information.

Routinely, the selection of samples to be batched for extraction and the samples to be used for QC analysis purposes (i.e. matrix spikes and duplicates) in the laboratory will be designated by field personnel. This information will be communicated to the laboratory via COC. However, the laboratory will be responsible for ensuring that QC analysis is performed for each batch of samples/extracts for each parameter.

2.3.1 Packing

Samples will be transported as soon as possible after sample collection to the laboratory for analysis. The following procedures are to be used when packing and transporting samples to the laboratory:

- Use waterproof metal or equivalent strength plastic ice chests or coolers;
- Place absorbent material in the bottom of the cooler;

- Package samples in individual plastic bags and place in cooler;
- Fill cooler with cushioning material;
- Package wet ice in plastic bags and place bags around, among, below, and on top of the samples;
- Put paperwork (chain-of-custody record, etc.) in a waterproof plastic bag and tape it to the inside lid of the cooler;
- Tape the cooler lid and drain shut with fiber-reinforced tape;
- Place two numbered and signed custody seals on cooler, one at the front right and one at the back left of cooler ;
- Put “This Side Up” and “Fragile” labels on all sides of any cooler containing glass bottles or jars;
- Attach completed shipping label to the top of cooler and ship following the carrier's instructions.

Detailed procedures for sample packaging is provided in SOP 2.0.

2.3.2 Shipping

Sample coolers are typically shipped by overnight express carrier to the laboratory. A copy of the bill of lading (air bill) is to be retained and becomes part of the sample custody documentation. The laboratory should be notified in advance of all shipments preferably by advanced scheduling and by telephone on the day of shipment. Detailed procedures for sample shipping is provided in SOP 2.0.

2.3.3 Sample Preservation and Holding Times

Chemical preservatives will be used in samples where appropriate and all samples will be placed on ice and cooled in ice chests for shipment to approximately 4 degrees Celsius (°C). Upon receipt at the laboratory, the samples will be stored in controlled and locked refrigerators at 4°C ±2°C until analyzed. The pH of acid or base preserved non-volatile aqueous samples and the temperature of the temperature blank will be checked upon sample delivery at the laboratory. VOA vials for sample analysis will not be opened until analysis begins. The laboratory will record the temperature and condition of the samples at the time of receipt on the COC. For samples received with a nonconforming pH or with temperature outside the acceptable range (4°C ± 2°C), the Project Chemist will be notified within 8 hours of nonconformance discovery. The Project Chemist in concurrence with the TM will decide on a project specific basis whether the analysis should proceed, or if samples should be recollected and resubmitted for analysis. Regardless, laboratory personnel will adjust the sample to proper pH as soon as possible. Samples collected and delivered to a laboratory within four hours of collection will be exempted from the temperature requirement as long as the samples were handled in accordance with the specified procedures. Sample containers, preservatives and holding times of samples will be observed as indicated in Table 2-1.

2.3.4 Laboratory Receipt and Entry of Samples

The integrity and documentation of sample custody starts when cleaned sample containers are shipped to the field under custody. Samples shipped to laboratories from the field are received by the sample custodian. Upon receipt of samples in the laboratory, the integrity of the shipping container is checked by verifying that the custody seal is not broken. The internal cooler temperature will be measured by means of a temperature blank. Sample containers are inspected for breakage, leakage, damage and the contents of the shipping container are verified against the COC records. Chain-of-custody Records are checked for accuracy and completeness, and receipt conditions will be documented on the COC. If the samples and documentation are acceptable, each sample container is assigned a unique laboratory identification number from the Laboratory Information Management Systems (LIMS) database. If the samples, documentation, or coolers are not acceptable, the Laboratory Project Manager (LPM) is informed verbally and with a completed laboratory NCR. The LPM will immediately notify the Project Chemist and TM. After discrepancies have been resolved, a LIMS record hard copy is generated to document the following:

- Date of sample receipt
- Sample accession number
- Source of sample

Each sample received will be assigned a unique laboratory sample accession number by the LIMS system at the time samples are logged in. One of the functions of the LIMS is to assist in tracking samples while they are in the custody of the laboratory. Other information recorded will include date and time of sampling, sample description, due dates, and required analytical tests. Samples are batched in lots of 20 or less at the time of sample preparation or at the time of analysis if no preparation is required. When LIMS log-in has been completed, the samples are transferred to the appropriate refrigerators in the sample control area. In order to minimize the potential for cross-contamination of samples, separate refrigerators are used for samples suspected to contain high levels of organic compounds and for samples receiving analysis for volatile compounds. The sample refrigerators are kept at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and their temperatures are recorded daily with thermometers verified against National Institute of Standards and Technology (NIST) thermometers. The refrigerators storing samples for volatile analysis are monitored for contamination with refrigerator blanks, which are analyzed weekly.

Samples are distributed to the laboratory from sample control by either a sample custodian or laboratory chemist. Internal chain of custody is initiated whenever a sample is removed from the sample control area. When samples are returned to the sample control refrigerators by laboratory personnel, internal chain of custody is completed.

The following illustrates the process that a sample takes from receipt to storage for disposal:

- Document physical condition of sample and sample preservation
- Verify documentation and parameter assignment
- Log into LIMS
- Laboratory Project Manager sends acknowledgment FAX with cooler receipt to the Project Chemist
- Store sample according to preservation guidelines
- Transfer sample to lab with proper documentation (lab personnel removes samples from sample control and signs samples on lab sample custody sheet)
- Document analytical work
- Return unused portion of samples to sample control
- Return sample to client or arrange for sample disposal

2.3.5 Pre-Analysis Storage

Personnel from the laboratory will receive and log in the samples. The samples are then placed into temporary storage until analyzed. Samples are stored as prescribed in the approved Laboratory QA manual. Methods of storage are intended generally to:

- Retard biological action
- Retard hydrolysis of chemical compounds and complexes
- Reduce volatility of constituents
- Reduce adsorption effects.

Preservation methods are generally limited to pH control, chemical addition, and refrigeration.

2.3.6 Post-Analysis Storage

Original water samples will be stored refrigerated at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for a minimum of 2 months after the final data are submitted. Original soil samples and all sample extracts/digestates will be stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for a minimum of 6 months after final data are submitted. Samples for metals analysis only and metals digestates may be stored at room temperature. Disposal of all samples and extracts/digestates will be in accordance with federal and state regulations.

2.4 Analytical Methods Requirements

2.4.1 Overview of Analytical Methods

This section contains an overview of the preparation and instrumental procedures to be used for this project. Detailed descriptions of specific methods, with tables summarizing calibration procedures, QC sample acceptance values and corrective action, and LOQs, are given in Section 2.4.2 and the method specific tables located in Appendix B.

2.4.1.1 Organic Analyses

Organic Extractions

Prior to analysis on an instrument, analytes of interest must be separated from the matrix and concentrated. Target analytes are removed by serially extracting a known volume or weight with a solvent, collecting, then concentrating the solvent to a specified volume.

For aqueous organic extraction methods, it is recommended that volume be measured as described below:

- On the sample container, mark the top of the water column
- Pour the contents of the sample container into a separatory funnel
- Rinse the emptied sample container with solvent and add to the separatory funnel
- Fill the sample container with water
- Measure the water in the sample container with a graduated cylinder.

General Gas Chromatography

Gas chromatographs achieve separation by partitioning solutes between a mobile gas phase and a stationary liquid phase on solid support material. A typical analysis would proceed as follows. The organic extract of a sample is injected into a heated injection port. The solvent and solutes are immediately vaporized and swept onto a separation column by inert carrier gas. The solutes are adsorbed onto the stationary phase of the column and then are desorbed by fresh carrier gas. The sorption-desorption process occurs repeatedly as the sample moves through the column and each analyte will be retained based on its unique solubility with the stationary phase. After passing through the column, the solutes are eluted into a detector system.

Compound identification is based on the time it takes a compound to travel through a column. The retention time of a compound is determined during instrument calibration with target analytes. Since not all compounds have unique retention times, non-MS GC methods often require sample extracts to be analyzed on a second, dissimilar column to decrease the probability of false positives.

Second column confirmation will be provided for gas chromatography methods for all single peak analytes found above the reporting limits given in the first table of the method specific tables found in Appendix B of this document using a dissimilar column.

Although SW-846 3rd Edition permits the use of higher order calibration curves, this CDQMP specifies that only linear curve fits be used in the quantitation of confirmed analytes. The analyst may choose to use either a linear curve forced through the origin or the linear curve as determined through regression routines. Specific criteria to be used for either type of linear curve is specified in the text and Appendix B, Tables, for the applicable methods.

General Detector Systems

Detector systems detect target analytes in the column effluent. Some are specific to classes of compounds (e.g., photoionization and electron capture), and some are relatively unselective (e.g., flame ionization). Selective detectors often provide lower reporting limits by increasing the signal to noise ratio and by their selectivity and provide an additional level of confidence during compound identification. Mass spectrometers provide a high level of confidence in compound identification because they provide a characteristic ion pattern for fragmented target analyte molecules.

Once they are calibrated, detectors enable quantitation of target analytes. Calibration consists of the establishment of a dynamic working range and periodic continuing standards to show that the instrument is still operating within acceptable limits.

General Gas Chromatography/Mass Spectroscopy Methods (GC/MS)

GC/MS methods couple gas chromatographic techniques with mass spectrometry to allow confirmation of a compounds' identity and concentration. After partitioning by GC, the sample is metered into a mass spectrometer and bombarded with ions until molecular fragments result. Each molecular fragment is characteristic for a compound and can then be compared to reference spectra using computer routines. The reference spectra plus the retention time are used to confirm the identity of the compound. Quantitation is performed by comparing the response of the primary (or secondary as necessary) ions relative to an internal standard with a multipoint initial calibration curve.

2.4.1.2 Metals Analyses

Two techniques, inductively coupled plasma (ICP) atomic emission spectroscopy and atomic absorption (AA), will be employed to measure levels of specified metals in the samples. Sample digestion is required prior to most ICP and AA analyses.

Inductively Coupled Plasma/Atomic Emission Spectroscopy (ICP) Procedures

ICP determines elements in solution. All matrices including groundwater, surface water, aqueous samples, industrial wastes, soils, sludges, TCLP and STLC extracts, and sediments require digestion prior to analysis. Aqueous samples and leachates may be digested using preparation methods SW-3010A or SW-3020A as described in the text. Solid samples may be digested using methods SW-3050B.

Method SW-6010B provides a simultaneous or sequential multi-element determination of elements by ICP. Element-emitted light is measured by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic line emission spectra are produced by radio-frequency inductively coupled plasma. The spectra

are dispersed and the intensities of the lines are monitored by photomultiplier tubes. Background correction is required for trace element determination.

Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) Procedures

ICPMS determines elements in solution. All matrices including groundwater, surface water, aqueous samples, industrial wastes, soils, sludges, and sediments require digestion by Methods SW-3020A (water) or SW-3050B (soil - modified for ICP/MS analysis) prior to analysis. Method SW-6020 Modified provides a simultaneous multi-element determination by ICP/MS. The method measures ions produced by radio-frequency ICP. Analytes are nebulized from the sample and the resulting aerosol is transported by argon gas to the plasma torch. The ions are entrained in the plasma gas and introduced, by means of a water-cooled interface, into a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratios and quantified by a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate non-conformance. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

Analysis by Atomic Absorption Spectroscopy

Elements such as arsenic, selenium and lead may be determined using atomic absorption techniques rather than ICP, in order to achieve the required detection limits or where interferences are encountered. If required because of interferences, thallium may also be determined using atomic absorption spectroscopy.

Graphite Furnace AA

Metals in solution can be determined by atomic absorption(AA). Prior to analysis, soil samples are prepared using the digestion procedure described in Method SW-3050B. Aqueous or leachate samples may be prepared using Method SW-3010A or SW-3020A. The digestate is introduced into the graphite furnace, electrothermally dried, charred, and atomized. The resulting absorption of a specific light beam from the hollow cathode or electrodeless discharge lamp (EDL) lamp is proportional to the metal concentration. Background correction will be used for all analyses. Samples with concentrations outside the linear calibration range will be diluted. The matrix may be modified by the addition of certain compounds or elements, as recommended by the determinative methods, to reduce interferences. The presence of interferences will be verified and documented by applying the procedures as outlined in the method specific table located in Appendix B.

Flame (Direct Aspiration) AA

Direct aspiration is used for organic lead determinations (LUFT Manual, 1989, and Method SW-

7420), and may be used for other metals upon prior arrangement with the laboratory. Prior to analysis by direct aspiration, samples are extracted using the procedure described for organic lead LUFT and Method SW-3010A. Following sample preparation, a representative aliquot is aspirated into an air/acetylene flame. The resulting absorption of a specific light beam from the hollow cathode or EDL lamp will be proportional to the metal concentration. Background correction will be employed for all analyses.

Mercury by Cold Vapor Atomic Absorption

Mercury will be determined in selected solid samples using SW-7471A and in water samples using SW-7470A. Methods SW-7470A and SW-7471A are cold-vapor atomic absorption procedures for determining the concentration of mercury in extracts, groundwater, and waste samples. Sample preparation is specified in the method. Following dissolution, mercury in the sample is reduced to the elemental state, aerated from solution, and the vapor passed through a cell positioned in the light path of an atomic absorption spectrometer. Permanganate is added to the sample during preparation to reduce interferences from sulfides and chlorides.

2.4.2 Method Descriptions

The following text provides a brief summary description for each analytical method. Method specific tables are located in Appendix B. Since EPA has discontinued promulgation of new and improved methods, for all analytical methods performed for TEAD projects, the most recently published version of the EPA method shall be used. The actual version of the method used shall be recorded in the site specific documents and laboratory reports.

2.4.2.1 Organics

2.4.2.1.1 Sample Preparation

SW-3510- Separatory Funnel Liquid-Liquid Extraction

Method 3510 is applicable to the isolation and concentration of organic compounds from aqueous samples. A measured volume (usually one liter) of sample is placed into a separatory funnel, adjusted if necessary to a specific pH, and serially extracted with methylene chloride. The extract is then dried with anhydrous sodium sulfate, exchanged (as necessary) into a solvent compatible with the determinative method, and concentrated to the appropriate volume.

SW-3520 - Continuous Liquid-Liquid Extraction

Method SW-3520 is a procedure for isolation and concentration of organic compounds from aqueous samples. A measured volume (usually one liter) of sample is placed into a continuous

liquid-liquid extractor, adjusted if necessary to a specific pH, and extracted with Freon or methylene chloride for 18 hours to 24 hours. The extract is then dried, exchanged (as necessary) into a solvent compatible with the determinative method, and concentrated to the appropriate volume.

SW-3540 - Soxhlet Extraction

The procedure extracts nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. It is applicable to the isolation of water-insoluble and slightly water soluble organics for further analysis by gas chromatography. The solid sample is mixed with anhydrous sodium sulfate to form a free-flowing powder, placed in an extraction thimble, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is then dried, exchanged (as necessary) into a solvent compatible with the determinative method, and concentrated to the appropriate volume.

SW-3550 - Sonication Extraction

Method SW-3550 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, wastes, and sludges. The sonication process ensures intimate contact of the sample matrix with the extraction solvent. A weighed sample of the solid material is mixed with the anhydrous sodium sulfate, ground to form a free-flowing powder, then sonicated sequentially with three solvent aliquots. Freon and methylene chloride are typically used as solvents, although other solvents may be used for specific analytical applications. The extract is separated from the sample by vacuum or gravity filtration, or centrifugation, and then dried with anhydrous sodium sulfate and concentrated to the appropriate volume. The resulting solution is analyzed using the appropriate method.

SW-5030 - Purge and Trap

For liquid matrices, an aliquot of the sample is placed in the purge chamber and an inert gas is bubbled through the sample at ambient temperatures. The volatile components are then transferred from the aqueous matrix to a sorbent column where they are trapped. After purging is completed, the sorbent column is heated and backflushed with an inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components which are detected by the appropriate detector. An extraction method can be employed for nonaqueous and solid samples when high concentrations are expected. This involves one dilution of the sample into methanol. An aliquot of this methanol extract is then added to reagent water and purged as discussed above. For low-level soil samples, five grams of the sample is combined with five milliliters of reagent water, and the purge chamber is heated to 40°C. Twenty five milliliters of a sample are typically purged when performing low-level aqueous analyses.

SW-5035 - Closed System Purge and Trap and Extraction

For low-level soil samples, five grams of the sample are weighed in the field at the time of collection and added to the pre-weighed, septum sealed, screw-cap vial which already contains a stirring bar and sodium bisulfate preservative solution. Alternatively, the sample is collected with an EnCore™ sampler and the sampler is used as the storage device. Analysis must be performed within 48 hours unless the EnCore™ sampler is frozen during storage. Immediately prior to analysis, five milliliters of reagent water, surrogates and internal standards (as applicable) are added, without opening the sampling vial. The vial containing the sample is heated to 40°C. The contents of the vial are then purged using an inert gas combined with agitation and the volatile components are transferred to a sorbent column where they are trapped. After purging is completed, the sorbent column is heated and backflushed with an inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components which are detected by the appropriate detector.

An extraction method can be employed for oil soluble in water-miscible solvents and solid samples when high concentrations are expected. This involves one dilution of the sample into methanol. An aliquot of this methanol extract is then added to reagent water and purged as discussed in SW-5030.

2.4.2.1.2 Gas Chromatography

SW-8021 - Halogenated Volatile Organic Compounds by GC/HECD and Purgeable Aromatic Compounds by GC/PID

Method SW-8021 is a purge-and-trap (method SW-5030 or SW-5035) based procedure to determine halogenated volatile organic compounds and aromatic volatile organic compounds by gas chromatography. A temperature program is used in the gas chromatograph to effect an efficient separation of the organic sample components. Halogenated compounds are detected by a Hall Electrolytic Conductivity Detector (HECD). Aromatic compounds are detected by a photoionization detector (PID).

Volatile compounds in water or low-level contaminated soils can be introduced directly into the gas chromatograph by purge-and-trap, method SW-5030 or SW-5035. Medium-level contaminated soils may require methanolic extraction, as described in method SW-5030, prior to purge-and-trap. The sample volume or sample weight purged may vary to meet contract required quantitation limits as described in the project specific QA plan.

SW-8015 Modified - Total Petroleum Hydrocarbons by GC/FID

This method determines total petroleum hydrocarbons as gasoline, diesel or jet fuel by SW-8015B Modified. Volatile petroleum hydrocarbons (gasoline) are analyzed by purge-and-trap

method SW-5030 or SW-5035. Semivolatile petroleum hydrocarbons such as diesel and/or jet fuels are analyzed after extraction by SW-3510 (aqueous) or SW-3550 (soils). A sample, after purge-and-trap or extraction, is injected into a temperature programmed gas chromatograph and component detection is achieved by a flame ionization detector (FID). Generally, the carbon ranges listed below are typical of the fuels described:

Gasoline	C-6 to C-10
Diesel	C-10 to C-24
JP-4	C-8 to C-13
Motor Oil	C-24 to C-36

Gasoline in aqueous or low-level contaminated soil samples can be determined directly by purge-and-trap, method SW-5030 or SW-5035, and desorption into the gas chromatograph. Medium level contaminated soils may require methanolic extraction, as described in method SW-5030 prior to purge-and-trap.

Samples to be analyzed for diesel and jet fuel require extraction with methylene chloride prior to analysis. The extract may be concentrated prior to injection into the gas chromatograph.

Occasionally, a chromatogram may suggest that a mixture of fuels with overlapping carbon ranges is present in the field sample. The laboratory may then calibrate and quantify the total hydrocarbon concentration using one reference fuel. For example, if both JP-4 and gasoline are analyzed by the purge-and-trap method, then the laboratory has the option to quantify the result by using either a JP-4 or gasoline curve. The laboratory should strive to be consistent in their quantitation practice and document any anomalies in the narrative accompanying the data report.

SW-8081, SW-8082 - Organochlorine Pesticides and Polychlorinated Biphenyls (PCB)

Method SW-8081 and SW-8082 are a gas chromatography/electron capture detector methods for the detection of organochlorine pesticides and polychlorinated biphenyls (PCBs, commonly identified as Aroclor mixtures). These target analytes produce chromatograms with single peaks, or in the case of Aroclors and toxaphene, multiple peaks in recognizable patterns. Identification is based on the comparison of a resulting sample chromatogram to that of a standard. Quantitation is performed relative to the initial calibration.

Water samples are extracted at a neutral pH with methylene chloride by methods SW-3510 or SW-3520C. Method SW-3510 is a separatory funnel extraction technique and SW-3520 is a continuous liquid-liquid extraction. Soil samples are extracted with methylene chloride and acetone using method SW-3550, a sonication extraction procedure. Extracts are solvent exchanged into hexane and undergo clean up procedures as deemed necessary for the sample.

If an Aroclor (or any multi-eluter) is detected in the sample, then that Aroclor (or any multi-eluter) may be quantitated on a separate GC calibrated for that compound. All multicomponent

bracketing standards must be within $\pm 15\%$ from the expected concentration, as quantitated from the calibration factor as determined from the ICAL. ICAL, ICV, and CCV criteria must be met on the column used for quantitating and final reporting of the target analyte. At least five of the largest representative peaks are chosen for quantitation of the Aroclors. For the quantitation of Aroclor 1221, three peaks will be summed. PCB detections do not require a second column confirmation. The characteristic peak pattern serves as a primary level of qualitative identification.

SW-8141 - Organophosphorus Pesticides by GC

Method SW-8141 is a gas chromatographic method for the detection of various organophosphorus pesticides. A temperature program is used in the gas chromatograph to effect an efficient separation of the organic sample components. These sample components produce chromatograms with single peaks. Identification is based on the comparison of a resulting chromatogram to that of a standard.

Water samples are extracted at a neutral pH with methylene chloride by methods SW-3510 or SW-3520. Method SW-3510 is a separatory funnel extraction technique and SW-3520 is a continuous liquid-liquid extraction. Soil samples are extracted with methylene chloride and acetone using method SW-3550, a sonication extraction procedure. If method SW-8141 is used to analyze soils for organophosphorus pesticides, then extraction methods SW-3540 and SW-3541 will be used.

Extracts are solvent exchanged into hexane and undergo clean up procedures as deemed necessary for the sample.

SW-8151 - Chlorinated Herbicides

Method SW-8151 provides extraction, esterification and gas chromatographic conditions with electron capture detection for the analysis of chlorinated acid herbicides. Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type.

The herbicides are extracted from soil by shaker with acetone/ethyl ether, and from water by partitioning in a separatory funnel with ethyl ether. Extracts are hydrolyzed with aqueous KOH, acidified, and then extracted into ethyl ether. The extracts containing the protonated herbicides, are concentrated by rotary evaporation and nitrogen blow-down. The concentrates are methylated with diazomethane and solvent exchanged into hexane.

SW-8260 - Volatile Organics by GC/MS

This method is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure and is used to determine volatile organic compounds in a variety of solid waste

matrices. It is applicable to nearly all types of sample matrices, including water and soil. The volatile compounds are extracted and introduced into the gas chromatograph by the purge-and-trap method. The components are separated via the gas chromatograph and detected using mass spectrometer which provides both qualitative and quantitative information.

Volatile compounds in water or low-level contaminated soils can be introduced directly into the gas chromatograph by the purge-and-trap method (SW-5030B or SW-5035). Medium-level contaminated soils may require methanolic extraction, as described in method SW-5030B, prior to purge-and-trap.

SW-8270 - Semivolatile Organic Compounds by GC/MS

This method can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols.

Prior to using this method, samples must be prepared using the appropriate sample preparation method: for soil samples, sonication extraction (SW-3550) is used, and for water samples, separatory funnel (SW-3510) or continuous liquid/liquid extraction (SW-3520) are used.

SW-8280 - Polychlorinated Dibenzo Dioxins and Polychlorinated Dibenzofurans by GC/MS

Methods SW8280 is used to detect dioxins and furans in a variety of matrices and uses additional quality control to allow more sophisticated determinations of detection limits and matrix spike recoveries than other routine GC and GC/MS methods.

SW-8280 requires isotopically labeled analogs of target analytes to be spiked into each sample before extraction. SW-8280 uses six C¹³ analogs. These isotopically labeled analogs elute and behave as target analytes do, without interfering with the analysis. Target analytes are quantitated relative to the isotope analog and therefore their calculated concentration is compensated for extraction efficiency. The assessment of matrix effects on method performance, assessed by matrix spikes and matrix spike duplicates in other GC and GC/MS methods, can be met in SW-8280 with the isotopically labeled analogs. These isotopes are spiked into each sample and therefore matrix effects on method performance can be judged by the recovery of these isotopes, for each sample. Sample analysis acceptance is controlled by the performance of these isotopes in the sample. The batch specific LCS will use isotopically labeled analogs of the target analytes and unlabeled natives to control the batch. In summary, no MS/MSD will be performed for SW-8280 sample analyses and batch control will be done by the recovery of the spiked, isotopically labeled, analogs and unlabeled natives.

All PCDD and PCDF analyses performed (for EPA since 1982) have used a technique for calculating the detection limit for each of the chlorination levels and each congener by using the noise level present in the elution window and the height of the chromatographic peak of the internal standard. Both the signal to noise and peak height are determined by the GC/MS data system and the result of the calculation is a detection limit that is specific to the homologous series and sample.

There is a three tiered approach to reporting and detection limits. In the absence of target analytes, a sample specific estimated detection limit (EDL) is calculated based on method signal to noise ratios. The target analyte is then reported as “not detected” at the EDL. When target analytes are found, they are reported down to the lower calibration limits without conditional modifiers such as a J flag. If below the lower calibration limit, the target analyte will be qualified as such.

If there is a peak which meets the signal-to-noise criteria, but not all of the other identification criteria (i.e. retention time, ion ratio, absence of diphenyl ethers, and analyst judgement), an EMPC (estimated maximum possible concentration) based on the ion peak is calculated. The target analyte is reported as “not detected” at that calculated detection limit and is qualified as an EMPC.

SW-8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)

This method provides instrument and extraction procedures for the detection and quantitation of PCDDs (tetra through octa-chlorinated homologues) and PCDFs (tetra through octa-chlorinated homologues) in a variety of sample matrices and part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations.

Method SW-8290 is used to detect dioxins and furans in a variety of matrices and uses additional quality controls to allow more sophisticated determinations of detection limits and matrix spike recoveries than other routine GC and GC/MS methods.

SW-8290 requires isotopically labeled analogs of target analytes to be spiked into each sample before extraction, and uses ten C¹³ analogs, one furan and one dioxin at each chlorination level. These isotopically labeled analogs elute and behave as target analytes do, without interfering with the analysis. Target analytes are quantitated relative to the isotope analog and therefore their calculated concentration is compensated for extraction efficiency.

There is a three tiered approach to reporting and detection limits. In the absence of target analytes, a sample specific estimated detection limit (EDL) is calculated based on signal-to-noise (S/N) ratios at the retention time of the analyte. The target analyte is then reported as “not

detected” at the EDL. When target analytes are found, they are reported down to the lowest calibration standard concentration without conditional modifiers such as a J flag. Below the SW-846 specified reporting limits, qualitatively confirmed analytes are reported as “estimated” down to the target detection limit (TDL) to denote the less certain quantitation. The TDL is a value set by the lab at which there is no significant chance of false positives. If there is a peak below the TDL, and all qualitative criteria such as retention time, ion ratios, signal to noise ratio, the absence of diphenyl ether, and analyst judgment, are not met, a detection limit based on the ion peaks is calculated and the target analyte is reported as “not detected” at that calculated detection limit.

The assessment of matrix effects on method performance can be met in SW-8290 with the isotopically labeled analogs. These isotopes are spiked into each sample and therefore matrix effects on method performance can be judged by the recovery of these isotopes, for each sample. Sample analysis acceptance is controlled by the performance of these isotopes in the sample. Furthermore, the batch specific LCS are also not required since the batch specific method blank uses isotopically labeled analogs of the target analytes and controls the batch.

In summary, no MS/MSD is performed for SW-8290 sample analyses (unless specifically requested by the client), and batch control will be done by the recovery of the spiked, isotopically labeled, analogs in the method blanks.

2.4.2.1.3 High Performance Liquid Chromatography

SW-8310- Polynuclear Aromatic Hydrocarbons

Method SW-8310 is a liquid chromatography method with ultra-violet and fluorescence detection for the analysis of polynuclear aromatic hydrocarbons (abbreviated as PAH or PNA). These target analytes produce chromatograms with single peaks. Identification is based on the comparison of a resulting sample chromatogram to that of a standard. Quantitation is performed relative to the initial calibration.

Water samples are extracted with methylene chloride by methods SW-3510 or SW-3520. Method SW-3510 is a separatory funnel extraction technique and SW-3520 is a continuous liquid-liquid extraction. Soil samples are extracted with methylene chloride and acetone using method SW-3550, a sonication extraction procedure. Soil extracts are solvent exchanged and undergo a silica gel clean up procedure. Extracts are then solvent exchanged to methanol for analysis.

SW-8330, SW-8321 Modified - Nitroaromatics and Nitroamines by HPLC.

Method SW-8330 is a high performance liquid chromatography/ultra-violet (HPLC/UV) method and Method SW-8321 Modified is a high performance liquid chromatography/mass spectrometer

(HPLC/MD) method for the extraction and detection of explosives residues in waters, soils, and sediments. All samples and extracts are analyzed on an HPLC fitted with a C-8 reverse phase column at a UV detection of 250 nm. Positive detections may be confirmed on a cyano-column.

Aqueous samples suspected of containing low level concentrations of explosives residues are extracted by “salting out” an aliquot of sample with sodium chloride, extracting with acetonitrile, then analyzing the extract. Aqueous samples suspected of containing high levels of explosive residues are analyzed on the HPLC using direct aqueous injection. High level aqueous samples are filtered prior to analysis.

For soil and sediment samples, a 2-gram sample aliquot is extracted with acetonitrile, aliquoted, treated with calcium chloride solution, filtered, then analyzed by HPLC. If soils and sediments appear non-homogeneous they are air dried, ground, and sieved through a 30 mesh screen before sample extraction.

Army Method UT094, SW-8321A Modified or equivalent (Thiodiglycol and Thiodiglycolic Acid)

Method UT094 and SW-8321A Modified are high performance liquid chromatography/ultra-violet (HPLC/UV) methods for the extraction and detection of thiodiglycol and thiodiglycolic acid in waters, soils, and sediments. All samples and extracts are analyzed on an HPLC fitted with a C-18 reverse phase column at a UV detection of 215 nm. Positive detections may be confirmed on a cyano-column.

SW-8321 Modified also allows for the use of liquid chromatography/thermo spray/mass spectrometer (LC/TSP/MS). For LC/TSP/MS, the extracts and standards are analyzed on an HPLC fitted with a reverse phase column and introduced into the mass spectrometer by thermospray. The advantage of this method is positive confirmation of target compounds by mass spectra and lower limits of detection. Quantitation is performed using internal standard techniques with d_5 atrazine as the internal standard.

2.4.2.2 Inorganics

2.4.2.2.1 Sample Preparation

SW-3020 - Acid Digestion of Aqueous Samples and Extracts for Total Metals

This digestion procedure is used for the preparation of aqueous samples and extracts that contain suspended solids. The procedure is used to prepare samples for analysis by GFAA. A mixture of nitric acid and the material to be analyzed is refluxed in a covered vessel. This step is repeated with additional portions of nitric acid, and subsequent additions of hydrogen peroxide, until the digestate is light in color or until its color has stabilized. After the digestate has been

brought to a low volume (approximately 10-20 mls), it is cooled and brought up to volume with dilute nitric acid such that the final dilution contains 3 percent (v/v) HNO₃. If the sample contains suspended solids, it must be centrifuged, filtered, or allowed to settle. This procedure includes modifications to Method SW-3020. The modifications are the addition of hydrogen peroxide and the reduction in volume to 10-20 mls, rather than 5 mls as specified in the method. The modifications are included to allow for the analysis of arsenic and selenium, in that Methods SW-7060 (arsenic) and SW-7740 (selenium) call for the addition of hydrogen peroxide, and less volume reduction.

SW-3020 Modified - Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP/MS

This digestion procedure is used for the preparation of aqueous samples and extracts that contain suspended solids. The procedure is used to prepare samples for analysis by ICP/MS. A mixture of nitric acid and the material to be analyzed is refluxed in a covered vessel. Hydrogen peroxide is added until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume (approximately 25 mls), it is cooled, 1 ml of 1:1 hydrochloric acid/water is added and the digestate is brought up to volume with deionized water. If the sample contains suspended solids, it must be centrifuged, filtered, or allowed to settle. The modifications to Method SW-3020A for analysis by ICP/MS include the addition of hydrogen peroxide and hydrochloric acid to aid in the digestion of certain elements (i.e. silver), and less volume reduction during digestion to allow for the analysis of the more volatile elements (i.e. antimony).

SW-3050 - Acid Digestion of Sediments, Sludges, and Solids

This digestion method is used to prepare sediment and soil samples for analysis by ICP, ICP/MS, graphite furnace atomic absorption (GFAA) and flame atomic absorption (FLAA). A representative portion of the sample is digested in 1:1 nitric acid. A final reflux procedure is performed using concentrated hydrochloric acid for FLAA or ICP, or concentrated nitric acid for GFAA. Hydrogen peroxide is added during the digestion procedure. The final volume is adjusted to 100 ml.

SW-3060, - Alkaline Digestion for Hexavalent Chromium

A 2 gram aliquot of sample is digested in 8ml of NaCO₃ /NaOH digestion solution on a hotplate for 30-45 minutes. The solution is cooled, filtered, and quantitatively transferred to a 100 ml volumetric flask. Just prior to analysis the solution is neutralized with HNO₃. A 9.5 ml portion of the digestate is then transferred to a 10 ml volumetric flask and is ready for analysis by method SW-7196A (Cr⁺⁶).

2.4.2.2.2 Atomic Emission

SW-6010 - Inductively Coupled Plasma (ICP)

Inductively coupled argon plasma (ICP) determines sample elements in the acid digestate of a sample. Simultaneous ICP uses multi-element atomic emission spectroscopy to identify and quantify metals. An aerosol of the sample is metered into the argon plasma. Element specific atomic emission spectra are produced by radio-frequency ICP. The spectra are dispersed and the lines monitored by photomultiplier tubes. The background is measured and the results are corrected for background levels and interelement interferences.

On a daily basis, the ICP will be calibrated using three standards. Alternatively, a laboratory may standardize the instrument using a blank and a single standard if a detection limit standard and an upper calibration range standard are included in the analysis sequence. Ongoing instrument checks must include calibration verification standards, interelement check standards, and blanks. Specific criteria and frequency are described in the method specific table located in Appendix B.

SW-6020 - Inductively Coupled Plasma/Mass Spectroscopy

On a daily basis, the instrument will be tuned prior to calibration. Alternatively, a laboratory may standardize the ICPMS using a blank and a single standard if instrument sensitivity and linearity can be demonstrated empirically. Other instrument controls include internal standard monitoring, calibration verification standards, interference correction calculation checks, and blanks. Specific criteria and frequency are described in the method specific table located in Appendix B.

2.4.2.2.3 Atomic Absorption

SW-7000 - Total Metals by GFAA

Graphite furnace atomic absorption spectroscopy (GFAA) determines metals present in the acid digestate of a sample. A representative aliquot of a sample is placed into a graphite tube “furnace,” evaporated, charred, and the element of interest atomized. A light beam from a hollow cathode lamp or electrodeless discharge lamp is directed through the furnace, into the monochromator, and onto a detector that measures absorbance.

The instrument is calibrated using a multipoint linear curve on each day of analysis. Instrument performance is monitored using calibration verifications, and blanks. See the method specific table located in Appendix B for specific criteria and frequency.

SW-7470, SW-7471 - Mercury by Cold Vapor AA

Sulfuric acid, nitric acid, and potassium permanganate are added sequentially to a known sample amount. Potassium persulfate is added to each sample, then digested for 15 minutes in an autoclave at 120°C at one atmosphere pressure (alternatively, a water bath may be used to digest samples at 100°C for 2 hours). After cooling, sodium chloride-hydroxylamine sulfate is added to reduce the permanganate. Stannous sulfate is added just prior to aeration of the sample and introduction into the spectrophotometer.

The instrument is calibrated using a multipoint linear curve (5 points) digested with the samples. Instrument performance is monitored using calibration verifications, and blanks. See the method specific table located in Appendix B for specific criteria and frequency.

2.4.2.2.4 Spectrophotometric Methods

EPA-365.2 - Phosphorous, All Forms

Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form a antimony-phosphor-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration measure by absorbance at 650 nm.

SW-7196 - Hexavalent Chromium

This method is applicable to water samples, leachates and digestates (SW-3060). A 9.5 ml aliquot of sample is transferred to a 10 ml volumetric flask and 0.2 ml of diphenylcarbazide solution is added. Enough sulfuric acid (H₂SO₄) is added to adjust the pH to approximately 2, and the sample is diluted to a full volume of 10 ml with ASTM Type II water. After standing for 5-10 minutes for full color development, the absorbance is read in a 1 cm cell at 540 nm. See the method specific table located in Appendix B for specific criteria and frequency.

EPA-335.3/SW-9010 - Cyanide

These methods are applicable to the determination of cyanide in drinking water, surface waters, domestic and industrial wastes and leachates. The cyanide as hydrocyanic acid, is released from cyanide complexes by means of UV digestion and distillation. Cyanides are converted to cyanogen chloride by reactions with chloramine-T which subsequently reacts with pyridine and barbituric acid to give a red-colored complex.

2.4.2.2.5 Ion Chromatography

EPA-300.0/SW-9056 - Anions by Ion Chromatography

These methods are applicable to the analysis of chloride, bromide, fluoride, nitrate, nitrite, ortho-phosphate, and sulfate in drinking, surface and saline waters, and domestic and industrial wastes. The EPA-300.0 method is primarily for drinking waters, and the SW-9056 method has been adapted for the analysis of soil matrix. Anions are determined by introducing a water or leachate sample into an ion chromatograph. The anions of interest are separated and measured using a system comprised of a guard column, separator column, suppressor column and conductivity detector. The system eluent is a Na₂CO₃-NaHCO₃ solution.

Non-aqueous samples may be analyzed by leaching a 10 gram sample for 1 hour with deionized water at a ratio of 1:5 (w/v), filtering, then analyzing the resulting leachate. See the method specific table located in Appendix B for specific criteria and frequency.

Army Method UT04 or equivalent (Organic Acids)

Organic acids are analyzed by IC. This method utilizes a gradient pump, which meters selected eluents at specific rates. The eluents used are ASTM type II water and 200 mM sodium hydroxide. High levels of chloride in a sample may interfere with methyl phosphonic acid (MPA). However, if MPA is an analyte of interest, the gradient program can be modified to help minimize this interference. Isopropylmethyl phosphonic acid (IMPA) is indistinguishable from ethylmethyl phosphonic acid (EMPA) using this method.

2.4.2.2.6 Gravimetric Methods

EPA-160.1 - Total Dissolved Solids

A well shaken 100 ml aliquot of the sample is filtered through a glass fiber filter. The filtrate is then transferred to a preweighed evaporating dish and dried until a constant weight is obtained. The resultant weight of the residue (Filterable residue) is calculated in mg/L.

EPA-160.2 - Total Suspended Solids

A well shaken 100 ml aliquot of the sample is filtered through a preweighed glass fiber filter. The filter is then dried until a constant weight is obtained. The resultant weight of the residue (Non-filterable residue) is calculated in mg/L.

2.4.2.2.7 Miscellaneous Methods

EPA-415.1 - Total Organic Carbon

Non-Purgeable Organic Carbon is determined by the UV promoted oxidation technique. An aliquot of sample is decanted into vials to minimize particulate interference when injected into a reaction vessel containing 2 percent $K_2S_2O_8$ and a UV lamp to promote oxidation. The resulting CO_2 is measured on a NDIR detector and the peak area is integrated by the instrument.

EPA-150.1/SW 9045 - pH

For water samples (EPA-150.1), the pH of the sample is determined with stirring using a combination electrode. The pH meter is calibrated using purchased standard buffers of known pH. For soil samples (SW-9045), a few drops of HCl is added to about 1 gram of sample to test for calcareousness. If the sample effervesces, it is considered calcareous. The sample is mixed (20g:20ml) with either ASTM Type II water or with a 0.01 M $CaCl_2$ solution, depending on whether the soil is calcareous or non-calcareous. The pH of the sample is then determined using a combination electrode as discussed above.

EPA-120.1- Specific Conductance

The specific conductance of a water sample is measured by the use of a self-contained, temperature corrected conductivity meter. A conductance cell and a Wheatstone bridge are used to measure the conductance of the sample as a ratio of the electric current through the cell to the applied voltage. Results are reported in umhos/cm.

EPA-180.1 - Turbidity

This method is based upon the comparison of intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference solution. The higher intensity of scattered light, the higher the turbidity. The standard reference solution used to calibrate the instrument is a suspension of Formazin, prepared under closely defined conditions. Readings are made in nephelometric turbidity units (NTUs).

2.4.4 Preventive Maintenance Program

The objective of a preventive maintenance program (PMP) is to ensure instrument operation is appropriate for project and method DQOs. This PMP focuses on three areas: maintenance responsibilities; maintenance schedules; and inventory of spare parts and equipment.

Maintenance Responsibilities

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

Maintenance Schedules

The effectiveness of any maintenance program depends to a large extent on adherence to specific routine maintenance for each major equipment item. Other maintenance activities may also be identified as requiring attention on an as-needed basis. Manufacturers' recommendations and/or sample throughput provide the basis for the established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g., GC/MS instruments, atomic absorption spectrometers, analytical balances, etc.). Service engineers are employed on the premises to maintain and repair major instrumentation as needed. Maintenance activities for each instrument are documented in a maintenance log. Maintenance schedules and a list of spare parts for the laboratory are listed below.

Spare Parts

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 necessary spare parts. Sufficient equipment is on hand to continue analyses in the event that an instrument encounters problems. In addition to backup instrumentation, a supply of spare parts such as gas chromatography columns, fittings, septums; atomic absorption lamps, mirrors, diaphragms; graphite furnace tubes; and other ancillary equipment is maintained.

2.4.5 Laboratory Data Reduction and Review

Data Reduction

In most cases calculations from raw data are included in discussions of analytical procedures presented in the EPA methods. These data reduction and review procedures will not be presented in this document. Details of data reduction, calibration, and reporting not addressed in the referenced documents are discussed in this section.

Data reduction calculations used for this project are included on the standard reporting forms developed by the laboratory and associated with each individual method or group of methods. Calculations which are not present on standard reporting forms include computer-based data reduction programs. The laboratory is responsible for maintaining a list of these data reduction programs and for being able to demonstrate their validity. Computer programs and spreadsheets developed at the laboratory to aid in the reduction of data must be validated, with appropriate documentation, prior to use. The complete calculation procedures used in computer-based data reduction programs (e.g., GC/MS and GC analyses) are based on the calculation procedures

specified in each method and will not be covered in this document. All information used in the calculations (e.g. raw data, calibrations, tuning records, results of standard additions, interference check results, and blank or background-correction protocols) is recorded in order to enable reconstruction of the final result at a later date. All information regarding the preparation of the sample (e.g. weight or volume of sample used, percent dry weight for solids, extract volume, dilution factor used) is also maintained in order to enable reconstruction of the final result at a later date.

Some instruments are configured to operate independently without computers. For these, the signal is recorded as a strip chart trace, numerical output on a printer strip, or direct reading from a digital or analog dial. In such cases, additional work is required by the analyst to reduce the data to a reportable format. The original signal must be multiplied by a calibration factor or compared with a standard curve. The aliquot result must be divided by the mass or volume of sample to produce a concentration-based final result. Most calculations are carried out on hand-held scientific calculators; simple programs are used for some. All of these data are recorded in a dedicated laboratory notebook or bench sheet for the particular determination in question. Results for single or multiple component tests are hand entered by the analyst in the assigned book.

Some laboratory tests, such as titrations or sensory evaluations, do not have instrumental raw data. For these, the quantitative result or observation is recorded directly in a bound laboratory notebook or bench sheet by the assigned analyst. Calculations like those described above may be needed; calculations used are recorded in the same laboratory notebook.

Data storage and documentation will be maintained using logbooks and data sheets that will be kept on file. Computer acquired data are stored on magnetic tape, floppy disks, or other media, and are generally archived for a period of one year. Paper hard copies of raw data are kept on file for ten years.

Data Review Assessment

The laboratory system for ensuring valid data includes several levels of review. Each level commands specific action to prevent the unqualified release of erroneous data and to correct any problems discovered during the review process.

All analytical data generated at the Contract Laboratory are extensively checked for precision, accuracy, and completeness (a thorough evaluation of representativeness and comparability involves additional data which may not be available to the laboratory). The data validation process consists of data generation, reduction, and three levels of review, as described below.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in

laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. At a minimum the analyst reviews the data package to ensure that:

Sample preparation information is correct and complete:

- Analysis information is correct and complete
- The appropriate SOPs have been followed
- Analytical results are correct and complete
- QC samples are within established control limits; blanks are acceptable
- Special sample preparation and analytical method requirements have been met
- Project-specific requirements have been met

Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, out of control forms, if required, are complete, holding times are documented, etc.).

This initial review step, performed by the analyst is designated Level 1 review. The analyst then passes the data package to an independent reviewer who performs a Level 2 review.

Level 2 review is performed by a group leader or data review specialist whose function is to provide an independent review of the data package. This review is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented
- QC samples are within established guidelines
- Qualitative identification of sample components is correct
- Quantitative results and calculations are correct
- There are no transcription errors
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented, out-of-control forms, if required, are complete, holding times are documented, etc.)
- The data are ready for incorporation into the final report
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed, and all of the analytical results from 10 percent of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is considered complete. If any problems are found with the data package, an additional 10 percent of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety. Errors detected in the review process are referred to the analyst(s) for corrective action. Level 2 data review is documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

Before the report is released to the client, the Laboratory Project Manager reviews the report and chain of custody to ensure that the data meets the overall objectives of the project. This review is labeled Level 3 review. The supporting documentation includes, at a minimum:

- Laboratory name and address
- Sample information (including unique sample identification, sample collection date and time, date of sample receipt, and date(s) of sample preparation and analysis)
- Analytical results reported with an appropriate number of significant figures
- Reporting limits reflecting dilutions, interferences, and correction for dry weight as applicable
- Method references
- Appropriate QC results (correlation with sample batch traceability and documentation)
- Data qualifiers with appropriate references and narrative on the quality of results

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data are consistently of high quality.

Procedures for Handling Unacceptable Data

It is the analyst's responsibility to check the QC information against the project-specific limits for the analysis. When an analysis of a QC sample (blank, spike, check standard, replicate, or similar sample) shows that the analysis of that batch of samples is not in control, the analyst will immediately bring the matter to the attention of the group leader. The group leader will, if necessary, consult with the Laboratory QA Manager and/or the Laboratory Project Manager to determine whether the analysis can proceed, if selected samples should be rerun, or specific corrective action needs to be taken before analyzing additional samples. Out-of-control analyses must be documented. The analyst or group leader will file an "Anomaly Report" with the Laboratory QA Manager for laboratory analysis out-of-control events that require documentation. The Project Chemist will be notified as soon as feasibly possible of any out-of-control events resulting in unacceptable data.

2.5 Quality Control Requirements

Relevant techniques associated with quality control activities for individual protocols will be specified with the description of the particular work process. This may include Program procedures, plans, and project-specific documents. In general, the quality control requirements will be commensurate with the necessary level of rigor needed to provide the appropriate level of confidence in data quality.

2.5.1 Analytical Quality Control Requirements

Analytical or method quality control determines whether a method is performing within acceptable limits of precision and accuracy. There is a laboratory component and a “matrix” component to this determination. The laboratory component measures the performances of the laboratory analytical processes during the sample analyses. The matrix component measures the method performance on a specific matrix. Some quality control elements uniquely measure the laboratory component of method performance but all QC elements measuring the matrix component contain the laboratory component.

Method blanks and laboratory control samples uniquely measure the laboratory component of method performance. Matrix spikes, matrix spike duplicates, laboratory sample duplicates, surrogates, post-digestion spikes measure the matrix component of method performance.

On a project or sampling event level, additional quality control elements are used to assess field sampling techniques and environmental conditions during sample collection and transportation. Field sample duplicates (in contrast to laboratory sample duplicates), field blanks, equipment blanks, and trip blanks are used to assess field precision and accuracy.

2.5.2 Definition of Terms

This sections states the quality control definitions which will be used for work at TEAD.

Detection and Quantitation Limits

Four detection limit terms are used:

- Instrument detection limit (IDL)
- Method detection limit (MDL)
- Limit of Detection (LOD), and
- Limit of Quantitation (LOQ).

The IDL is an empirically derived value which measures the sensitivity of an instrument (in contrast to a method) by repeatedly analyzing standards over several days and multiplying by a factor of three the standard deviation of the instrument response. IDLs are used for metals methods.

The MDL is an empirically derived value used to estimate the lowest concentration a method can detect in a matrix-free environment. SW-846 defines the MDL as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from the analysis of replicate samples of a given matrix, containing analytes, which have been processed through the preparation or extraction procedure. The guidance in 40 CFR136 Appendix B is used to produce MDLs. MDLs are updated by the laboratory annually at a minimum and after significant

instrument maintenance.

The EPA MDL procedure has been criticized as a poor estimator of the DL for the following reasons:

1. It is a single laboratory, short-term estimator that fails to account for analytical bias, changing instrument conditions, or analyst skill.
2. It assumes uniform variance across all possible spike concentrations, failing to account for the fact that variance increases at higher concentrations.
3. It assumes that measured values at the spike concentration are normally distributed. By using this procedure and spiking at very low concentrations, laboratories have been able to calculate MDLs that cannot be achieved in practice.

For the reasons discussed in the previous paragraph, the DoD QSM requires that laboratories verify measures of method sensitivity, in terms of the LOD and LOQ, at least quarterly.

Limit of Detection: Determination and Verification Requirement

A laboratory shall establish a detection limit (DL) using a scientifically valid and documented procedure for each suite of analyte-matrix-method, including surrogates. The detection limit shall be used to determine the LOD for each analyte and matrix as well as for all preparatory and cleanup methods routinely used on samples, as follows:

After each detection limit determination, the laboratory must immediately establish the LOD by spiking a quality system matrix at approximately two to three times the detection limit (for a single-analyte standard) or one to four times the detection limit (for a multi-analyte standard). This spike concentration establishes the LOD. It is specific to each combination of analyte, matrix, method (including sample preparation), and instrument configuration. The LOD must be verified quarterly. The following requirements apply to the initial detection limit/LOD determinations and to the quarterly LOD verifications.

- The apparent signal to noise ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second-column confirmation, or pattern recognition.) For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentrations.
- If a laboratory uses multiple instruments for a given method the LOD must be verified on each.
- If the LOD verification fails, then the laboratory must repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.

- The laboratory shall maintain documentation for all detection limit determinations and LOD verifications.

Limit of Quantitation: Establishment and Verification of Requirement

For DOD projects, the LOQ must be set within the calibration range prior to sample analysis. At a minimum, the LOQ must be verified quarterly.

The laboratory procedure for establishing the LOQ must empirically demonstrate precision and bias at the LOQ. The LOQ and associated precision and bias must meet client requirements and must be reported. If the method is modified, precision and bias at the new LOQ must be demonstrated and reported.

All reported LOD, LOQ and sample reporting limits (SRL) shall be adjusted for the size of sample aliquots, concentration/dilution factors, and percent solids.

Project-Specific Requirements for Method Sensitivity

Environmental data is used to accomplish one or more of the following tasks:

- Determine whether a chemical substance is present in an environmental sample at or above some threshold value or action level;
- Verify that a pollutant concentration remains below a permit limit;
- Evaluate potential risks to human health or the environment;
- Monitor changes in concentrations of contaminants; or
- Determine the effectiveness of remediation activities.

As defined in the DoD QSM, the RL is the lowest concentration value that meets project requirements for reporting quantitative data with known precision and bias for a specific analyte in a specific matrix. The project specific RLs need to be set to support the DQOs for collection of that data.

Documenting Uncertainty for Low-Concentration Data

Detection and quantitation limits are laboratory specific. Steps must be taken to document measurement uncertainty for low concentration data as follows:

- The laboratory must provide its DL, LOD, and LOQ with associated precision and bias for each target analyte, in each matrix of concern (e.g., reagent water, clean sand, etc.), and

verify that these values meet project-specific RLs. The laboratory SOPs for establishing the DL and for establishing and verifying the LOD and LOQ must be reviewed.

- The laboratory must verify the LOD by processing an LOD verification check sample with each batch of samples. This is a quality control sample that is spiked at a concentration at or slightly above the LOD to evaluate whether the analyte of interest is in fact “detectable” in the matrix of interest. To confidently report non-detects, for TEAD projects the reporting for non-detects is less than the LOD.
- If the project-specific RL is near the LOQ, the laboratory must verify the LOQ in the project-specific matrix by analyzing a minimum of four replicate samples with known concentrations at the LOQ.
- The raw data (e.g., chromatograms) must be reviewed for low-concentration data. If a result is reported above the DL, the signal-to-noise ratio must be at least 3.
- Sample results must be compared with blank results. If sample results (including chromatograms) cannot be distinguished from blank results, then they are not meaningful.

Batch

Many analytical laboratory processes are batch process and there the batch is a basic unit for the frequency of some quality control elements. Two types of batches can be identified: the preparation and instrument batch. A preparation batch (herein referred to as “batch”) is defined as a group of twenty or less samples which are prepared (e.g., extracted or digested) within the same time period or in limited continuous sequential time periods. Keeping batches “open” over several hours or days is not permissible; samples and their associated QC samples must be prepared in continuous process. The preparation batch consists of twenty or fewer environmental samples and the associated QC samples: method blank, Laboratory Control Sample (LCS), matrix spike, and matrix spike duplicate or sample duplicate. Samples in each batch are of similar matrix (e.g., soil, sludge, liquid waste, water), are treated in a similar manner, and use the same reagents.

The instrument batch is a group of twenty or less samples which are analyzed together within the same analytical run sequence or in continuous sequential time periods. In general, if an instrument is not used for periods of time or shut down (e.g., overnight) then a new instrumental batch must be started.

For volatile organics analyses (VOA) by GC or GC/MS the preparation and instrument batch definitions become less distinct since the sample preparation (purge and trap) is performed as

part of the instrumental analysis and sample preparation is more of a sequential, rather than batch, process. For the purpose of QC frequency, VOA GC and GC/MS batches are defined as twenty or less samples analyzed within a calibration (and for GC/MS, tune) time period, or within sequential continuous calibration time periods.

In general, preparation batches should be analyzed together, as a unit, within the same instrument batch. If samples from the same preparation batch are not analyzed within the same instrument batch (e.g., because of dilution requirements or matrix interference) the following is required:

- All samples from the preparation batch must be clearly associated with their corresponding preparation batch QC samples, and appropriate corrective actions must be performed on all samples in the batch, based on the results of the associated preparation batch QC.
- All instrument QC for each instrument batch (initial and continuing calibrations, instrument blank analyses, and tuning, etc.) must meet the established criteria for the method.
- Instrument cleanliness must be proven through the analysis of an instrument blank, the preparation batch blank, or a preparation blank from another batch. (The preparation batch LCS and MS/MSD need not be analyzed on additional instruments.)
- When preparation batches must be split among instruments to meet expedited turn around times or to meet other project requirements, each instrument batch needs to contain quality control elements equivalent to the quality control elements available in single instrument batch analyses.

When the terms (preparation) batch or instrument batch are used in this document, they are used as defined above.

Method Blank

A method blank is used to monitor the laboratory preparation and analysis systems for interferences and contamination from glassware, reagents, sample manipulations, and the general laboratory environment. The method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing, and which is taken through the entire sample preparation process. A method blank is included with each batch of samples. Some inorganics methods do not have a distinct preparation, and for these tests, the instrument blank, which contains all reagents used with samples and is equivalent to the method blank, is considered to be the method blank.

Instrument Blank

An instrument blank is used to monitor the cleanliness of the instrument portion of a sample analysis process. Instrument blanks consist of the solvent or acid solution of the standard used to calibrate the instrument. With an exception for metals analyses, instrument blanks are analyzed each instrument batch whenever a method blank is not analyzed in that instrument batch.

Routine metals analyses receive an instrument blank every ten samples. Instrument blanks are also analyzed on an as-needed basis for troubleshooting.

Laboratory Control Samples (LCS)

Laboratory control samples are well-characterized, laboratory generated samples of a known matrix used to monitor the laboratory analytical process independent of matrix effects. LCS samples are spiked with a known quantity of specific target analytes. Sodium sulfate and/or other approved matrices may be used for LCS preparation. LCSs are taken through the entire sample preparation and analytical process. LCSs measure laboratory performance regarding the accuracy of the preparation process by measuring spiked target analyte recoveries in a controlled matrix or matrix-free sample. LCSs are prepared and analyzed with each batch of samples. LCS results, together with matrix spike results, can establish the presence of matrix effects. The LCS spike compounds are given in the method specific tables. For methods where there is no distinct preparation, a continuing calibration standard may be used as the LCS, if it meets all LCS and matrix-matching criteria.

Laboratory Control Sample Duplicates (LCS/LCSD)

Duplicate laboratory control samples are two LCS prepared and analyzed together. Accuracy (recovery) and batch precision may be determined when LCS/LCSD are used. LCS/LCSD are used when not enough sample is available to prepare a matrix spike and matrix spike duplicate for a batch.

Matrix Spikes and Matrix Spike Duplicates (MS/MSD)

Matrix spikes measure matrix specific method performance. A matrix spike sample is prepared by adding a known quantity of target analytes to a sample prior to sample digestion or extraction. The MS and MSD samples are then analyzed with a third aliquot of the sample which remains unfortified. The accuracy of the matrix specific method performance may be determined by the recovery of the spiked analytes after native concentrations of the spike analytes are subtracted. If a matrix spike duplicate (MSD) is analyzed, the matrix specific precision of the method may be calculated. In general, for organics and inorganics analyses, an MS/MSD pair are prepared and analyzed with each batch. Some methods are not amenable to the spiking of target analytes into the sample matrix (i.e, pH). Precision will be determined using a sample duplicate as described below.

Laboratory Sample Duplicates (SD)

For laboratory sample duplicate analyses, a sample is prepared and analyzed twice. The matrix specific method precision may be calculated by dividing the difference in the results by the average. Laboratory sample duplicates are prepared and analyzed with each batch of samples for inorganic analyses. For inorganic analyses the matrix spike RPD limits found in the method-specific Tables in Appendix B will be applied to the sample duplicate results. Organic analyses use MSD to obtain precision data. Corrective actions are described in the tables.

Surrogate Compounds

GC and GC/MS analyses include the addition, subsequent quantitation, and ultimate recovery calculation of surrogate compounds. Surrogate standards help to monitor both performance of the analytical system and the effectiveness of the method in dealing with each sample matrix. Surrogate compounds are:

- Compounds not requested for analysis
- Compounds that do not interfere with the determination of required analytes
- Compounds that are chemically similar to the required analytes, yet are not naturally occurring
- Compounds exhibiting similar response to analytes under determination.

Surrogate compounds are added to every sample and QC sample at the beginning of the sample preparation, and the surrogate recovery is used to monitor matrix effects and sample preparation. Surrogate control criteria are applied to all samples, QC samples, and method blanks. Re-analysis and re-extraction may be performed if surrogate criteria are not met. Specific method surrogates, the recovery acceptance windows, and the control logic are given in the method specific descriptions.

Internal Standards

Internal standards are compounds which analytically behave similarly to the target analytes. Internal standards are compounds not found in the sample, are added at the time of instrumental analysis, are used to quantitate results, and are used to correct for injection variability. Mass spectrometer methods use internal standards. Mass spectrometer methods have control limits on internal standard areas.

2.5.3 Laboratory Batch Quality Control Logic

Frequency of batch quality control

For organics analyses each batch will contain a method blank, an LCS, and an MS/MSD pair.

For some inorganics analyses, each batch will contain a method blank, an LCS, a MS, and a SD. For VOA GC/MS analyses, a method blank will be analyzed during each 12 hour tune.

For each shipment of twenty samples to the contract laboratory, one sample will be provided in sufficient quantity such that an MS/MSD can be analyzed in addition to actual sample analysis.

Batch Quality Control Logic

This section provides a general description of batch control logic and corrective actions which will be used. Required batch quality control samples for each analytical method is detailed in the method specific tables located in Appendix B. Analytical batches will be controlled by method blank and LCS results. For analyses which are amenable to matrix spiking, MS/MSD recoveries and RPD will be reviewed for systematic trends or errors which may be representative of the batch, as well as the effect of the matrix on method performance, and may result in corrective action for the batch. The sample chosen for MS/MSD analysis, therefore, should be representative of the other samples in the batch and only project specific field samples will be used for MS/MSD procedures. Samples used for MS/MSD analysis will be designated in the field and is identified on the COC. Surrogate recoveries will be reviewed for matrix effects as well as individual sample errors. For analyses which do allow matrix spiking, sample duplicates will be analyzed to measure precision.

The method blank measures laboratory introduced contamination for the sample batch and batch corrective action is initiated when contamination is found; this may include re-analysis of the blank, re-analysis of the samples, re-preparation and re-analysis of the blank, QC, and samples, and assessment of the impact of the contamination on batch sample data. Although it is a goal to have no detected target analytes in the method blanks, analytes may be periodically detected in blanks due to the nature of the analysis or the reporting limit for the analyte. For example, in organic volatile analyses methylene chloride, acetone, and 2-butanone (MEK) may sometimes be found in the blank, and in organic semi-volatiles analyses, the phthalate esters may sometimes be found in the blank. In instances where DQOs require reporting samples to the MDL, background levels of contaminants are likely to be detected.

The method blank definition in SW-846 states that no target analytes above the MDL should be detected in the method blank. This must be the goal of the laboratory but program specific requirements allow for batch acceptance when there is no blank contamination above one half the PQL. Blank acceptability may be project-specific so that project DQOs may be balanced with analytical capabilities.

The first step of corrective action is to assess the effect on the samples; for example, if an analyte is found only in the blank but not in any batch samples, or if the analyte in the blank is less than one tenth the value in the sample, re-extraction and re-analysis of the batch may not be necessary. Investigating and eliminating the source of the contamination and documenting the

evaluation would be the appropriate action. Blank subtraction is not allowed (unless required by the applicable method). During analysis, the method blank, and any samples containing the same contaminant, would be re-analyzed, and if the contamination remains, the contaminated samples of the batch would be re-extracted and re-analyzed with a new blank and QC. The Program Chemist will be contacted if batch re-preparations do not lead to method blanks which meet the above criteria.

LCS are evaluated by comparing the recovery of spiked target analytes to the recovery windows given in the method-specific tables contained in this document. For organic analyses the LCS are spiked with a set of compounds representative of the target analyte list and for inorganic analyses LCS are spiked with all target analytes. The analytes spiked into the LCS are listed in the method specific tables. When a limited spike list is used, all spiked compounds must be within the recovery windows for the batch to be considered acceptable and when a full spike list is used, a batch may be considered acceptable for those analytes which had acceptable recoveries in the LCS. If analytes are outside of the acceptance windows, corrective action must be initiated.

The first step of the corrective action process is to evaluate the effect on the samples; for example, if an analyte in the LCS has a recovery above the upper acceptance window, and other QC elements of the batch and sample analysis indicate that other samples in the batch do not have detectable concentrations of target analytes, re-extraction of the batch may not be necessary, otherwise, reextract and reanalyze affected samples. Corrective action would consist of an attempt to locate the cause of the non-conformance and documenting the evaluation in the laboratory report narrative. However, if recoveries in the LCS are sufficiently above the acceptance window to cause the analyst to suspect a systematic error, then the batch must be rejected and the preparation repeated. An example of a systematic error may be inexplicable double percentage recoveries as a result of a concentrating spike solution. As a guideline, when samples results are all non-detect and recoveries exceed the acceptance window by greater than 25%, then the analysts should investigate other causes contributing to the high recoveries. In general, if recoveries of a compound or element spiked into the LCS is in excess of the upper control limit and associated sample results are non-detect then corrective action may not be necessary; if associated results are positive however, corrective action must be taken. In addition, if a compound or element spiked into the LCS has an unacceptable recovery with respect to the lower control limit then corrective action must be taken. If a compound or element spiked into the LCS has an unacceptable recovery, the LCS, Blank, and all associated samples must be re-extracted and reanalyzed. When the LCS has a representative spike list and when a full target analyte spike list is used, the batch samples, blank and LCS, must be reprepared and reanalyzed for the failed analytes.

For those analyses which do not allow matrix spikes, an LCS and sample duplicate will be analyzed with each batch of samples. Batch control will be the same as that described for LCS.

The within-batch precision is measured by calculating the relative percent difference (RPD) of any target analytes found in the primary and duplicate analysis of the sample. The acceptance windows for LCS accuracy, and the associated corrective actions for failed QC, are given in the method-specific tables in this document.

Matrix Spike and Matrix Spike Duplicate Evaluation

For those methods which are amenable to matrix spikes, an MS/MSD pair is analyzed with each batch of samples for organic analyses, and for inorganics analyses, a MS and a laboratory sample duplicate are analyzed with each batch of samples. Both organic and inorganic batches are evaluated for matrix precision and accuracy. Accuracy is evaluated by calculating the recovery of spiked analytes and precision is evaluated by calculating the relative percent difference (RPD) of the recoveries. The recovery and RPD are compared to the acceptance limits given in the method specific tables. In the event that a matrix spike analyte fails precision or accuracy criteria, corrective action must be initiated.

Matrix spike data evaluation is more complex than blank or LCS data evaluation since matrix spikes measure matrix effects in addition to sample preparation and analysis effects. The heterogeneity of soil, grab samples, and sequentially collected water samples further complicates the evaluation since matrix specific accuracy and precision assume that the native concentration in the three sample analyses is constant. However, appropriately trained personnel aware of the data's end use may improve data quality by an evaluation of matrix spike data. In consideration of these limitations, the laboratory will not qualify data based on matrix spike performance but will perform corrective actions as outlined below.

When an MS/MSD pair fail in accuracy or precision for any spiked analyte, the impact on the associated batch will be evaluated. If there is significant evidence that the sample matrix interferes with the precision and accuracy assessment (i.e. significant chromatographic peaks interfere with target analyte identification in a GC analysis, or poor post-spike recovery occurs for a metals analysis, or sample is visibly non-homogeneous) this evidence will be documented and included in the laboratory report and clearly described in the case narrative. If chromatographic interference is cited as a cause for poor recovery or precision, then a copy of the chromatogram will be included in the final data report. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may contribute a significant error to the precision and accuracy calculations making the accuracy and precision measures unrepresentative of the true method and matrix performance. For this reason, if the native concentration is four or more times the spiked concentration, the MS/MSD are not required to meet the control criteria. In these situations, no other corrective action may be necessary.

If an MS/MSD pair fail to meet accuracy or precision criteria and no significant non-target analyte interference exists, the original MS/MSD extract is re-analyzed once. If the re-analysis produces an acceptable result, only the re-analysis will be reported if it was performed within holding time. If the second analysis of the original MS/MSD extract does not meet acceptance criteria, re-extraction and re-analysis of the MS/MSD will be performed and evaluated. If re-analysis still fails to meet accuracy or precision criteria or did not meet the analytical holding time, then results from both MS/MSD analyses are reported.

Sample duplicates will be evaluated for precision in the same manner, and corrective actions will be performed as indicated in the method specific tables.

The failure of a matrix spike, spike duplicate, and/or sample duplicate analysis to meet the established control criteria will additionally result in an evaluation of the batch for systematic errors which may have affected the batch. Other information such as surrogate recoveries and the appearance of chromatograms (GC and GC/MS), post spike recoveries (metals), method blank and LCS results, expected or detected analyte concentrations, the appearance of samples or extracts, and the results of other analytical tests may be considered in this evaluation. In all situations, the evaluation and corrective actions performed will be clearly and completely documented in the laboratory report case narrative.

Additional Methods of Matrix Spike Evaluation

For inorganics methods such as metals by GFAA or ICP, additional procedures may be used by the analyst to ascertain physical or chemical interferences inherent in the sample matrix. The matrix spike sample may be serially diluted until the percent recovery is within control limits or the analyst may perform a post-digestion spike on the unspiked matrix sample then perform additional corrective actions. These procedures should be used when the matrix spike recoveries are outside project-specified control limits, there is no other apparent reason for the outlier, and the analyst chooses to cite matrix interference as the cause for anomalous recoveries.

In the absence of other guidance, analysts will evaluate post-digestion spike recoveries as follows:

- If the %R of the post-spike is within $85\% \leq \%R \leq 115\%$ and the sample result is $< \text{LOQ}$ or $\geq \text{LOQ}$, report the result.
- If the %R of the post-spike is $115\% \leq \%R \leq 150\%$, and the result is $< \text{LOQ}$, report the result.
- If the %R of the post-spike is $115\% \leq \%R \leq 150\%$ and result is $\geq \text{LOQ}$, dilute and reanalyze. Quantitate by the method-of-standard-addition if necessary.
- If the $\%R \geq 150\%$ and the result is $< \text{LOQ}$, verify that there are no errors in spiking, and

report the result.

- If the $\%R \geq 150\%$ and the result $> LOQ$, dilute and reanalyze. Quantitate by the method-of-standard-addition if necessary.
- If the post-spike recovery is $40\% \leq \%R \leq 85\%$ and the sample result is $< 0.5 \times LOQ$, report as “not detected” at the LOQ.
- If the post-spike recovery is $40\% \leq \%R \leq 85\%$ and the sample result is $\geq 0.5 \times LOQ$, dilute and reanalyze. Quantitate by the method-of-standard-addition if necessary.
- If the post-spike recovery is $< 40\%$ and the sample result is $< LOQ$ or $\geq LOQ$, dilute and reanalyze. Raise the reporting limit accordingly.

Labeled isotopes or internal standards added and used as described in coupled mass spectroscopy methods may also serve to indicate the presence of a matrix interferent. Refer to the method specific table located in Appendix B for specific evaluation procedures and criteria.

Laboratory Batch Quality Control for Field, Equipment and Trip Blanks

The section below outlines the quality control applied to trip blanks, field blanks, and equipment blanks from sampling equipment.

Trip blank vials are sent with empty sample containers to the field and are shipped back to the laboratory with field samples to measure potential contamination from storage, collection, and shipment in the field and laboratory. Field blanks are created in the field and are intended to measure background contamination in the field. Regardless of the matrix of the project samples, trip and field blanks are reagent water and are usually only analyzed for volatile contamination. Trip and field blanks may be processed without site- specific matrix spike analyses. These blanks may be processed with matrix spikes or laboratory sample duplicates from another site, if the matrix adequately matches the matrix of the blank. Environmental samples will be utilized for matrix spikes. If matrix spikes or sample duplicates from another project are not available, these blanks may be analyzed with two LCS.

Equipment blanks assess the decontamination procedures of the field sampling equipment, and consist of reagent water, or water known to be free of target analytes. Equipment blanks are analyzed for all the parameters which are to be performed on the associated samples. Equipment blanks from soil sampling equipment are processed in the manner described above for the field and trip blanks. Equipment blanks from water sampling equipment are processed in the same manner as the associated field samples, with the laboratory batch quality control described above, since their matrices are compatible.

2.5.4 Laboratory Data Completeness

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under correct, normal conditions.

The target for completeness is 90 percent for all parameters except for holding times and sample preservation for which the target value is 100%. Data completeness is a measure of the extent to which the database resulting from a measurement effort fulfills objectives for the project. For this project each analytical procedure and sample has a target of 90% completeness and will be defined as the percentage of valid data requested.

$$C\% = S/R (100\%)$$

where:

C = completeness

S = number of successful analyses

R = number of requested analyses

Successful analyses are defined as those where the samples arrived at the laboratory intact, properly preserved, in sufficient quantity to perform the requested analyses, and accompanied by a completed chain of custody. Furthermore, the sample must be analyzed within the specified holding time and in such a manner that analytical QC described in this document are met. Factors that adversely affect completeness include:

- Receipt of samples in broken containers
- Receipt of samples in which chain of custody or sample integrity is compromised in some way
- Samples received with insufficient volume to perform initial analyses or repeat analyses, if initial efforts do not meet QC acceptance criteria
- Improperly preserved samples
- Samples held in the field or laboratory longer than expected, thereby jeopardizing holding time requirements
- Failure to reextract and reanalyze as required.

Despite strict adherence to a quality assurance plan, errors may occur in laboratory and field operations. While the laboratory shall strive to achieve the highest level of completeness possible, the following level of completeness will be the minimum acceptable: at least 90 percent of all analytical methods will have acceptable quality control.

Completeness for the entire project also involves completeness of field and laboratory documentation, whether all samples and analyses specified in the workplans have been processed, and the procedures specified in the, CDQMP and SAP have been implemented.

2.6 Instrumentation Calibration and Frequency

2.6.1 Standards

The accuracy of sample target analyte quantitation is directly related to the accuracy of the standards used for instrument calibration. To ensure the highest quality standard, primary reference standards used by Contract Laboratory are obtained from reliable commercial sources. Inorganic standards must be traceable to the National Institute of Standards and Technology (NIST) and organic standards must be traceable to NIST, or American Association of Laboratory Accreditation (A2LA) vendors when available. When standards are received at the laboratory, the date received, supplier, lot number, purity and concentration, and expiration date are recorded in a standard preparation log book. Vendor certifications sent with the standards are also filed and are available upon request.

Standards purchased by Contract Laboratory may be in a pure form, in a stock, or working standard solution. Often dilutions are made from vendor standards. All standards made are given a standard identification number and have the following information recorded in a standards log book: source of standard used to prepare dilution; preparer's initials; initial concentration; final concentration; solvent; source and lot number of solvent; volume of final solution; volume of standard diluted. Records must unambiguously trace the preparation of standards, their use in calibration, and the quantitation of sample results. After preparation and before routine use, the identity and concentration of standards are verified. Verification procedures include a check for chromatographic purity (if applicable) and verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source. Reagents are also examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory. Standards are routinely checked for signs of deterioration (e.g., discoloration, formation of precipitates, and changes in concentration) and are discarded if deterioration is suspected or their expiration date has passed. Expiration dates may be taken from the vendor recommendation, the analytical methods, or from internal research.

2.6.2 Instrument Calibration

This section discusses general requirements for instrument calibration and standards preparation and traceability. Test specific calibration details for the methods are given in the method specific tables.

Calibration is a reproducible reference point to which all sample measurements can be correlated. Instrumentation calibration is necessary for accurate sample quantitation. Calibrations establish the dynamic range of an instrument, establish response factors to be used

for quantitation, and demonstrate instrument sensitivity. Criteria for calibration are method specific, are taken from the published analytical methods, and are executed as described in each method specific table found in Appendix B. Accurate sample quantitation also relies on accurate standards. Standard accuracy may be established by tracing the quantitation standard to a source of known and documented quality or by comparison of standards from different sources. Instrument calibrations and standards are unambiguously documented so that the process of calibration can be re-created.

2.6.2.1 Organic Methods Calibration

The field of chromatography involves a variety of instrumentation and detection systems. While calibration requirements vary depending on the type of analytical system and methodology, the following principles of calibration generally apply: calibration occurs before any sample analysis; initial multipoint (five or more points) calibrations are performed prior to analysis and periodically as necessary; daily calibration verification standards are analyzed prior to sample analysis; and continuing calibration standards are analyzed at a specific frequency (every ten analyses) throughout the sample analysis. Sample quantitation must be based on the initial calibration. GC/MS and non-GC/MS chromatographic methods base quantitation on the initial multipoint calibration. Sample quantitation may be with an external calibration technique or an internal standard calibration technique. Quantitation by external calibration involves the measurement of an analyte's response in a sample compared to the instrument response obtained from a known reference standard. Internal standard calibration techniques require one or more internal standards to be spiked in all samples and standards and then quantitate target analytes relative to the internal standard response. Internal standard techniques are used for GC/MS methods and may be used for GC methods. The method-specified criteria for the performance and response of internal standards must be met to assure accurate quantitation. All samples must be bracketed by continuing calibration standards which meet the established criteria.

Gas Chromatography

This section discusses general calibration techniques for non-GC/MS methods such as SW-8021, SW-8015, SW-8081, SW-8082, SW-8141, SW-8151, and SW-8310. External or internal standard calibration techniques may be used for calibrating the gas chromatograph.

Initial calibrations are performed upon initial instrument set up, failure of the daily, or continuing standard, and upon any major change in the system. However, before initial calibrations are performed, the instrument operating conditions are verified, any routine preventative maintenance is performed, and an instrument blank is analyzed to test for, or show the absence of, interferences. The initial five point calibration consists of a standard containing each analyte of interest at five concentration levels for SW-846 8000 series methods. One of these standards must be at the LOQ). The other standards should bracket the expected concentrations in real

samples, but not exceed the working linear range of the detector being used. From the initial calibration, calibration factors are calculated for each analyte of interest to evaluate the system performance. For target analytes with multiple peaks, such as PCBs, diesel, gasoline, and toxaphene, the total area may be summed and used for the area.

The CF is used to evaluate instrument response linearity for each analyte of interest across the calibrated range. Linearity is determined by the correlation coefficient, r , or the percent relative standard deviation (%RSD) of the best-fit line. If the %RSD of the calibration factor is less than 20% over the working range, linearity through the origin can be assumed, and the average CF can be used. For SW-846 methods the %RSD must be less than or equal to 20%, or the correlation coefficient, r , must be greater than or equal to 0.995. The use of r or %RSD must be uniformly applied to a calibration sequence and instrument.

The initial calibration is checked at least daily by injecting a daily calibration standard. This standard is usually the mid-range standard of the initial calibration and is injected before any samples or method blanks are analyzed. The percent difference (%D) is calculated and should be within ± 15 percent of the average response factor of the initial calibration curve or the quantitated value should be within 15% of the expected value. A continuing calibration standard is analyzed every ten analyses and at the end of an analytical run to further evaluate system performance. All samples must be bracketed by continuing calibration verification standards which meet the established criteria. The %D of the continuing calibration standards must either meet the same criteria as the daily standard or be within ± 15 % of the expected concentration using the average CF from the ICAL. Occasionally, an analyst may acquire an ICV or CCV where the %D is greater than the 15% window. It is the responsibility of the analyst to evaluate the standards for any adverse trends and to evaluate the data for acceptability. For example, an analyst may deem that sample data are reportable when a single, closing CCV has a %D of +25% and all of the bracketed samples have no reportable analytes.

Gas Chromatography/Mass Spectrometry (GC/MS)

Every 12 hours, prior to calibration or sample analysis, the mass spectrometer must be tuned. For volatiles methods, bromofluorobenzene (BFB) is used and for semivolatile methods, decafluorotriphenylphosphine (DFTPP) is used. The resultant mass spectra for BFB and DFTPP must meet all of the method-specified criteria before sample analysis begins. These criteria are demonstrated each 12 hour shift. Tuning criteria are given in the methods and are stated in the method specific requirements in the Appendix F tables.

Initial calibrations are performed upon instrument setup, failure of the continuing standard, or upon any major change in the system. Initial calibrations for SW-846 methods use at least five calibration concentrations with the lowest standard at or near the method reporting limit. Initial calibrations must contain all analytes of interest and contain internal standards. The initial

calibration is evaluated at least once each 12 hour shift by checking the response of certain key compounds referred to as System Performance Calibration Compounds (SPCC) and Calibration Check Compounds (CCC). The SPCC evaluate system sensitivity and the CCC evaluate system linearity. A relative response factor (RF) is calculated for the analyte of interest relative to the internal standard whose retention time is closest to that compound.

From the RF at each concentration an average RF is calculated. The SPCC are checked for a minimum average RF and the CCC are checked for maximum percent relative standard deviation (%RSD) of their RF across calibration concentrations.

After the initial calibration has been found acceptable, before sample analysis, and every 12-hours during sample analysis, a tuning standard and calibration standard must be analyzed. The initial calibration curve is verified by the analysis of a continuing calibration verification standard that is at a concentration near the midpoint concentration for the working range of the GC/MS. The acceptance criteria for continuing standards is based on SPCC and CCC criteria, retention time criteria, and internal standard area criteria. SPCCs are checked for instrument sensitivity and CCC compounds are checked for daily drift from the average RF of the initial calibration. The method-specified minimum RF for the SPCCs and the method-specified %D requirement for CCCs must be met, or corrective action must be performed, prior to sample analysis. The internal standard retention times in the continuing calibration standard must be within ± 30 seconds of the previous continuing calibration standard and the internal standard areas must be within a factor of two from the last continuing calibration standard. Samples are quantitated in accordance with the method using linear curve fitting routines only.

If any criteria are failed during initial, continuing, or tuning calibration, corrective action must be taken before sample analyses may proceed.

2.6.2.2 Metals Methods Calibration

The most frequently used methods for environmental metals analysis use either GFAA or ICP emission spectroscopy. The calibration standards used by these methods are initial calibrations, initial calibration verifications (ICV), initial calibration blanks (ICB), continuing calibration verifications (CCV) and continuing calibration blanks (CCB).

Inductively Coupled Plasma (ICP)

The ICP is calibrated daily prior to any sample analyses using criteria prescribed in the analytical method. It is preferable that the ICP be standardized using a curve comprised of a blank and three standards. An acceptable alternative to the use of a multipoint curve would be to standardize the instrument using a blank and a single standard. After standardization, a contract reporting limit solution (CRI) at the PQL and the high calibration standard are analyzed. The CRI should be $\pm 50\%$ of the expected response. Concentration values of the upper range

standard should not deviate from the known concentration by more than $\pm 5\%$. The calibration is then verified (ICV, CCV) using a standard solution from an independent source. The ICV and CCV values must fall within $\pm 10\%$ of the true value for analysis to continue. The working range of the instrument is established daily with the high level calibration standard and sample quantitation may not be performed outside this linear range.

The calibration is monitored throughout the day by analyzing a continuing calibration blank (CCB) and a continuing calibration verification standard (CCV). If the verification standards or blank do not meet established criteria, the analysis is stopped and corrective action must be performed prior to the analysis of samples. All samples must be bracketed by CCBs and CCVs which meet the established criteria. The corrective action procedures include reanalyzing samples back to the last acceptable calibration check.

An inter-element check standard is analyzed at the beginning and end of each analytical run on the ICP to verify that inter-element and background correction factors have remained constant. Results outside of the established criteria trigger reanalysis of samples. The calibration blank solution is flushed through the system for at least one minute before the analysis of each sample.

Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

The ICP/MS is tuned (optimized) according to method specifications daily. Mass calibration and resolution checks are also performed daily, and must meet the method criteria prior to sample analysis. The ICP/MS is calibrated daily prior to any sample analyses using criteria prescribed in the analytical method. The calibration is then verified using a standard from an independent source.

A calibration is established daily by analyzing a minimum of two standards, one of which is a calibration blank. The calibration standard and blank include internal standards which may be used to correct for matrix interferences. Naturally occurring isobaric interferences are automatically corrected by the instrument software and is verified by analysis of an interference check standard every 12 hours of analysis. The calibration is monitored throughout the day by analyzing a CCB and a CCV, every ten analyses. All samples must be bracketed by CCVs and CCBs which meet the established criteria. If the verification standard and blank do not meet established criteria, the analysis is stopped, and corrective action must be performed prior to the analysis of samples. The corrective action procedures include recalibration and reanalysis of samples back to the previously acceptable calibration check. A rinse blank solution (containing no internal standards) is flushed through the system between samples to prevent carry-over.

Atomic Absorption (AA)

Each AA unit is calibrated prior to any analyses being conducted. A calibration curve is prepared with a minimum of a calibration blank and three standards and then verified with a

standard that has been prepared from an independent source at a concentration near the middle of the calibration range. The calibration is then verified every ten injections on an ongoing basis with a CCB and a CCV. All samples must be bracketed by CCBs and CCVs which meet the established criteria. If the ongoing CCV and CCB do not meet established acceptance criteria, the analysis is stopped and corrective action must be performed prior to analysis of samples. The corrective action procedures include reanalysis of samples back to the previously acceptable calibration check. For GFAA, all samples are spiked after digestion to evaluate matrix effects or interferences. The method of standard additions or sample dilution is used when matrix interferences are present as determined by the results of the analytical spike. As prescribed by the determinative methods, chemical matrix modifiers are added to the digestates to reduce the effects of interferences contributed by the matrix.

2.6.2.3 Wet Chemistry and Other Methods Calibration

The field of conventional, non-metals analysis (wet chemistry or general chemistry) involves a variety of instrumental and wet chemical techniques. While calibration and standardization procedures vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply universally. Each system is calibrated prior to analyses being conducted. Calibration consists of defining the working range by use of a series of standard solutions (usually 4 or 5 standard levels) and identifying potential interferences. The calibration is checked on an ongoing basis (every ten analyses) to ensure that the system remains within specifications. If the ongoing calibration check does not meet established criteria, analysis is stopped and corrective action must be performed prior to the analysis of any samples. The corrective action procedures include examination of instrument performance and analysis information, consultation with the group leader, and a decision path to determine if recalibration and reanalysis of samples back to the previous acceptable calibration check is warranted. In general, the analyst must reanalyze samples back to the last acceptable calibration check. Continuing calibrations are not performed for non-instrumental methods such as Total Dissolved Solids.

2.6.2.4 Analytical Calibration and Result Calculations

2.6.2.4.1 Calibration Calculations

For all laboratory analyses, the analytical system is calibrated using either an external or internal standard technique.

External Standard

For the external standard technique calibration standards containing each analyte of interest are prepared at concentrations required in the method. The least concentrated standard will be at a

concentration corresponding to the method detection level (MDL). The remaining standards define the working range of the instrument. For each analyte at each standard concentration a calibration factor (CF) or a response factor (RF) is calculated.

The CF or the ratio of the response to the amount injected is calculated.

$$CF = (A_s)/(M_s)$$

where:

A_s = Response for the analyte to be measured.

M_s = Mass of standard injected (in nanograms).

The RF or ratio of the standard concentration to the response is calculated:

$$RF = (C_s)/(A_s)$$

where:

C_s = Concentration of the analyte in the standard.

A_s = Response for the analyte to be measured.

Internal Standard

The internal standard technique is used for gas chromatography/mass-spectroscopy (GC/MS) analyses and is similar to the external standard technique except that one or more internal standards (compounds that exhibit similar chemical and analytical behavior to the compounds of interest and are not present in the sample) are added to each calibration standard. For each analyte, at each standard concentration, the ratio of the response to the concentration for each analyte and its corresponding internal standard, defined as the relative response factor (RRF) is calculated.

$$RRF = (A_s C_{is}) / (A_{is} C_s)$$

where:

A_s = Response for the analyte to be measured.

A_{is} = Response for the internal standard.

C_{is} = Concentration of the internal standard

C_s = Concentration of the analyte to be measured.

For each analyte the percent relative standard deviation (%RSD) of the five calibration factors must be less than or equal to a QC limit, which allows the average CF, RF, or RRF to be used for calculation of analyte concentrations. If the %RSD of the CF or RF is greater than the QC limit over the calibration range, then linearity through the origin cannot be assumed.

When performing a linear regression of the instrument response versus the concentration of the standards, the instrument response is treated as the dependent variable (y) and the concentration

as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where:

y = Instrument response.

a = Slope of the line (also called the coefficient of *x*).

x = Concentration of the calibration standard.

b = The intercept.

The regression calculation will generate a correlation coefficient (R^2) 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^2 must be greater than or equal to 0.995.

The calculated intercept value needs to be evaluated before reporting sample results. A positive value for the intercept indicates that there is some threshold instrument response which is the limiting factor in establishing linearity. A negative intercept value can be transformed into a x-intercept value that represents a threshold concentration, which is the limitation. If the intercept is positive, then, as a general rule, results where the instrument response is less than three times (3x) the intercept value may be unreliable. This will afford some protection against false positive results. If the intercept is negative, results below the concentration of the lowest concentration calibration standard may be unreliable. These adjustments to the quantitation limits will apply to all samples analyzed using the regression line.

As discussed above the evaluation of continuing calibration acceptance is determined by %D which is calculated as follows:

$$\%D = (RF_i - RF_c / RF_i) 100$$

where:

%D = Percent difference

RF_i = average relative response factor from initial calibration

RF_c = Relative response factor from current calibration check standard.

2.6.2.4.2 Result Quantitation

Calculations to produce concentration in water and/or soil using the CF, RF, or RRF are presented in this section.

The concentration of each identified analyte in aqueous samples is quantified from the measured peak response using the CF as follows:

$$\text{Aqueous Concentration} = (A_x)(V_t)(D) / (CF)(V_i)(V_s)$$

where:

- A_x = Response for the analyte in the sample.
- CF = Average CF from initial calibration.
- V_i = Volume of extract injected. For purge and trap analysis $V_i = 1$
- D = Dilution factor, if dilution was made on the sample prior to analysis.
- V_t = Volume of total extract. For purge and trap analysis $V_t = 1$
- V_s = Volume of sample extracted or purged, ml.

The concentration of each identified analyte in soil samples is quantified from the measured peak response using the CF as follows:

$$\text{Concentration} = (A_x)(V_i)(D) / (CF)(V_i)(W)(P)$$

where:

- A_x = Response for the analyte in the sample.
- CF = Average CF from initial calibration.
- V_i = Volume of extract injected. For purge and trap analysis $V_i = 1$.
- D = Dilution factor, if dilution was made on the sample prior to analysis.
- V_t = Volume of total extract. For purge and trap analysis $V_t = 1$.
- W = Weight of sample extracted or purged (g).
- P = Percent dry weight of sample/100, or 1 for a wet-weight basis.

The concentration of each identified analyte in water and soil samples is quantified from the measured peak response using the RF as follows:

$$\text{Concentration } (\mu\text{g/g or mg/L}) = (\text{mean RF})(\text{area of signal})(\text{dilution factor})$$

The concentration of each identified analyte in aqueous samples is quantified from the peak response using the RF as follows:

$$\text{Aqueous Concentration } (\mu\text{g/L}) = (A_x)(C_{is})(D) / (A_{is})(RRF)(V_s)$$

where:

- A_x = Area of characteristic ion for compound being measured.
- C_{is} = Amount of internal standard injected (ng).
- A_{is} = Area of characteristic ion for the internal standard.
- RRF = Average RF from initial calibration.
- V_s = Volume of water purged (ml), taking into consideration any dilutions made.
- D = Dilution Factor, if a dilution was made on the sample prior to analysis.

The concentration of each identified analyte in soil samples is quantified from the peak response using the RF as follows:

$$\text{Soil Concentration } (\mu\text{g/g}) = (A_x)(C_{is})(D) / (A_{is})(RF)(W)(P)$$

where:

- A_x = Area of characteristic ion for compound being measured.
- C_{is} = Amount of internal standard injected (ng).
- A_{is} = Area of characteristic ion for the internal standard.
- RF = Average RF from initial calibration.
- D = Dilution Factor, if a dilution was made on the sample prior to analysis.

W = Weight of sample extracted or purged, g.
 P = Percent dry weight of sample/100, or 1 for a wet-weight basis.

In calculating sample concentrations using regression analysis, the regression equation is rearranged to solve for the concentration (x), as shown below:

$$x = (y - b) / a$$

where:

y = Instrument response.

a = Slope of the line (also called the coefficient of x).

x = Concentration of the calibration standard.

b = y -intercept.

2.7 Data Acquisition Requirements (Non-direct Measurement)

The need to assemble pertinent information previously developed by others will be determined. This is typically considered during the project planning stages. The scope of any resulting survey will be determined by the Technical Manager dependent on the needs of the project. Any limitations or potential reservations for the accuracy or credibility of acquired information that could affect project quality should be clearly identified. Acquired information may include:

- Applicable federal, state, and local regulations and rulings
- Program/site status
 - History/background
 - Future plans
 - Requirements/schedule
- Methodologies available for:
 - Field exploration, monitoring, testing, and sampling
 - Laboratory testing
 - Processing and volume reduction of radioactive/hazardous material
 - Isolation and disposal of radioactive/hazardous material
 - Numerical analysis and design
- Existing data generated for the specific region or site
 - Demographical
 - Geological (surface and subsurface)
 - Hydrological/meteorological (e.g., groundwater distribution and usage)
 - Geochemical
 - Geotechnical
 - Facility development and practices (past, present, and future)
 - Type, volume, and extent of contamination
 - Physical layout of man-made facilities

- Data generated on specific wastes, materials, or chemical compounds of interest
 - Processing
 - Physical
 - Chemical
 - Geochemical
 - Radiological
 - Mechanical
 - Thermomechanical
 - Toxicity/hazards and protection
 - Treatability

Previous or concurrent surveys, studies, analyses, and designs of a similar or parallel nature.

Sources for the above information may include:

- Government and private regulations, standards, guidelines, journals, periodicals, and data compilations
- Textbooks and maps
- Reports and manuals previously issued by USACE, DOE, EPA, or other organizations
- Results of currently ongoing investigations by government and private agencies, corporations, and research facilities
- Personal communications
- Aerial photographs and satellite imagery
- Procurement documents issued by the client.

Information collected will be documented to indicate its source. Documentation will, as appropriate, include author or individual contacted; source title; identification of periodical or journal; standard, guideline, or report number; identification of publisher or originating organization; page location; and date. Documentation must be sufficient to allow other individuals to easily obtain or verify the information.

Whenever possible, complete copies of articles, data compilations, maps, reports, and photographs will be included in the project files. If this is not feasible, copies of title pages and pertinent sections should be included with complete source documentation.

Personal communications such as interviews, correspondence, or telephone conversations will be completely documented in the form of trip reports, meeting notes, memoranda, and telephone records and the resulting documentation included in the project files. Documentation will provide, as appropriate, the date and the name, organization, address, telephone number, and

credentials of individuals contacted. A request should be made for formal written confirmation of critical data obtained verbally to serve as final documentation.

As necessary, an estimation of the quality/credibility of the information will be made. The collection of information must be consistent with the quality objectives of the project. Formal data quality objectives will be established for a project. Particular attention should be given to information that is collected that is not published from a peer reviewed source, or collected under the controls of a documented quality assurance program. This may include, but is not limited to personal interviews, internal reports and memoranda, or newspaper articles.

3.0 ASSESSMENT / OVERSIGHT

3.1 Quality Control

The purpose of this section is to describe the Quality Control (QC) Program to be implemented. The primary purpose of this QC Program is to provide a self-inspection system which allows the USACE a method of ensuring that all activities are performed in accordance with project requirements and conformance to the approved, Field Sampling Plan (FSP), Sampling and Analysis Plans (SAPs), Quality Assurance Project Plan (QAPP), and Project Health and Safety Plan (HASP).

The QC program consists of a three phase control program. The control program is implemented prior to initiating each definable feature of work and will remain in effect throughout its duration. The three phase control program includes:

- a preparatory phase;
- an initial phase; and
- a follow-up phase.

The QC program will also include inspections to be performed at the completion of a task. The Program Manager is responsible for implementing all phases of the quality control program. Health and safety audits will also be conducted to ensure that all work is being performed in compliance with the HASP. The health and safety audits will be performed by the Health and Safety Manager (HSO).

3.1.1 Definable Features of Work

A definable feature of work is a task which is separate and distinct from other tasks and has separate control requirements. The following definable features are identified but not limited to the following:

- field sampling;
- on-site analyses by the field laboratory;
- off-site analyses by the fixed-base laboratory;
- data management (including data reduction, validation, and reporting); and
- risk assessment.

The three phase control system will be implemented prior to the initiation of each feature of work.

3.1.2 Preparatory Phase

The preparatory phase of the three phase control program will occur prior to beginning work on a task. A preparatory inspection of a task may be necessary when the task is first performed at each of the sites. The preparatory inspection includes providing the contracting officer (CO) with a preparatory inspection outline and performing a preparatory phase inspection prior to beginning work on the task. The inspection will include the following items:

- A review of the SAP, FSP and CDQMP to ensure that the task has been approved by the Technical Manager.
- A check to ensure that all required permits and clearances for the task have been obtained.
- A check to ensure that all required training for the task has been obtained by all personnel performing task.
- A check to ensure that the required health and safety training and medical monitoring has been completed and that the task will be performed in strict compliance with the HASP.
- A check to ensure that all personnel performing the task have reviewed the SAP, FSP, CDQMP, and HASP.
- A discussion of the procedures which will be implemented for completing the task.
- A check to ensure that all the equipment and instruments required to perform the task are present.
- A check to ensure that all the required equipment and instruments for health and safety monitoring are present.
- A check to ensure that all the instruments are being calibrated to the manufacture and/or project specifications.
- An examination of the work area to ensure that all preliminary work has been performed and that conforms to the FSP, SAP and CDQMP.
- A check to ensure that provisions are in place to allow for the required QC and safety inspections and audits during the task.

3.1.3 Initial Phase

The initial inspections are performed when a representative portion of a task has been completed. The purpose of the initial phase is to ensure that tasks conform to the approved Work Plan, FSP, and QAPP. This phase includes a review of the procedures employed to complete the task and a check to ensure that the task is being performed according to the HASP.

3.1.4 Follow-Up Phase

Follow-up inspections will be conducted at regular intervals to ensure that the task is being performed in strict compliance to the project requirements. Follow-up inspections will be conducted at a minimum frequency of one (1) inspection per event for each task. If follow-up inspections identify items in the task which do not conform to the project requirements, additional preparatory or initial inspections may be required. A follow-up inspection may be

required at each work site for a specific task.

3.1.5 Completion Inspection

A completion inspection will be performed when all work on a task at a specific site is complete. A list of items which do not conform to the project requirements for the task will be developed. The Program Environmental Engineer will conduct a follow-up inspection to verify that the task was completed according to the project requirements and that corrective actions have been successfully implemented to address all deficient items.

3.2 Assessments and Response Actions

Both internal and external assessments are conducted to provide assurance that samples are collected and analyzed according to acceptable procedures. The assessments that are conducted include readiness reviews, system audits, surveillances, and the establishment of a NCR/CAR System.

3.2.1 Readiness Reviews

The goal of the readiness review is to ensure that the field team is prepared for all aspects of conducting field investigations. Items that are addressed include the review of supply procurement plans, contingency plans, securing of site clearances, and training of project personnel. Readiness reviews will be performed by the Project Leader prior to mobilization for field activities or at the direction of the Project Manager. Documentation will be in the form of a checklist that is specific to the type of field activities to be performed. Deficiencies discovered during readiness reviews will be communicated to the Project Manager prior to mobilization.

3.2.2 System Audits

System audits are formal evaluations of all aspects necessary to produce a desired result. This type of audit is limited to the pre-evaluation of subcontract laboratories. The purpose of the audit is to ensure that all procedures including supply procurement, sample receipt and tracking, analysis, data review and reporting, QA/QC, and nonconformance/corrective action are established prior to the first sample reaching the laboratory. The goal of the audit is to establish that the systems that are in place are sufficient to provide the quality of data necessary for the project activity.

System Audits will be conducted by the Project Chemist to verify the laboratories ability to adhere to QA/QC requirements during the analysis of environmental samples. Documentation will include an audit notification letter, an audit report, and an audit close-out letter that will be provided to the Project Manager. Audit reports will be provided to the Technical Manager within 10 days of the completion of the formal audit.

The audit notification letter will define the schedule of the audit, the activities to be reviewed, and the laboratory personnel that will be required. The audit report will include documentation of the opening meeting, results of review, documentation of the audit closing meeting, any areas found to be deficient, and schedule for completing corrective actions. The audit close-out letter will document the successful completion of corrective actions.

3.2.3 Surveillances

Surveillances or process audits are smaller and less extensive than system audits. The purpose of the surveillance audit is to review specific activities to ensure that established procedures are followed to achieve the desired result.

Surveillances will be conducted to verify field and laboratory adherence to requirements during the collection and analysis of environmental samples. Documentation will include descriptions of activities reviewed, discussions with project personnel, nonconformance/corrective actions, and recommendations for rectifying any quality deficiencies. Surveillance reports will be provided to the Technical Manager within 10 days of the completion of the surveillance.

3.2.4 Performance Evaluation Samples /Data Tracking Audits

Laboratory performance audits will consist primarily of blind performance evaluation samples submitted to the laboratory and/or data tracking audits completed on a real time basis while samples are being analyzed. PE Samples will consist of Standard Reference Materials (SRM) supplied by an approved vendor such as ERM will be submitted to the laboratories periodically throughout the course of the contract. Data from the blind PE samples and results of data tracking audits will be reviewed and provided to the Technical Manager within 10 days of the completion of the review.

3.3 Reports to Management

Each defined work element is responsible for producing a report to project management listing the activities conducted during a specific period of sampling and/or analysis. The reports generally will include the number of units collected or produced, NCR/CAR reports, audit/surveillance summaries, and QC summaries. All reports will be provided to the Program Environmental Engineer within the time frames discussed in the following subsections.

3.3.1 Field Activities

The Sampling Team Leader will provide a summary of field activities on a every other day basis to the Project Environmental Engineer, Project Geologist or Project Chemist as appropriate. The location of field activities (including field laboratory analysis), date and hours of operation, weather conditions, work performed, and any difficulties encountered during the period will be

summarized, Figure 3.6 is an example daily project report.

3.3.2 Drilling Subcontractors

The drilling subcontractor will provide a summary of drilling activities on a weekly basis. The location of drilling activities, date and hours of operation, work performed, and any difficulties encountered during the period will be summarized.

3.3.3 Subcontract Laboratory

Laboratories providing services to the Department of Defense, including USACE, must be accredited for the DOD Environmental Laboratory Accreditation Program (DOD ELAP) by a DOD approved Accrediting Body (AB) which is ISO/IEC 17011:2004 compliant. As an element of the DOD ELAP, all laboratories must demonstrate the ability to generate acceptable results from the analysis of proficiency-testing (PT) sample(s), subject to availability, using each applicable method in the specified matrix. DOD ELAP accreditation establishes that laboratories have an established and documented laboratory quality system that conforms to ISO/IEC 17025 as implemented by the *DoD Quality Systems Manual for Environmental Laboratories*, version 4.1, April 2009.

In addition to DOD ELAP accreditation, the laboratory shall hold current accreditation for all appropriate fields-of-testing under the State of Utah Bureau of Lab Improvement accrediting authority under the National Environmental Laboratory Accreditation Program (NELAP). Proof of current accreditation / certification for the applicable fields of testing is required prior to the laboratory acceptance of any samples for TEAD projects.

The subcontract laboratory will provide a summary of sample receipt, analysis, and reporting on a weekly basis. The report will include the number of samples received, analyzed, and reported by analysis method, discrepancies noted in sample receipt, and laboratory NCR/CAR reports. This report does not replace the requirement of the case narrative for each lot, but serves to alert project management of potential problems.

4.0 DATA VALIDATION AND USABILITY

4.1 Data Review, Validation and Verification Requirements

This section describes the approach to be used to reduce, verify, report, and manage collected data. Accurate data reduction, validation, and reporting protocols are necessary to interpret data and arrive at decisions. The quality of the data collection process will be assessed through reviews of all measurements performed. The purpose of this section is to discuss the evaluation and assessment of QC requirements necessary to document the quality of the collected data. The frequency of data review validation and verification is discussed below according to the category of data collected.

4.1.1 Field Sampling/Non-analytical Data

Field sampling data, including field logbooks and field activity forms, will be reviewed daily by the Project Leader. Boring logs will be reviewed by the project geologist.

4.1.2 Screening/Non-definitive Data

Screening data will be reviewed and verified by the analyst and the Program Chemist. The review of the data will ensure QA procedures were followed and QC requirements have been met. Screening analysis data will be reviewed against the acceptance criteria defined in the SAP. The review consists of evaluating the QA/QC data including instrument blanks, system blanks, and calibration data to make sure QA/QC requirements have been met and appropriate corrective actions taken. Screening results will be evaluated by comparing the screening data with the definitive data. A review of the QA/QC data will be summarized and presented as part of the QCSR.

4.1.3 Definitive/Confirmatory Data

Definitive data will be reviewed by the laboratory, the USACE and an independent third party contractor. Data verification will be performed on 90% of the results generated. Data validation will be performed by an independent third party on a minimum of 10% of the data generated. Additional data validation may be performed at the discretion of the Project Chemist and Technical Manager.

4.2 Validation and Verification Methods

The validation and verification of data takes place at varied levels within the full range of environmental services encompassing the scope of work associated with the contract. Program procedures, plans, and project-specific documents provide specific details of the individual

positions responsible for verification and validation activities involved with data management. In general, quality affecting records are reviewed at a level commensurate with the information being checked.

4.2.1 Data Verification

The following verifications will be performed on 100 percent of the analytical data.

- The organic data will be reviewed for holding times, blank analysis results, LCS, MD/MSD and surrogate recovery.
- The inorganic data will be reviewed for holding times, blank analysis results, pre-digestion matrix spikes, sample duplicate and LCS recoveries.
- Analytical results will be qualified as a result of the data validation process in accordance with the flagging convention tables included in Appendix C of this document.

The data verification of the project analytical data will be an ongoing process that will be performed by both the analytical laboratory generating the data and the Program Chemist. The initial step of the data verification process will be performed by the analytical laboratory. During this review, the calculations, QC sample data, spike recovery, instrument performance indicators, and project specification will be thoroughly inspected through peer level review prior to its release to the laboratory Project Manager. Any problems or Nonconformance issues encountered during the analysis will be noted in the project case narrative that precedes each data package. Where unexplainable variations appear, calculations will again be checked for errors and the sample collection and analytical procedures reviewed to identify any causes for the inconsistencies. All calculation errors will be corrected and anomalies in the sampling or analytical procedures documented and reported in the project analytical data package. The raw data are then QC reviewed for technical correctness by the laboratory Project Manager before final printing. After the data package has been completed, the transcription of 100% of the data is verified by the laboratory QA/QC Manager. The laboratory QA/QC Manager will also review the data for conformance to the project data quality objectives. The Project Chemist will be notified of any existing problems and will be updated as conditions dictate.

The laboratory system for ensuring valid data includes several levels of review. Each level commands specific action to prevent the unqualified release of erroneous data and to correct any problems discovered during the review process. All analytical data generated at the Laboratory are extensively checked for accuracy and completeness. The data review process consists of data generation, reduction, and three levels of review, as described below.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample preparation and analysis information is correct and complete
- The appropriate SOPs have been followed
- Analytical results are correct and complete
- QC samples are within established control limits; blanks are acceptable
- Special sample preparation and analytical requirements have been met
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, out of control forms, if required, are complete, holding times are documented, etc.).

This initial review step, performed by the analyst is designated Level 1 review. The analyst then passes the data package to an independent reviewer who performs a Level 2 review.

Level 2 review is performed by a group leader or data review specialist whose function is to provide an independent review of the data package. This review is structured to assure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented
- QC samples are within established guidelines
- Qualitative identification of sample components is correct
- Quantitative results are correct
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented, out-of-control forms, if required, are complete, holding times are documented, etc.)
- The data are ready for incorporation into the final report
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed, and all of the analytical results from 10 percent of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is considered complete. If any problems are found with the data package, an additional 10 percent of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety. Level 2 data review is documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

All data collected during the project will be reviewed and flagged with the appropriate data qualifiers before reported. Detection limits will vary with sample type and the level of interferences associated with the sample matrix. If anomalous results are obtained, every effort will be made to identify any problems in the sample collection, sample preparation, and/or analysis that could have contributed to the anomaly. If any problems have occurred, they will be reported and will include the results, and the appropriate qualifier, with an estimate of the impact the problem may have had on the data. If the sample results do not conform with the data quality

objectives, the data will be thoroughly reviewed in order to identify any existing problems and the sample analysis will be repeated if deemed necessary.

Following the analytical laboratory data review, the sample data will be submitted to the Program Chemist who will be responsible for the review and to compare all data with the project data requirements.

4.2.2 Data Validation

Independent of the laboratory review, data validation will be performed on 100 percent of definitive analysis performed for each method of analysis using the Flagging Conventions presented in Appendix C of this document and/or current guidance as provided in the DOD QSM and USACE Guidance (see references). Analytical results will be qualified as a result of the data validation process in accordance with the CDQMP flagging conventions. An additional 10% will be reviewed back to raw data including a review of COCs, holding times, chromatograms, spectra, instrument printouts, sample calculations, calibrations, instrument run logs, preparation logs, method and field blanks, field duplicates, matrix spike/matrix spike duplicates (MS/MSD), LCS, and case narratives.

Hierarchy of Applicable Guidance Documents for Data Validation / Review

The following hierarchy will be used in applying guidance / requirements documents to the review of project specific analytical data. If a site specific QAPP is not available the default requirements including data quality indicators (DQIs) are per the most current version of the DOD QSM.

1. Site-specific SAP / UFP-QAPP
2. DOD QSM, ver 4.1 (Apr 2009) to supplement any gaps in the WP/UFP-QAPP requirements.
3. EM 200-1-10, June 2005 Qualifiers

Data flags are assigned to analytical results for both organic and inorganic data based on the project data quality control requirements. Data flags are defined below:

- U The analyte was analyzed for, but was not detected above the level of the associated value. The associated numerical value (e.g., the MDL) indicates the approximate concentration.
- J The analyte was analyzed for and was positively identified, but the associated numerical value is estimated and may not represent the actual amount present in the environmental sample. The data should be considered approximate but usable for decision-making purposes.

- UJ The analyte was analyzed for, but was not detected above the level of the associated numerical value; however, the associated numerical value is approximate and may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R The data are unusable for all purposes. The analyte was analyzed for, but the presence or absence of the analyte has not been verified.

The data validation report consists of three sections. The following describe each section.

4.2.2.1 Data Validation Summary Report

The summary report is designed for each data package received from the laboratory. The report includes the analytical criteria that are reviewed for each analytical test method.

The organic data are reviewed for holding times, calibrations, blanks (i.e., laboratory blanks and field blanks), surrogates, matrix spike, matrix spike duplicates, internal standards, and laboratory control samples. The inorganic data are reviewed for holding times, blanks, calibrations, matrix spike, matrix spike duplicate or laboratory duplicate, ICP interference check, ICP serial dilution, furnace post digestion, and laboratory control samples. Field duplicate samples are reviewed if the field duplicate samples are identified for the project samples. Any major or minor deficiencies noted during the data validation process is noted in each category. If the data are required to be qualified due to any outlier in QC criteria, an explanation on how data are qualified is given in each category. The last part of the summary report includes the definitions of the data validation qualifiers that are assigned to the analytical data.

4.2.3 Data Usability

Analytical results will be qualified as a result of the data validation process in accordance with the flagging convention tables included in Appendix C of this document. Results will be compared to action levels and ARAR's to determine usability when QC criteria are not met.

4.3 Reconciliation with Data Quality Objectives

A Data Quality Assessment (DQA) will be performed and a report prepared following completion of data acquisition. The purpose of the DQA is to present an evaluation of the entire data collection program and document the successful completion of the DQOs. The DQA will provide documentation of the internal and external reviews of project operations during acquisition, validity of the collected data, and recommendations for data use. The DQA report will include:

- a) Summary of project DQOs;
- b) Summary of field QC operations;

- c) Summary of laboratory QC operations;
- d) Statistical summaries of the precision, accuracy, representativeness, and completeness of off-site laboratory data;
- e) Summaries of outlying observations and impact on DQOs; and
- f) Recommendations for data use.

The goal of the DQA report is to provide documentation that the data collection program has, by design, collected a sufficient quantity and quality of data to meet the needs of the project.

4.3.1 Analytical/Statistical Control Parameters

The purpose of this document is to facilitate implementation of the requirements of the DQOs for specific projects supporting the program and applicable regulatory requirements. To assure that data obtained is sufficiently accurate and consistent with the DQOs, the following procedures will be used for assessing the quality of the measurement data:

- Accuracy and Precision is the agreement between a measurement and the true value, and the degree of variability in the agreement, respectively. To determine the precision of the method and/or laboratory analyst, a routine program of replicate analyses is performed. The results of the replicate analyses are used to calculate the relative percent difference (RPD), which is the governing quality control parameter for precision. For replicate results relative percent difference is calculated:

$$\%RPD = [X_1 - X_2 / (X_1 + X_2)/2] * 100$$

where:

RPD = relative percent different

X₁, X₂ = value of sample 1 and sample 2

- To determine the accuracy of an analytical method and/or the laboratory analyst, a periodic program of sample spiking is conducted (minimum one spike and one spike duplicate per batch or one spike and one duplicate per batch). The results of sample spiking are used to calculate the quality control parameter for accuracy evaluation, the percent recovery (%R). Percent recovery is calculated:

$$\%R = (C_1 - C_2) * 100 / C_3$$

where:

R% = Spike amount recovered

C₁ = Concentration of analyte in spiked sample

C₂ = Concentration of analyte in unspiked sample

C₃ = Concentration of spike added

- Completeness is the adequacy in quantity of valid measurements to prevent misinterpretation and to answer important questions. For this project, the data completeness objective is 90 percent. The completeness requirements for holding times

will be 100 percent. If any sample exceeds the holding time specified by EPA SW-846 (or other guidance documents for other analyses) that sample may be required to be resampled and reanalyzed.

- Representativeness is a qualitative parameter that reflects the extent to which a given sample is characteristic of a given population at a specific location or under a given environmental condition. Representativeness is best satisfied by making certain that sampling locations are selected properly, a sufficient number of samples is collected, and an appropriate sampling technique is employed. Variations at a sampling point will be evaluated based on the results of field duplicates. For TEAD projects, good representativeness will be achieved through careful, informed selection of sampling sites, drilling sites, drilling depths, and analytical parameters; and through the proper collection and handling of samples to avoid interferences and to minimize contamination and loss.
- Comparability is the extent to which comparisons among different measurements of the same quantity or quality will yield valid conclusions. For TEAD projects, comparability among field measurements will be achieved through the use of standard procedures, standard field data sheets, and uniform concentration units. To ensure comparability, field procedures will be standardized and field operations will adhere to standard operating procedures. Laboratory data comparability will be assured by use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), and consistency in reporting units (ppm, ppb, etc.). Analysis of standard reference materials will follow USEPA or other standard analytical methods, which utilize standard units of measurement, methods of analysis, and reporting format.
- Sensitivity (Reporting Limits) Assuring the validity of quantitative measurements at low concentrations is an extremely difficult technical problem. With regulatory action levels being pushed lower and lower, the validity of any given measurement becomes even more important. The consequences of false positive or false negative data can be significant. The laboratory will report results below the reporting limit as “Not Detected” because, by definition, the reliability of the data at that level is questionable. Organic data that needs to be reported below the quantitation limit will have the data flagged accordingly.

Quantitation Limits are the extent to which the equipment, laboratory or field, or analytical process can provide accurate, minimum data measurements of a reliable quality for specific constituents in replicate field samples. It 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. The actual quantitation limit for a given analysis will vary depending on instrument sensitivity and matrix effects.

If dilution to bring the reported concentration of a single compound of interest within the linear range of the calibration, results in non-detect values for all other analytes with detected concentrations in the initial sample analysis, the results of the original run and

the dilution will be reported with appropriate notations in the narrative of the report. Matrix effects (i.e., highly contaminated samples requiring dilution for analysis, dilution to bring detected levels within the range of calibration, and matrix interference requiring elevation of detection limits) will be considered in assessing compliance with the requirements for sensitivity.

The quality assurance objectives for laboratory quality control data are designed to screen out data of unacceptable precision or accuracy and to provide data that will meet the data quality goals for the project.

Traceability is the extent to which data can be substantiated by hard-copy documentation. Traceability documentation exists in two essential forms: one that links quantitation to authoritative standards and a second that explicitly describes the history of each sample from collection to analysis.

The fundamental mechanisms that will be employed to achieve these quality goals can be categorized as prevention, assessment, and correction. These include:

- Prevention of defects in the quality through planning and design, documented instructions and procedures, and careful selection of skilled, qualified personnel
- Quality assessment through a program of regular audits and inspections to supplement continual informal review
- Permanent correction of conditions adverse to quality through a closed-loop corrective action system.

This document has been prepared in direct response to these goals. This plan describes the program and the procedures to be implemented for projects to be performed for TEAD. The objectives for precision and accuracy for each chemical are based mainly on the capabilities of the approved EPA analytical method with respect to laboratory quality control.