ENVIRONMENTAL DATA QUALITY MANAGEMENT PROGRAM SPECIFICATIONS
UNITED STATES ARMY CORPS OF ENGINEERS (USACE) – SACRAMENTO DISTRICT

VERSION 1.08

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LIST OF ACRONYMS

AA	**Atomic Absorption**
ARARs	**Applicable or Relevant and Appropriate Requirements**
ASTM	**American Society for Testing and Materials**
BFB	**1,4-Bromofluorobenzene**
BGS	**Below Ground Surface**
BNA	**Base Neutral Acid**
CAR	**Corrective Action Report**
CCC	**Calibration Check Compound**
CCV	**Continuing Calibration Verification**
CDQMP	**Chemical Data Quality Management Plan**
CL	**Control Limit**
CLP	**Contract Laboratory Program**
CMECC	**California Military Environmental Coordination Committee**
CO	**Contracting Officer**
COC	**Chain of Custody**
COPC	**Chemical of Potential Concern**
CQAR	**Chemical Quality Assurance Report**
CRQL	**Contract-Required Quantitation Limit**
CVAA	**Cold Vapor Atomic Absorption**
DCB	**Decachlorobiphenyl**
DFTPP	**Decafluorotriphenylphosphine**
DI	**De-Ionized**
DO	**Delivery Order**
DOD	**Department of Defense**
DQCR	**Daily Quality Control Report**
DQO	**Data Quality Objective**
DRO	**Diesel Range Organic**
ECD	**Electron Capture Detector**
EPA	**Environmental Protection Agency**
FID	**Flame Ionization Detector**
FLAA	**Flame Atomic Absorption**
FPD	**Flame Photometric Detector**
FSP	**Field Sampling Plan**
GC	**Gas Chromatography**
GC/MS	**Gas Chromatography/Mass Spectrometer**
GFAA	**Graphite Furnace Atomic Absorption**
GPC	**Gel Permeation Chromatography**
GRO	**Gasoline Range Organic**
HECD	**Hall Electrolytic Conductivity Detector**
HNO3	**Nitric Acid**
HPLC	**High Performance Liquid Chromatography**
HTRW	**Hazardous, Toxic, and Radioactive Waste**
IC	**Ion Chromatography**
ICP	**Inductively Coupled Plasma**
ICS	**Interference Check Standard**
ICV	**Initial Calibration Verification**
ID	**Identification**

IDL	**Instrument Detection Limit**
IR	**Infrared**
LCS	**Laboratory Control Sample**
LCSD	**Laboratory Control Sample Duplicate**
LIMS	**Laboratory Information Management System**
LQMP	**Laboratory Quality Management Plan**
LUFT	**Leaking Underground Fuel Tank**
MB	**Method Blank**
MD	**Matrix Duplicate**
MDL	**Method Detection Limit**
MEK	**Methyl Ethyl Ketone**
MIBK	**Methylisobutyl Ketone**
MQL	**Method Quantitation Limit**
MRD	**Missouri River Division**
MRL	**Method Reporting Limit**
MS	**Matrix Spike**
MSD	**Matrix Spike Duplicate**
MSA	**Method of Standard Additions**
NIST	**National Institute of Standards and Technology**
NPD	**Nitrogen Phosphorus Detector**
OIG	**Office of Inspector General**
OSHA	**Occupational Safety and Health Administration**
PAH	**Polynuclear Aromatic Hydrocarbons**
PARCC	**Precision, Accuracy, Representativeness, Completeness, and Comparability**
PBMS	**Performance Based Measurement System**
PCB	**Polychlorinated Biphenyl**
PDS	**Post Digestion Spike**
PES	**Performance Evaluation Sample**
PID	**Photoionization Detection**
PPB	**Parts Per Billion**
PPM	**Parts Per Million**
PQL	**Practical Quantitation Limit**
QA	**Quality Assurance**
QAO	**Quality Assurance Officer**
QAPP	**Quality Assurance Project Plan**
QC	**Quality Control**
QCSR	**Quality Control Summary Report**
RCRA	**Resource Conservation and Recovery Act**
RF	**Response Factor**
RFP	**Request For Proposal**
ROD	**Record of Decision**
RPD	**Relative Percent Difference**
RSD	**Relative Standard Difference**
RSE	**Relative Standard Error**
SAP	**Sampling and Analysis Plan**
SD	**Serial Dilution**
SOP	**Standard Operating Procedure**
SOW	**Scope of Work**
SPCC	**System Performance Calibration Check**

SPE	**Solid Phase Extraction**
SVOC	**Semivolatile Organic Compound**
TAL	**Target Analyte List**
TAT	**Turnaround Time**
TCL	**Target Compound List**
TEPH	**Total Extractable Petroleum Hydrocarbon**
TIC	**Tentatively Identified Compound**
TPH	**Total Petroleum Hydrocarbon**
TPPH	**Total Purgeable Petroleum Hydrocarbon**
TRPH	**Total Recoverable Petroleum Hydrocarbon**
USACE	**United States Army Corps of Engineers**
USCS	**Unified Soil Classification System**
USDOT	**United States Department of Transportation**
USEPA	**United States Environmental Protection Agency**
USGS	**United States Geological Survey**
UST	**Underground Storage Tank**
VOA	**Volatile Organic Analysis**
VOC	**Volatile Organic Compound**
XRF	**X-Ray Fluorescence**
ug/L	**Micrograms per liter**
ug/kg	**Micrograms per kilogram**
mg/L	**Milligrams per liter**
mg/kg	**Milligrams per kilogram**
^{0}C	**Degrees Celsius**
ul	**Microliter**
ml	**Milliliter**
r	**Linear Regression or Correlation Coefficient**
r^2	**Coefficient of Determindation**
%	**Percent**

1.0 SUMMARY

This section provides specifications for Environmental Data Quality Management for sampling and analysis associated with characterization of soils, ground water, and other media for this contract. This section delineates the responsibilities and procedures for all sampling and analytical activities to assure that the data obtained is of sufficient quality to meet intended uses and Applicable or Relevant and Appropriate Requirements (ARAR's) within the project. This section also provides guidance in the preparation of the Contractors Chemical Data Quality Management Plan (CDQMP) and delivery order specific Sampling and Analysis Plans. The CDQMP shall be composed of a Field Sampling Plan (FSP) and a Quality Assurance Project Plan (QAPP). The CDQMP shall include detailed plans for sampling, analysis, and chemical quality control (QC) activities. Unless otherwise specified in a delivery order, normal turn-around time (TAT) shall be defined as 21 days and shall be applicable for analyses for this project. The CDQMP is intended to be an installation-wide document covering the overall requirements for the field and analytical programs. The QAPP portion should be revised annually for long term (>2 years) projects. For those project that do not require a CDQMP, the requirements for work plans must be approved by the District Chemist prior to initiation of work.

Note: The successful offeror will be notified of any deficiencies in the CDQMP included in the RFP submittal. All deficiencies identified in comments provided to the Contractor by the Contracting Officer (CO) must be resolved to the satisfaction of the Government within 30 calendar days of receipt of comments and prior to the start of field work for any delivery orders involving sampling and analysis. Multiple cycles of review and comment may be required as necessary to complete revisions to the CDQMP to meet the requirements of this RFP. This work will be performed at no additional expense to the Government.

This document contains minimum standards. Each project must prepare a project-specific sampling and analysis plan (SAP) which stipulates the data quality objectives and chemical data quality requirements. The project-specific SAP must be provided to the laboratory prior to initiation of work.

2.0 REFERENCE PUBLICATIONS

The following documents were used to develop these specifications.

2.1 Environmental Protection Agency (EPA)

EPA SW-846	Test Methods for Evaluating Solid Waste, Third Edition (Update III), December 1996.
EPA QA/R-5	EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, Interm Final, November 1999.
EPA QA/G-4	EPA Guidance for the Data Quality Objectives Process, EPA/600/R-96/055, Final, September 1994.
EPA QA/G-5	EPA Guidance for Quality Assurance Project Plans, EPA/600/R-98/018, Final, February 1998.
EPA QA/G-9	EPA Guidance for Data Quality Assessment – Practical Methods for Data Analysis, EPA/600-R-96-/084, July 1996.
EPA	EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review. EPA540/R-94/012, February 1994
EPA	EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. EPA540/R-94/012, February 1994

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

EM 200-1-1	Validation of Analytical Chemistry Laboratories, July, 1994.
EM 200-1-2	Technical Project Planning Guidance for HTRW Data Quality Design
EM 200-1-3	Requirements for the Preparation of Sampling and Analysis Plans, September 1994.
EM 200-1-6	Chemical Quality Assurance for HTRW Projects, October 1997.
ER-110-1-263	Engineering and Design Chemical Data Quality Management for Hazardous Waste Remedial Activities, December 1997
Draft	Shell for Analytical Chemistry Requirements, Version 1.0, November 1998.
USACE	CRREL Special Report No. 96, Comparison Criteria for Environmental Chemical Analyses of Split Samples Sent to Different Laboratories – Corps of Engineers Archived Data, Grant, C.G., Jenkins, T.F., and Mudambi, A.R., USACE Cold Regions & Environmental Research Laboratory, Hanover NH, May, 1996

Deviations from EM 200-1-3 minimum requirements must go through the approval process described in EM 200-1-3. As documents listed above are revised, the contractors performing work on long-term projects (greater than 2 years duration) are responsible for updating the CDQMP to be compliant with most recent guidance based on approval of District Chemist.

2.3 OTHER

CMECC	Best Practices for the Detection and Deterrence of Laboratory Fraud, California Military Environmental Coordination Committee, Chemical Data Quality/Cost Reduction Process Action Team, Version 1.0, March 1997

3.0 SUBMITTAL REQUIREMENTS

The Contractor shall submit the following items required by this section:

Chemical Data Quality Management Plan (Section 5.1-5.4)
Analytical Data Package Reports (Section 5.5)
Data Validation Reports (Section 5.6)
Daily Quality Control Reports (Section 5.7)
Quality Control Summary Report (Section 5.8)
Non-Routine Occurrences Report (Section 5.9)
Data Report for the Chemical Quality Assurance Report (CQAR) (Section 5.10)

4.0 CHEMICAL ANALYSIS

The Contractor shall execute chemical analyses as described in delivery orders for this contract. The CDQMP shall contain all details described in Section 5.0 (including subsections) of these specifications for the following analyses that will be relevant for work on this contract: EPA Method 8000B; 8021A; 8041; 8081A; 8082; 8141A; 8151A; 8260B; 8270C; 8280A; 8290; 8310; 8321A; 8330; 8015B (purgeable); 8015B (extractable); 418.1; 413.2; metals by 7000A series; metals by 6010B; metals by 6020, mercury by 7470A/7471A, and hexavalent chromium by Method 7196A. Analytical procedures shall conform to the most recently promulgated version of SW-846 (currently Update III, December 1996) and the State requirements for the specific project. Total petroleum hydrocarbons (TPH) as gasoline range organics, diesel range organics, and motor oil organics reporting and quantitation shall conform to the requirements outlined in Method 8015B, unless State requirements supersede this method.

This specification establishes a basic approach for application of analytical chemistry methods (e.g., SW-846, performance-based methods) by the USACE. The concepts included here specify a baseline implementation of several analytical chemistry methods. However, when a performance-based analytical approach is employed, additional regulatory approval may be necessary to ensure acceptance of data generated.

In order to promote flexibility as well as some degree of consistency in the data generated to support USACE HTRW projects, when inconsistent or mutually exclusive method requirements are encountered, the following hierarchy applies: (1) Project-specific documents (e.g., SAP), (2) USACE Engineer Manuals or other policy guidance, and (3) the SW-846 methods. Hence, the laboratory should be aware of and review these sources to determine project-specific data quality objectives (DQOs) and applicable project requirements.

4.1 PERFORMANCE BASED METHODS IMPLEMENTATION

As the various Federal, State, and Local regulatory agencies acknowledge the adoption of Performance Based Measurement Systems (PBMS) as a means to achieve required environmental monitoring, the applicability of performance based methods to individual projects will increase. PBMS are defined by USEPA as a set of processes wherein a monitoring program's DQOs are designated, rather than specifying the approved standard analytical method necessary. To date however, the details for establishing data quality and performance requirements for required monitoring to support the assessment and selection of performance based methods have not been fully defined within the various USEPA and state environmental offices. In addition, progress in updating federal, state and local regulations to incorporate the PBMS philosophy, and remove the requirements for specified standard reference methods for the use of new and innovative technologies are necessary to help assure successful PBMS implementation. Currently, PBMS has encouraged the application of field analytical technologies to environmental restoration projects.

This performance based method approach empowers the analytical service (data) provider with the flexibility to vary aspects of an analytical system and protocols as long as the demonstrated method performance meets the requirements established by the data user(s). A PBMS may employ completely different chemistries or detection systems from those identified in current standard reference methods; may alter a sample preparatory or determinative procedures that enhance or inhibit extraction/digestion or signal efficiency; or may encompass only minor modifications to a standard method's instrument configuration. Due to this inherent flexibility, additional effort is necessary in the planning and executing phases to ensure successful implementation of performance based methods. ***This may include any or all of the following: (1) establishing and maintaining proper PBMS documentation (i.e., method SOPs, records of data analyses/results), (2) USACE and regulatory agency review/approval, (3) evaluation of method performance via data quality indicators, and (4) comparison of PBMS data to data generated from a standard reference method.*** Before implementation of performance based methods, the analytical service provider must establish the capabilities of the method/technique, to include selectivity, sensitivity, and range of detection, precision and bias. These are evaluated against performance criteria established by USEPA, state

regulatory agencies, or the technical project planning team to assess the usability of the PBMS or PB method. ***The accuracy of the developers / manufacturers' claims and technical data, and the comparability amongst various techniques should be scrutinized for it is an area which requires standardization.*** In the event that the method capabilities do not meet project requirements, differences shall be reconciled prior to project execution. Reconciliation may require modifying the selected method, choosing an alternative method or techniques, or modifying the project DQOs. ***Project application of performance based methods requires that performance be demonstrated for the analytes of concern, at the levels of concern in the matrix of concern within a specified acceptable error tolerance.*** Data generated from performance based methods are evaluated using the same procedures as standard reference methods, as presented in Section 4.3 through 4.9. In addition, if the PBMS (1) is considered an emerging technology, (2) lacks established records of use, or application to environmental matrices, or (3) varies significantly from the standard reference method, suggest acquiring a percentage of split samples for redundant analysis by the standard reference method. This will allow a comparison or calculation of a correlation factor between the data sets to evaluate the usability of the performance-based method in that project matrix.

4.2 SW-846 METHODS ORGANIZATION

The following sections summarize the guidance requirements for typical HTRW projects. Program and project-specific requirements should be presented in the CDQMP.

4.2.1 SW-846 Methods Implementation

EPA Publication SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," contains the analytical testing methods that the EPA has evaluated and found to be acceptable for analysis of samples under Subtitle C of the Resource Conservation and Recovery Act (RCRA). As stated in the Final Rule that incorporated the Third Edition of SW-846 (and its updates) into the RCRA regulations, use of this publication is required for only certain activities in the RCRA program. In other situations, this EPA publication functions as a guidance document setting forth acceptable, although not required, methods to be implemented by the user, as appropriate, in satisfying RCRA-related sampling and analysis requirements. These methods are intended to promote precision, accuracy, low bias, sensitivity, specificity, and comparability of analyses and test results. SW-846 includes several separate test methods addressing hundreds of analytes or compounds. For any given analyte or compound, multiple methods, with varying detection limits, are potentially available from this resource. USACE data needs focus on the use of SW-846 for the methods are comprehensive for many environmental matrices and chemical parameters, they are current with instrument capabilities and industry standards, and are flexible to adaptation based on project-specific requirements.

4.2.2 SW-846 Method Updates

SW-846 is a dynamic document that is subject to change as new information and data are developed. Advances in analytical instrumentation and techniques are continually reviewed by the EPA Office of Solid Waste and Emergency Response, and periodically incorporated into SW-846 updates to support changes in the regulatory program and to improve method performance. Any of these promulgated or draft SW-846 methods or other methods may be used by the USACE to support the project-specific requirements. However, it should be noted that recent SW-846 updates have deleted several methods where technology was considered outdated (i.e., packed chromatographic columns), as well as the incorporation of several new field screening methods. Therefore, it is advisable to maintain current knowledge of these method advances, and design projects taking advantage of the most recently promulgated methods.

4.3 GENERAL ANALYSIS REQUIREMENTS

The following sections outline the requirements for application of these requirements for HTRW projects. It is important to note that sporadic marginal failures are not accepted unless approved by the USACE Project Chemist. Problematic analytes should be identified and QC limits adjusted accordingly. Chemicals of concern should be specified in the SAP and corrective action should be taken if the QC criteria are not met. If chemicals of concern

have not been established, the laboratory should control on a representative subset (gas, ketone, ether, chlorinated solvent, etc.) to establish system performance and standard quality.

To ensure that quality data are continuously produced during analysis and allow the eventual compliance review, systematic QC checks are incorporated into the analyses to show test results remain reproducible and that the analytical method is actually measuring the quantity of target analytes without unacceptable bias. Systematic QC checks include the scheduled analyses of replicates, standards, surrogates, or spiked samples. *Method quality objectives (acceptance criteria or ranges) for these systematic QC checks are established to allow the review of data quality indicators providing an assessment of data usability and contract compliance.* This program of systematic QC checks may be viewed from two aspects, batch QC and matrix-specific QC.

4.3.1 Project Application

The requirements presented in this guidance shall be applied to all analytical methods unless specifically overridden by project-specific requirements. Target compound and analyte lists are variable and are dependent upon project-specific considerations. Examples of common target compound and analyte lists are included for eight SW-846 methods.

4.3.1.1 Method Development and Initial Demonstration of Capability. For each method performed, the laboratory shall maintain documentation that demonstrates each analyst's ability to perform the method within the sensitivity and precision/bias limits as stated in the published method, and any requirements outlined within the project SAP. Repeat these procedures when there is significant change in the method, instrumentation, or personnel. For each new method the laboratory shall perform and maintain documentation for the following:

- Develop a detailed standard operating procedure (SOP) before implementation of that method. Refer to EPA QA/G-9 for SOP requirements.

- Evaluate method sensitivity by performing an initial method detection limits (MDL) study for each matrix per Section 5.2.3.4.1.6 Sensitivity. Due to the difficulty in obtaining a solid interference-free matrix for metals determinations, process spiked reagent water for both the aqueous and solid digestion method to estimate aqueous and solid MDLs for graphite furnace atomic absorption (GFAA) and ICP analyses.

- Determine an appropriate practical quantitation limit (PQL) or method quantitation limit (MQL) and method reporting limit (MRL) for each compound and matrix based upon the calculated MDL and the guidance established in Section 5.2.3.4.1.6.

- Perform an initial demonstration for the method, noting all key employees' (i.e., technicians and analysts) ability to perform the method within the precision/bias limits as stated in the published method. A minimum of four laboratory control samples shall be carried through the method at the same time, or over a period of consecutive days. This control sample shall be obtained from an outside source, if available, or from a lot independent of the calibration standards. The concentration of each target analyte shall be approximately 10 times the MDL. Using the four results, calculate the mean recovery, and standard deviation for each parameter or target analyte of interest. Compare the laboratory's method precision and bias to the method performance summary presented within the published reference method. If any target analytes exceed the acceptance range, the performance is unacceptable. For all unacceptable target analytes or parameters, corrective actions shall be taken to locate the source of the problem, and repeat the test. The laboratory must maintain documentation for each analyst performing analysis.

4.3.1.2 Continuing Demonstration of Capability. Each analyst shall be required to demonstrate their continuing capability to perform any given method by ensuring the following:

- All applicable SOPs are kept current and represent the laboratory's current implementation of the method.
- The sensitivity of each method shall be demonstrated quarterly by analyzing the MDL check sample, or annually via an MDL study.
- Make any adjustments to the PQL, based upon noted changes in method sensitivity.
- Analyze a minimum of one (1) blind performance evaluation (PE) sample successfully on an annual basis.
- The precision and bias of the method shall be demonstrated by analyzing laboratory control samples and other quality control (QC) check samples with each batch of samples processed, and monitored by review of method control ranges/charts.

4.4 DATA FRAUD AND INAPPROPRIATE PRACTICES

The data produced by a laboratory typically provide the primary basis for environmental cleanup decisions and enforcement actions. The data may also end up in court. ***Data must be produced according to the project-specific requirements as specified in the final approved project documents.*** The laboratory must be aware of these requirements and be able to show that these requirements were followed. Documentation that would clearly show how all of the analytical values were obtained must be maintained. ***The unfortunate aspect of data fraud/inappropriate practices, is the inability to readily detect them without significant cost. Project quality assurance (QA) procedures employed should be designed to help deter and expose any misrepresentation of data. Refer to Section 5.0 for information on application of various QA procedures to aid in the prevention of fraudulent activities. Best Practices for the Detection and Deterrence of Laboratory Fraud, California Military Environmental Coordination Committee, Chemical Data Quality/Cost Reduction Process Action Team, Version 1.0, March 1997 provides guidelines for laboratory fraud deterrence.***

4.4.1 Data Fraud

Data fraud can be loosely defined as a gross deviation from contract-specified or method-specified analytical practices, combined with the intent to conceal the deviation. The difference between poor analytical judgement and fraud may be assessed in the documentation of intent within laboratory records. Gross deviations from specified procedures should be investigated for potential fraud, and findings of fraud prosecuted to the fullest extent of the law. A few examples of fraudulent practices have been identified below:

- Inappropriate use of manual integration to meet calibration or method QC criteria would be considered fraud. For example, peak shaving or peak enhancement is considered fraudulent activities if performed solely to meet QC requirements.
- Time travel of analyses to meet method 12-hour clock requirements.
- Falsification of results to meet method requirements.
- Reporting of results without analyses to support (e.g., "dry-labing").

4.4.2 Inappropriate Practices

Several inappropriate practices have also been identified which deserve prudent action. ***Issues of this caliber should not be tolerated and corrective action taken immediately to resolve all matters between the laboratory and the data user.*** These inappropriate practices may include:

- Selective exclusion of data to meet QC criteria (i.e., initial calibration points dropped without technical or statistical justification).

- Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports which are not linked to the data set reported, or QC control limits presented within the laboratory QA Manual, which are not indicative of historical laboratory performance or used for batch control.

- Notation of matrix inference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices [e.g., method blank or laboratory control sample (LCS)].

To avoid miscommunication, a laboratory must clearly document all errors, mistakes, and basis for manual integration within the case narrative. Include corrective actions when necessary, and provide appropriate peer review of this information. ***Notification should also be made to the appropriate people such that appropriate corrective actions can be initiated.*** It is recommended that laboratories also maintain an electronic audit trail that clearly shows all changes to data, who made the change, date, and why.

4.5 ANALYTICAL STANDARDS PREPARATION AND TRACEABILITY

The laboratory shall have, in-house, the appropriate standards for all target analytes. These standards can either be prepared from neat-high purity bulk materials or purchased as certified solutions. A critical element in the generation of quality data is the purity/quality and the traceability of the standard solutions and reagents used in the analytical operations. Primary reference standards and standard solutions used by the laboratory shall be obtained from reliable commercial sources (i.e., NIST, EPA, etc.) to ensure the highest purity possible. Certificates shall be available upon request that verify each standard's purity or concentration. The use of correction factors for all standards that are not at least 99.9% pure for inorganic analyses and 96% pure for organic analyses will be required. Care should be exercised in the proper storage and handling of all standards and standard solutions. The laboratory shall continuously monitor the purity or quality of reagents and standard solutions through a series of well-documented procedures. Requirements for standards re-preparation shall be based on unacceptable performance. For example, initial calibration standards shall be verified with a freshly prepared initial calibration verification (ICV). For analyses that allow analytical sequence initiation by a continuing calibration verification (CCV) standard, the frequency of standard re-preparation will be based on whether standard performance is compliant with the method acceptance criteria. The quality of CCVs failing to meet method criterions should be verified against a freshly prepared CCV. In general, stock and working standards shall be checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. All standards and standard solutions shall be fully documented to verify compliance with USACE requirements.

4.5.1 Sample Screening

It is highly recommended that the laboratory screen samples or extracts by methods of their choice to determine which target analytes are present and at approximately what levels.

4.5.2 Target Compound Lists

Target analyte lists necessary for a project should be identified within project contract documents based upon project-specific data quality objectives. However, for instances where a particular SW-846 method is specified but the target analyte list for the method is not, Tables 4-1 through 4-6 may be used to identify target analyte lists for the

following SW-846 methods: 8021, 8081, 8082, 8260, 8270, and 8330. These lists were compiled of target compounds common to the various versions of each SW-846 method. Note however, that the most recent revision of several organic methods may contain additional target compounds not included here. The organic target analyte lists (Tables 4-2, 4-3A, 4-4, 4-5A, and 4-5B) were augmented to include those compounds included within the Target Compound List (TCL) as defined by the EPA Contract Laboratory Program (CLP). ***Each list should be reviewed based upon project data needs and edited accordingly. Special considerations for target compound reporting for the following methods should be evaluated and clearly identified within project contract documentation.***

4.5.2.1 Method 8021 – Volatile Organic Compounds (VOCs) by GC/PID-HECD. The target analyte list for Method 8021 includes those analytes previously associated with deleted SW-846 methods 8010 and 8020 and some additional target analytes. ***Therefore, depending upon project requirements, the entire 8021 target analyte list or a subset may be specified for the project. The following target analyte lists may apply: (1) the full 8021 target analyte list, (2) halogenated volatile compounds (HVOs) (compound list from deleted Method 8010), (3) aromatic volatile compounds (AVOs) (compound list from deleted Method 8020), (4) benzene, toluene, ethylbenzene, and total xylenes (BTEX), and/or 5) methyl tertiary butyl ether (MTBE).***

4.5.2.2 Method 8081 – Organochlorine Pesticides by GC/ECD. ***Note whether multi-component pesticides (i.e., chlordane and toxaphene) are actually compounds of concern.*** The additional instrument and method QC samples required for these multiple-component analytes significantly increase the level of effort for this method. ***It should also be determined if chlordane quantitation should be performed and reported as technical chlordane or the individual chlordane isomers (i.e., alpha and gamma chlordane).*** In the absence of guidance to the contrary, assume that quantitation is required for toxaphene, and the individual chlordane isomers (rather than for technical chlordane). Recently promulgated revisions of Method 8081 do not include polychlorinated biphenyls (PCBs) as target compounds. Therefore, guidance on PCB reporting is not included here. Reference Section 4.5.2.3 for additional information on reporting considerations for PCBs.

4.5.2.3 Method 8082 - PCBs by GC/ECD. Regulatory aspects of PCBs are based upon the quantitation as Aroclors. However, when not used for regulatory purposes and depending upon project requirements, the analytical results may be reported as individual PCB congeners, or the values summed over an appropriate chromatographic range and reported as total PCBs. When weathered PCBs are encountered and the data use requires the use of Aroclors, then the quantitation as Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Peaks within the sample chromatogram not related to PCBs should be subtracted from the total area. Full documentation of this approach must be provided in the case narrative when this option is chosen. Caution should be exercised when using differing quantitation techniques, comparability of project data may be reduced if new data is handled significantly different from previous data. Studies have shown that concentrations derived from samples as Aroclors were larger than those determined using the congener method. Due to the potential regulatory aspect and unless otherwise indicated, all samples must be analyzed for the PCB compounds as Aroclors.

4.5.2.4 Method 8330 - Explosives by HPLC. Due to the lack of resolution between 2,4-DNT and 2,6-DNT, and between 2-Am-DNT and 4-Am-DNT, reporting of these compounds may be combined and reported as 'isomeric pairs'.

4.6 ANALYTICAL METHODS SUMMARY

The guidance introduces two (2) inorganic (6010, 7000) and six (6) organic (8021, 8081, 8082, 8260, 8270, 8330) SW-846 analytical methods. ***The guidance has deliberately omitted method revision numbers from the analytical method designations, so as to enforce its application to any revision of the method in use by USACE. Note also that many of the QA/QC principles and policies included herein, apply to methods not directly addressed.*** Technical details on the eight methods implementation and default limits for performance-based QC parameters are

presented. ***As noted, these acceptance limits are considered a baseline standard, but may be modified based upon project-specific DQOs. Reference USACE Engineering Manuals EM 200-1-2, Technical Project Planning Process guidance for information on the establishing project DQOs, and EM 200-1-6, Chemical Quality Assurance for HTRW Projects for a review of Chemical Data Quality Management (CDQM) elements available to aid in the design of a project chemical data quality management program. Project-specific contract documents (e.g., scopes of work, guide specifications, etc.) should reference or identify all applicable analytical methods and QC elements necessary for the project to assure correct and accountable execution of the work.*** If, however, this information is not adequately defined, then the laboratory shall default to using the latest promulgated revision of the appropriate SW-846 method, and application of the QC acceptance limits described herein as the default USACE requirements. The following guidance outlines general requirements, which apply uniformly to all methods by subject heading with any additional parameter or method-specific requirements presented in subsequent sections by chemical parameter, analytical technique, or the individual chromatographic method.

4.6.1 Inorganic Analytical Methods. The inorganic methods presented focuses exclusively on metals' analyses. This encompasses inductively coupled argon plasma-emission spectroscopy (ICP) and graphite furnace-atomic absorption spectroscopy (GFAA), and cold vapor-atomic absorption (CVAA) methodologies. Project inorganic method requirements should be clearly identified based on project DQOs. ***Note than when the quantitation limit of a metal (e.g., Sb, Pb, As, Tl, and Se by ICP) is higher than the project-required action level, an alternate analytical method capable of achieving a lower quantitation limit for that metal should be used.*** Baseline inorganic QC requirements are discussed in subsequent sections by the individual method, and summarized in attached tables.

Classical (wet chemistry) techniques are not addressed directly within this guidance. However, the field of conventional, non-metals analysis involves a variety of instrumental and wet chemical techniques. Instruments include spectrophotometers and other analyzers.

4.6.1.1 Inorganic Preparatory Methods. Several preparatory method options may exist for each determinative method and matrix. However, comparability of the data generated from different preparatory procedures is not guaranteed, nor likely. ***Therefore in order to ensure comparability of data generated throughout the life of a project or between different laboratories, proper preparatory methods must be clearly identified for each chemical parameter/matrix and maintaining consistent analytical protocols.***

Aqueous liquid samples for ICP may be processed by a hotplate technique following Methods 3005 or 3010, or by using a microwave technique following Method 3015. Aqueous liquid samples for GFAA are processed by a hotplate technique following Method 3020, or using a microwave technique following Method 3015. ***When a comparison of dissolved metals and total recoverable metals results are anticipated, it is recommended that both the field-filtered and non-filtered water samples be subjected to the proper digestion procedures (preparatory method) prior to analyses. This ensures a matrix matching of the acid concentrations between the samples. If only dissolved metals' results are required, the preparatory method is optional, and analysis by direct aspiration is allowed. Under these circumstances and per method requirements, the calibration standards must be changed to matrix match the samples analyzed. The matching of acid concentrations between samples and standards assures similar viscosity and surface tensions, which affect aspiration characteristics and thus may vary the resulting concentrations.*** Solid samples are processed for ICP and GFAA by hotplate following Method 3050, or by microwave following Method 3051. Preparatory procedures for the CVAA analysis of mercury are incorporated into the individual analytical methods (7470 for liquids and 7471 for solids).

Proper preparatory procedures to be employed should be identified within the project contract documents (e.g., SOW, SAP, guidance specification, etc.). When the method of digestion is not specified, the laboratory must attempt to obtain this information from appropriate USACE project technical personnel. In lieu of project

specific information, the default preparatory procedures shall follow hotplate techniques following Method 3005 for ICP and Method 3020 for GFAA (3005 for antimony) for aqueous matrices, and Method 3050 for solid matrices. It should be noted that future updates of SW-846 are anticipated to combine these preparatory methods under a common methodology.

4.6.1.1.1 Method 6010. This method is used to determine the concentrations of select metals in the digestates of liquid and solid matrices, using inductively coupled plasma-atomic emission spectroscopy (ICP-AES). The requirements apply to simultaneous or sequential ICPs. ICPs may be equipped with a torch which is viewed from the radial or axial (e.g., trace ICP) position. For the ICP, Mass Spectral (MS) detectors are also available.

4.6.1.1.2 Method 7000. The SW-846 7000 series methods are used to determine the concentrations of select metals in the digestates of liquid and solid matrices, employing flame, graphite furnace, gaseous hydride and cold vapor techniques in conjunction with atomic absorption spectroscopy (AAS). This discussion will be limited to graphite furnace-atomic absorption (GFAA), with an appropriate background correction system. Recommend GFAA instruments have a Zeeman background correction capability. ***Graphite furnace atomic absorption (GFAA) is commonly used for several elements due to its sensitivity capabilities.*** It should be noted that the proposed Update IV of SW-846 includes all GFAA methods being combined under Method 7010. Mercury shall be analyzed by a cold-vapor AA technique. The AA can be integrated with an appropriate cold vapor accessory for mercury analyses, or independent cold vapor units are also available.

4.6.2 Organic Analytical Methods

The principles and QC requirements established within SW-846 Method 8000 apply to all organic chromatographic methods (e.g., GC, GC/MS, and HPLC methods). Therefore, they are presented generally by topic. *Packed-column methods were formally deleted from SW-846 with the promulgation of SW-846 Update III on June 13, 1997.* These methods, in general, possessed less stringent performance criteria (e.g., column resolution is lower and method QC is less stringent) than their associated capillary column method. ***Due to these factors, the laboratory should default to the use of capillary column methods, (e.g., Methods 8260B, 8081A/8082, and 8021B for the deleted Methods 8240, 8080, and 8010/8020, respectively).*** The laboratory shall not use capillary columns in conjunction with packed column methods in order to apply less stringent QC criterion.

4.6.2.1 Organic Preparatory Methods. Several preparatory method options may exist for each determinative method and matrix. However, comparability of the data generated from different preparatory procedures is not guaranteed nor likely. ***Therefore in order to ensure comparability of data generated throughout the life of a project or between different laboratories, proper preparatory methods must be clearly identified for each chemical parameter/matrix and maintain consistent analytical protocols.*** Liquid samples may be prepared for extractable organic analyses using a separatory funnel following Method 3510, a continuous liquid-liquid extractor following Method 3520, or solid-phase extraction by Method 3535. Liquid samples for purgeable organic analyses utilizing purge and trap procedures follow Method 5030. Nonaqueous samples should be prepared by solvent dilution techniques following Method 3580 for extractable organic analyses and Method 3585 for purgeable analyses. Solid samples may be processed for extractable organic analyses by soxhlet extraction procedures following Method 3540, automated soxhlet by Method 3541, pressurized fluid extraction by Method 3545, or ultrasonic extraction procedures by Method 3550. For petroleum hydrocarbons analysis, a supercritical fluid extraction may be used following Method 3560. ***Typically, Method 3550 (sonication) is used to prepare solid samples known to have high concentrations of target compounds, whereas Method 3540 (soxhlet), 3541 (soxhlet), and 3545 is generally used in an unknown situation or when low level concentrations are known or suspected.*** Solid samples for purgeable organic analyses utilize Method 5035. ***Several notable changes in the protocols covering soil sampling, analysis, preparation have occurred with the promulgation of Method 5035. These changes will require a significant increase in the coordination between field and laboratory personnel. Refer to USACE policy guidance titled USACE Sample Collection and Preparation Strategies for Volatile***

Organic Compounds in Solids for details on implementation. When the method of preparation is not specified, the laboratory must attempt to obtain this information from appropriate USACE project technical personnel. If no information is provided for the project specific preparatory methods required, the default preparatory procedures for extractable organic analyses shall follow Method 3520 for aqueous samples; Method 3540 or 3541 for solid samples; and those noted above for purgeable organic analyses.

It is anticipated that project fieldwork will entail the use of proper sample handling protocols, which result in the acquisition of a representative sample. These include the use of appropriate sample containers, obtaining sufficient sample volumes, and proper preservation techniques based on the anticipated chemical analyses. Refer to EM 200-1-3 for information on proper sample containers, sample volumes, and preservatives necessary. As noted in section 5.1 these items are verified upon sample receipt, and any discrepancies notified back through appropriate channels. For chemical parameters which do not allow this assessment during sample login (e.g., VOCs), verification is done post sample sub-sampling or analysis, and any problems are noted within the case narrative.

Whenever possible, a quantitative transfer of the entire (1-Liter) aqueous liquid sample is done to ensure there is no loss of target compounds through the adhesion of contaminants on the walls of the sample bottle. A solvent rinse should be performed to avoid this loss. This procedure; however, may not be possible when significant amounts of sediment are present within the water sample. ***Due to the problems these fines may invoke on the extraction process, recommend that appropriate project technical personnel be contacted to verify the procedures to employ. (e.g., decanting water sample, physical separation of the phases and subsequent analysis of each, etc.)*** To avoid cross-contamination, the laboratory should mark the meniscus on the liter sample container, pour out the sample into the extraction apparatus, and solvent rinse the sample container. To determine original sample volume the laboratory should pour water to original sample volume, transfer the water to a class-A graduated cylinder and record the volume.

4.6.2.2 Organic Cleanup Methods. If significant non-target interference exists, corrective action shall include implementing appropriate cleanup procedures. Dilution techniques should not be used in preference to cleanup procedures for organic methods. The laboratory shall have a minimum capability of at least one cleanup method for each type and range of organic analyses it provides services. Refer to the individual determinative methods and Method 3600 to identify recommended cleanup methods based on the type and concentration of interferences present, the selectivity of the determinative method, and project method reporting limit requirements. However, analyst professional judgement should also be used to identify appropriate cleanup techniques to employ. ***If cleanup procedures are not routinely employed by a laboratory, a formal notification procedure must be in place to advise the client of this.***

4.6.2.3 Method 8021. This method is used for the analysis of select volatile organic compounds in aqueous and solid matrices by purge and trap device according to methods prescribed above and subsequently analyzed by GC using a HECD and PID in series.

4.6.2.4 Method 8081. This method is used to determine the concentrations of select organochlorine pesticides in the extracts of liquid and solid matrices, using fused-silica capillary columns with an electron capture detector (ECD). ***Method 8081A no longer includes PCBs as target compounds to eliminate the complications inherent to the combined pesticide/PCB analysis. Therefore, PCB analysis must be performed using Method 8082. This may be accomplished by submitting an additional environmental sample for PCB analysis; or the extract may be split prior to implementation of any cleanup procedures, processing individual extract portions for pesticide analysis following Method 8081 and the other portion for PCB analysis following Method 8082.***

4.6.2.5 Method 8082. This method is used to determine the concentrations of select polychlorinated biphenyls (PCBs) as the seven Aroclors, as individual PCB congeners, or as total PCBs in the extracts of liquid and solid matrices, using fused-silica capillary columns with electron capture detectors (ECDs). ***Refer to project required chemical parameters and Section 4.6.8 in order to determine the necessity for an additional environmental sample for PCB analysis, or the use of an aliquot from the extract (prior to cleanup procedures) for both pesticide and PCB analyses.***

4.6.2.6 Method 8260. This method can be used for the analysis of select volatile organic compounds (most compounds with boiling points below 200°C) in aqueous and solid matrices by purge and trap device according to methods prescribed above and subsequently analyzed by GC/MS. Volatile water-soluble compounds can be analyzed with this method but higher quantitation limits may apply. A notable deviation allowed by Method 8260B (vs. 5030) is the utilization of a heated purge for aqueous samples.

4.6.2.7 Method 8270. This method is used to analyze the extracts of aqueous and solid samples for semivolatile organic compounds (SVOCs), also referred to as base/neutral and acid extractables (BNAs). The extracts are analyzed by GC/MS using a capillary column.

4.6.2.8 Method 8330. This method is used for the analysis of select explosives in the extracts of solid and liquid matrices. The extracts are analyzed by high performance liquid chromatography (HPLC) with a UV detector, using C-18 and cyanide reversed-phase columns as the primary and confirmatory columns, respectively. The method specifies extraction procedures for solid samples, and low-level and high-level aqueous samples. In general, aqueous samples for low concentration are extracted by a salting-out extraction procedure using acetonitrile and sodium chloride. Aqueous samples for the high concentration is diluted with acetonitrile, filtered, and analyzed by direct injection. Soil and sediment samples are extracted using acetonitrile in a cooled ultrasonic bath and filtered prior to analysis. ***Project-specific approval should be sought for the use of solid phase extraction (SPE - Method 3535) in lieu of the low-level salting out procedure described in Method 8330, or the use of a photodiode ray detector as the confirmation technique.***

4.7 PRELIMINARY METHOD SET-UP

In addition to the general items noted in Section 4.6, method initiation must include the following procedures as applicable.

4.7.1 Inorganic Analyses - Method 6010

The following sections outline the general procedures for method initiation for Method 6010.

4.7.1.1 Linear Dynamic Range. The upper limit of the linear dynamic range for each ICP must be determined for each analyte wavelength used in order to determine an appropriate concentration for the high calibration standard. This is done for each analyte by analyzing successively higher standard concentrations (approximately 3 to 5 standards) until--because of curvature--the highest analyte concentration is ± 10% of the "expected" concentration obtained by extrapolating the calibration line from the lower standards. The concentration chosen for the highest standard must then be chosen below the upper limit of the linear range. The linear dynamic range must be checked initially and whenever there is a significant change in instrumental hardware or operating conditions. If the ICP is routinely calibrated using one standard and a blank, the linear dynamic range must be checked every six months.

4.7.1.2 Interelement Spectral Correction Factors. All interelement spectral correction factors must be determined per method requirements initially and updated at least once every six months, based upon failure of the interelement check standard, or whenever there are significant instrument modifications.

4.7.2 Organic Analyses - Methods 8000 Series

Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Excessively wide retention time windows may result in false positive results that cannot be confirmed upon further analysis. Retention time windows must be determined as specified in the latest revision of Method 8000 for all chromatographic methods, except when mass spectroscopy (MS) or infrared (IR) detectors are employed. Calculate absolute retention time windows for each compound and surrogate for each chromatographic column employed per method instructions. New retention time windows must be established whenever a new chromatographic column is installed, or when there are significant changes in the operating conditions. The use of reasonable "default" values, programmed into instrument software for the width of the retention time window is allowed if (1) the laboratory demonstrates that the calculated three-sigma width is consistently less than the default width, and (2) the default width is not "excessively large" (i.e., more than 1% to 2% of the absolute retention time).

4.7.2.1 Method 8081. Retention time windows must be established as specified in Section 4.7.2 for each surrogate and single-component pesticide target compound, and for at least three to five characteristic peaks of multiple-component pesticides. For multi-component pesticide standards, the analyst should also rely heavily on pattern recognition and the analyst's experience in the interpretation of the chromatograms.

4.7.2.2 Method 8082. Retention time windows will vary based upon the project requirements for PCB quantitation as noted in Section 4.6.7.3. Absolute retention times will be used when identification of PCBs as Aroclors is performed. Retention time windows must be established as specified in Section 4.7.2 for each surrogate and congeners or for at least three to five characteristic peaks of each Aroclor. If PCB congeners are quantitated, normally internal standard calibration techniques are used and relative retention times are determined.

4.8 INSTRUMENT PERFORMANCE CHECKS

Several methods outline additional QC procedures to verify the instrumentation is in good working condition. These QC samples must be analyzed and meet method-specified acceptable limits prior to commencing sample analyses.

4.8.1 Method 6010 - Interference Check Standard (ICS). An ICS (interference check standard) must be analyzed at the beginning of the analytical sequence to verify the correction factors established in Section 4.7.1 are valid. The ICS typically consists of a set of solutions: ICS-A contains only the interferents (at relatively high concentrations) and ICS-AB contains both the interferents and the analytes of interest. The interferents in both solutions must be present at the concentration that is at least as high as the high-level calibration standard. The ICS-AB solution must contain the analytes of interest (the metals which are not interferents) at concentrations approximately mid-level. The metals of interest in the ICS-AB solution must be within 20% of their expected values. When the ICS check is unacceptable, take corrective action to remedy the failure. Check that the background correction factors applied are appropriate, and readjust if necessary. If the ICS fails immediately after the daily initial calibration, recalibrate and reanalyze the ICS. If the ICP can display over corrections as negative readings, then the ICS-A solution alone may be used to check for interferences. If the analytes of interest are within two times the absolute value of the MDLs (± |MDLs|), the ICS check is acceptable and the ICS-AB solution need not be analyzed.

4.8.2 Method 8081 - Injection Port Inertness Check. Verify injection port inertness by performing %Breakdown checks for 4,4'-DDT and endrin as specified in Method 8081. The mid-level standard containing only endrin and 4,4'-DDT must be analyzed at the beginning of the analytical shift/sequence, before the initial calibration or the continuing calibration verification. If the %Breakdown is not less than 15% for either DDT or endrin, perform

injector maintenance (e.g., column clipping). Do not proceed with the calibration or analysis until the %Breakdown for each compound is less than 15%.

4.8.3 Methods 8260 and 8270 - Mass Spectrometer (MS) Tuning. Verify that the MS meets standard mass spectral abundance criteria prior to initiation of any analyses by the injection of 4-bromofluorobenzene (BFB) tune standard for Method 8260 and decafluorotriphenylphosine (DFTPP) for Method 8270. The tune standard must be analyzed: (1) At the beginning of the analytical shift/sequence and (2) every 12 hours of continuous analysis. The 12-hour clock starts at the time of injection of the tune standard. Recommend evaluating the ion abundance by using any of the following scan scenarios: (1) use one scan at the apex peak, (2) use the one scan either directly preceding or following the apex, (3) use the mean of the apex and the proceeding and following scans, or (4) use the average across the entire peak. The tune must satisfy the ion abundance acceptance criteria listed within the appropriate method. Background correction should be compliant with method specifications and employ only for the purpose of correcting for instrument background ions. If a 12-hour tune fails, take corrective action (e.g., clean the MS source) and re-inject the tune standard (BFB/DFTPP). Do not proceed with analysis until the tune is acceptable. Once an acceptable tuning procedure has been established for the GC/MS analyses, the procedure must be documented in the SOP and consistently performed by all analysts performing analysis.

4.8.4 Method 8270. In order to verify column condition and injection port inertness, the DFTPP tune standard shall contain appropriate volume of 4,4'-DDT, benzidine and pentachlorophenol as stated within Method 8270.

4.8.4.1 Injection Port Inertness Check. Similar to Method 8081, the injection port inertness of the GC portion of the GC/MS is evaluated by the %Breakdown of 4,4'-DDT. This procedure is done to verify acceptable instrument performance, regardless of whether DDT is a target compound. The %Breakdown of 4,4'-DDT to 4,4'-DDE and 4,4'-DDD should not exceed 20%, in order to proceed with calibration procedures.

4.8.4.2 Column Performance Check. The condition of the GC column is evaluated by the tailing of benzidine and pentachlorophenol (PCP). Benzidine and pentachlorophenol must be present at their normal responses, with no visible peak tailing, as demonstrated by the peak tailing factors. The acceptance criteria for the peak tailing factor for benzidine is < 3.0 and pentachlorophenol is < 5.0.

4.9 CALIBRATION PROCEDURES AND FREQUENCIES

The calibration of instruments and support equipment are required to ensure that the analytical system is operating correctly and functioning at the proper precision, bias (accuracy) and sensitivity. ***The frequency of calibration and calibration verification are presented below, based upon by the various analytical methods, industry standards, or may be changed based upon project-specific DQOs.*** Tables 4-7 through 4-14 are enclosed to highlight key information on calibration procedures and acceptance limits for each SW-846 method discussed.

4.9.1 Analytical Support Areas Calibration Verification. Suggest referring to the Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid, ASTM D5522-94, Annual Book of ASTM Standards, for additional details on the following procedures and performance criteria. Thermometers must be calibrated as specified by the ASTM standard. All thermometer calibrations must be traceable to the instrument used for calibration.

4.9.1.1 Balances. The calibration of analytical balances shall be verified on first daily use at a mass or masses which bracket, or are representative of the measurements routinely performed at that balance. The quality of the weights used for this calibration verification shall be documented and in accordance with the quality requirements established within the referenced ASTM standard. Balance calibration verifications shall be documented in appropriate logbooks. Acceptance criteria shall be clearly identified. Apply a 1% performance criterion to top-loading balances, and 0.1% to analytical balances. Refer to Standard Test Method of Testing Top Loading, Direct-

Reading Laboratory Scales and Balances, ASTM Methods Vol. 14.02 E 898-88, June 1990 and Standard Practice for the Evaluation of Single-Pan Mechanical Balances, ASTM E 319-85, Annual Book of ASTM Standards for additional details.

4.9.1.2 Refrigerators/Freezers. All refrigerators and freezers shall be monitored for proper temperature by measuring and recording internal temperatures on a daily basis. The calibration of all thermometers used for these measurements shall be verified at least annually against NIST-certified or NIST-traceable thermometers. Electronic thermometers shall be calibrated at least quarterly. Temperatures shall be recorded in appropriate logbooks. Acceptance ranges shall be clearly identified. Maintain refrigerators to 4 ± 2^0C, and freezers to -10 to -20^0C. Refer to Standard Test Method for Inspection and Verification of Liquid in Glass Thermometers. Refer to ASTM Methods Vol. 14.03 E 77-89, June 1990 for additional details on thermometers calibration.

4.9.1.3 Pipets and Other Volumetric Labware. All volumetric devices, glassware, or labware shall be initially inspected, and all cracked or damaged items pulled from use. The calibration of variable volume Eppendorf-type pipets shall be verified at the volume of use, or at two volumes which bracket the range of use on the day of use, or at a minimum of weekly. The calibration of all fixed volume Eppendorf type pipets shall be verified monthly. In addition, the accuracy of all nonstandard labware (K-D tubes, Zymark tubes, plastic cups, centrifuge tubes, etc.) used to measure the initial sample volume, or final volume of sample extracts/digestates must be verified. Accuracy must be verified to within 3%. If the check reveals greater than 3%, steps should be taken to improve the accuracy of these measurements, or use alternative procedures, which meet this requirement. It is also recommended that the calibration of all other volumetric glassware (flasks and pipets) be verified at the time of purchase for each lot of labware received. Each calibration check shall consist of at least three measurements, the average calculated, and recorded in appropriate logbooks. Refer to Standard Practice for Calibration of Volumetric Ware, ASTM Methods Vol. 14.02 E 542-94 for additional details.

4.9.1.4 Water Supply System. The laboratory shall maintain an appropriate water supply system that can furnish high purity water that can meet the needs of the various analytical areas. Method blanks' performance provides an indication of the source water suitability for the analysis. However, the water supply system should be monitored on a regular basis (i.e., daily or before use) by conductivity readouts or implementation of general chemistry parameters. Appropriate general chemistry parameters should be based upon the analysis performed at the laboratory. Refer to ASTM D 1193-91, Standard Specification for Reagent Water for additional details.

4.9.1.5 Other Analytical Support Equipment. Other support equipment used to maintain appropriate temperatures as prescribed within the analytical method (i.e., hotplates, water baths, etc.) should be monitored for compliance with the method-specified ranges. Recommend notation of any critical times or temperatures onto appropriate bench sheets or laboratory logbooks. All HVAC systems must be routinely monitored for potential contamination of the analytical system. Chronic long-term or systematic contamination is not acceptable. Corrective action procedures must be initiated upon detection of ambient or systematic contamination. Resolution of the nonconformance must be documented.

4.9.2 Initial Calibration Curve. An analytical instrument is considered calibrated when an instrumental response can be related to the concentration of an analyte. This relationship may be depicted graphically, and referred to as a 'calibration curve'. Initial calibration curves must be established based upon the requisite number of standards identified within the method for each target analyte (and surrogate for organic analyses). As described in Section 5.2.3.4.1.6, the practical quantitation limit(s) shall be established by the laboratory at the low standard for each target analyte. All reported concentrations for target analytes shall be within the high and low initial calibration standards. Data generated below the low standard shall be reported as estimated (J-flag) values. Data generated above the high standard shall be diluted into the calibration range and reanalyzed. The frequency requirements for

the initial calibration vary amongst the individual methods and are presented below. Tables 4-7 through 4-14 highlight key information on initial calibrations by method also.

4.9.2.1 Inorganic Analyses. For metals analyses, an initial calibration must be performed at the beginning of each analytical shift, and when a CCV fails or significant instrument maintenance is performed. Linearity is acceptable only if the linear regression coefficient $r > 0.995$. If $r > 0.995$, take corrective action and recalibrate.

As previously noted, classical (wet chemistry) techniques are not addressed directly. But while calibration and standardization procedures vary depending on the type of system and analytical methodology, the general principles outlined in these calibration sections apply universally. Analytical systems for wet chemistry techniques shall be calibrated prior to analyses being conducted. The calibration consists of defining the working range by use of a series of standard solutions. A minimum of five to seven standards is typically used. The calibration shall be verified on an ongoing basis (every ten to twenty samples at a minimum and at the end of the analysis sequence) to ensure that the system remains within specifications.

4.9.2.1.1 Method 6010. The term "standard" may refer to a "mixed" standard solution containing all the metals of interest (when the metals are compatible) or to a set of standard solutions where each standard contains a subset of the (compatible) metals of interest. The initial calibration must be established following one of the options presented below.

- Calibration Option 1. Perform the initial calibration with a high-level standard and a calibration blank. The concentration of the single standard establishes the linear calibration range, and must fall below the upper linear dynamic range of the instrument (see Section 4.7.1.1). To ensure accuracy of concentrations at the PQL, verification at a low-level standard is prepared from the primary source standard and results must be within ± 20% of its expected value. If the 20% criterion cannot be consistently met, then the concentration of the daily low-level CCV standard (and associated quantitation limits) should be increased until compliance is attained. If the PQL check standard fails during execution of the analysis, the laboratory PQLs must be evaluated for compliance with the project specific requirements and the data quality objectives.

- Calibration Option 2. The ICP-AES may be alternatively calibrated with three standards and a calibration blank. Evaluate linearity as described in Section 4.9.2.1. The concentration of the low-level calibration standard must be set no lower than the PQL for each analyte. The concentration of the high-level standard establishes the linear calibration range, and must fall below the upper linear dynamic range of the instrument (see Section 4.7.1.1).

All standards and samples analyzed shall have a minimum of three exposures and the mean of each set of exposures used for quantitation. The exposure times should be optimized for instrumental response and analysis time. Evaluate the RSD for high-level and mid-level standards and calibration verification standards to < 5%. Take corrective action (e.g., recheck the appropriateness of the exposure time) and recalibrate if the QC criteria are not met.

4.9.2.1.2 Method 7000. An initial calibration for GFAA must be established from at least three standards and a calibration blank. CVAA calibration requirements are similar to the standard AA procedures but with a minimum of 5-points. Evaluate linearity as described in Section 4.9.2.1. For GFAA a minimum of duplicate injections shall be performed for all standards and samples to improve precision and help reduce furnace pipetting uncertainty. The RPD between duplicate injections for all standards shall be < 10%. If unacceptable, reanalyze the standard. If still unacceptable, perform instrument maintenance as needed to correct the problem and recalibrate.

4.9.2.2 Organic Analyses. The initial calibration curve is established as specified in the individual methods, using (a minimum of) five standards for all single-component target compounds and surrogates, and at least three standards for multiple component target compounds (e.g., toxaphene, chlordane, and PCBs). Care should be exercised to avoid using inappropriate practices identified in Section 4.4.2. Once verified, an initial calibration is valid until a CCV fails or significant instrument maintenance is performed. The shapes of calibration 'curves' are typically a linear function between the concentration of each target compound to the instrument response. However, many method target compounds listings have been expanded to include compounds, which cannot be optimized without application of models for quadratic or higher order mathematical functions. When these models are employed, additional standards must be analyzed to accurately delineate the relationship as outlined in Method 8000B.

Linearity may be determined using linear regression analysis for each target compounds by calculating the "correlation coefficient" (r). The resulting line would normally not be forced through the origin, or use the origin as a calibration point, unless it is demonstrated that the intercept of the regression line is not statistically different from zero at the 95% level of confidence. Another term used to describe the goodness of fit of the line is 'Coefficient of Determination' (r^2), the squared correlation coefficient). Alternatively for chromatographic methods, the average calibration factor (CF) or response factors (RF) may be calculated for each target compound. Linearity may be evaluated by calculating the percent relative standard deviation (%RSD) of the CFs/RFs from the initial calibration standards for each target compound. Linearity is presumed if the "correlation coefficient (r) is equal to or greater than 0.995 or the coefficient of determination (r^2)" is equal to or greater than 0.99, or if the %RSD is less than or equal to 15% or 20% (depending on the method specifications). A visual inspection of the calibration curve should also be used as a diagnostic tool when nonlinear behavior is observed to verify if there is a large percentage error in any particular portion of the calibration curve. If the visual inspection indicates problems, or if one of the above criterions is not met, then the laboratory shall evaluate the following items for implementation based on an understanding of the detector response/contaminant concentration relationship:

- Check the instrument operating conditions or the initial calibration standards used and make adjustments to achieve a linear calibration curve.

- Narrow the calibration range using the same number of standards as required by the individual method. In general, the highest standard would be lowered first. The consequences of all actions taken must also be addressed, i.e., reduction of the calibration range, raising of the PQL, etc.

- Evaluate the use of a nonlinear calibration curve, when applicable. When nonlinear calibration models are used, the resultant line should not be forced through the origin and the origin should not be used as a calibration point. No higher than a third order (cubic) calibration model shall be used. Note that when a nonlinear calibration model is employed, more data points are needed to maintain at least three degrees of freedom. For example, use of a quadratic function requires a six-point initial calibration curve. The resulting 'coefficient of determination' (r^2) should be greater than or equal to 0.99 for this to be considered acceptable.

- Use of alternative techniques (e.g., relative standard error (RSE)) outlined in the EPA Memorandum titled, Clarification Regarding Use of SW-846 Methods, dated 7 August 1998.

- Despite implementation of the above alternatives, method limitations may exist which make the acceptance criteria unattainable for all target compounds. Therefore, SW-846 has incorporated an allowance to evaluate the mean of the RSD values for all target compounds in the calibration is less than the method acceptance criterion. To avoid the inclusion of target compounds showing gross method failure, this approach may be utilized as long as the target compounds do not exceed the criteria established for poor performers in the enclosed method-specific tables. ***If the averaging option is***

employed, the laboratory must communicate the following information within the case narrative to the client: summary of all of the target compounds exceeding method acceptance criteria, the individual RSD results for those compounds, and the mean RSD calculated.

4.9.2.2.1 Method 8021. Apply the principles as stated in Section 4.9.2.2 and summarized in Table 4-9. Poor performers for Method 8021 are typically associated with the gaseous compounds and those identified with poor purging efficiency on Table 4-1. Marginal failure for %RSD for these compounds shall not exceed 40%.

4.9.2.2.2 Method 8081. Several single-component pesticides may co-elute on certain GC columns. Therefore, it may be necessary to use two calibration mixtures to ensure sufficient separation for quantitation. Choose calibration mixes to minimize the peak overlap. Surrogates may be calibrated from either mix. For each multiple-component pesticide (e.g., toxaphene), analyze a mid-level standard to aid in pattern recognition. Based upon the positive identification of either compound in the samples, calibrate the instrument for that multi-component pesticide with a minimum of three standards and reanalyze the extract to enable accurate quantitation. Note that if technical Chlordane is required, a separate three-point calibration must be performed using technical Chlordane standards. Professional judgement should be employed in conjunction with the method instruction to determine the approach used to calculate the appropriate CF(s) (e.g., the use of total area or selection of a minimum of 4 to 6 characteristic peaks for toxaphene and 3 to 5 for chlordane). Calibration factors are then used to calculate the mean calibration factors, standard deviation, and relative standard deviation and apply the principles as stated in Section 4.9.2.2 for both single and multi-component pesticides and as summarized in the Table 4-10. Marginal failure for %RSD for poor performing compounds shall not exceed 40%.

4.9.2.2.3 Method 8082. ***Procedures for initial calibrations will vary based on the project requirements for PCB quantitation as noted in Section 4.6. (e.g., PCBs as Aroclors, PCB congeners, or total PCBs)***. When PCBs are to be determined as Aroclors, external standard calibration techniques should be used; when determined as PCB congeners, an internal standard calibration should be used. Table 4-11 summarizes appropriate QC limits.

- Aroclors. The approach taken for an initial calibration will differ depending on the project DQOs. For instance, projects, which have defined a few specific Aroclors associated with the site, recommend the following procedures. Perform the initial calibration using five standards for each Aroclor identified by the project. When samples contain a known mixture of different Aroclors, the analyst may perform a five-point calibration using that Aroclor mixture. When a multi-point calibration is performed for individual Aroclors, calculate and use the calibration factors from a minimum of 3 to 5 peaks for those standards and evaluate linearity as presented in Section 4.9.2.2. If the PCBs are unknown or the types of PCBs have not been determined, recommend the following procedures. Perform the initial calibration using five standards for a mixture of Aroclor 1016 and Aroclor 1260 standards in order to determine linearity of the detector response. For the remaining five Aroclors, a mid-level standard is analyzed to aid in pattern recognition. Based upon the positive identification of any PCBs in samples corresponding to the Aroclors with only the mid-level standard analyzed, calibrate the instrument for that PCB with a minimum of three standards and reanalyze the extract to enable accurate quantitation. Again, using a minimum of 3 to 5 peaks, calculate appropriate CFs for the 1016/1260 and any positively identified PCB standards and apply the principles as outlined in Section 4.9.2.2 to evaluate linearity.

- PCB Congeners. Table 4-3B identifies 19 congeners that have been successfully tested by the method. However, the procedure may be appropriate for additional congeners. When PCB congeners are to be determined, decachlorobiphenyl (DCB) is recommended for use as the internal standard. Perform a five-point initial calibration using standards containing all PCB congeners. Calculate the response

factor (RF) for each congener in the calibration standards, and evaluate the linearity of the initial calibration using principles as outlined in Section 4.9.2.2.

4.9.2.2.4 Method 8260. Apply the principles as stated in Section 4.9.2.2, in addition to the items presented below. Poor performers for Method 8260 are typically associated with the gaseous compounds and those identified with poor purging efficiency on Table 4-4. Marginal failure for %RSD for these compounds shall not exceed 30%. QC elements and acceptance limits are summarized in Table 4-12.

- Verify the mean Response Factors (RFs) for the SPCCs (system performance check compounds) satisfy the minimum RFs requirements specified in Method 8260. If these criteria are not met, evaluate the system (e.g., for standard mix degradation, injection port inlet contamination, contamination at the front end of the analytical column and active sites in the column or chromatographic system). Take corrective action and recalibrate for all target compounds.

- If the regression coefficient r > 0.995 or RSD < 30% for CCCs, this is indicative of system leak or column degradation. Take appropriate corrective action (e.g., instrument maintenance) and recalibrate for all target compounds and surrogates.

4.9.2.2.5 Method 8270. Apply the principles as stated in Section 4.9.2.2, in addition to the items presented below. Poor performers for Method 8270 are typically associated with the compounds, which exhibit poor chromatographic behavior. Marginal failure for %RSD for these compounds shall not exceed 40%. QC elements and acceptance limits are summarized in Table 4-13.

- Verify the mean Response Factors (RFs) for the SPCCs (system performance check compounds) satisfy the minimum RFs requirements specified in Method 8270. If these criteria are not met, evaluate the system (e.g., for standard mix degradation, injection port inlet contamination, contamination at the front end of the analytical column and active sites in the column or chromatographic system). Take corrective action and recalibrate for all target compounds.

- If the regression coefficient r > 0.995 or RSD < 30% for CCCs, this is indicative of system leak or column degradation. Take appropriate corrective action (e.g., instrument maintenance) and recalibrate for all target compounds and surrogates.

4.9.2.2.6 Method 8330. Perform the initial calibration as specified in Section 4.9.2.2 with the following points considered. Marginal failure for %RSD for these compounds shall not exceed 30%. QC elements and acceptance limits for Method 8330 are summarized in Table 4-14.

- Due to the lack of resolution between 2,4-DNT and 2,6-DNT, and between 2-Am-DNT and 4-Am-DNT, calibrations of these compounds may be based on 'isomeric pairs'. Improved resolution may be obtained using a Supelco C-18 column with an eluent of 55/45 (v/v) methanol/water at 0.8 mL/min.

- The C-18 column may be substituted with a C-8 column (as the primary column) if 2-NT and 4-NT are not target analytes or project-specific approval is obtained. (These two analytes generally coelute on C-8 columns.) Note that a C-8 column must not be used in place of the confirmatory CN-column.

4.9.3 Initial Calibration Verification. The initial calibration curve shall be verified as accurate with a standard purchased or prepared from an independent source. This initial calibration verification (ICV) involves the analysis of a standard containing all of the target analytes, typically in the middle of the calibration range, each time the initial calibration is performed. The % recovery of each target analyte in the ICV is determined from the initial calibration

and compared with the specifications for the CCV in each method (except for mercury by CVAA) as outlined in Tables 4-7 through 4-14.

Note for methods which report several (>5) target analytes, a small percentage of sporadic marginal failures may be tolerated (i.e., will not trigger re-extraction and analysis of the entire batch). This is subject to approval by the district chemist and based on the data quality objectives. The number of target analytes reported for the method will dictate the number of allowable QC failures as given below. Refer to the individual method tables for details on the implementation of this concept.

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

The marginal failure allowance entails the application of an expanded acceptance criterion. If these QC criteria are not met, a new initial calibration must be performed.

4.9.3.1 Method 8081. A separate ICV standard is required for each multiple-component target compound (e.g., toxaphene and chlordane), if a calibration is performed based upon its presence in samples.

4.9.3.2 Method 8082. The ICV standards may be limited to contain a mixture of Aroclors 1016 and 1260 or the project-specified Aroclors.

4.9.4 Initial Calibration Blanks (ICBs) and Continuing Calibration Blanks (CCBs)

ICBs and CCBs are required for inorganic metals analyses to verify the system is free of contamination. The frequency of ICB/CCB analyses is presented in Tables 4-7 and 4-8 as outlined within Methods 6010 and 7000. The concentrations of each target analyte in the ICB/CCB must be less than or equal to the MDL check sample (~ 2 times the MDL) as presented in Tables 4-7 and 4-8. Samples must not be analyzed until the ICB is acceptable, and all results must be bracketed by passing CCBs in order to be considered valid.

4.9.5 Continuing Calibration Verification (CCV)

CCVs are analyzed to determine whether the analytical system is working properly, and if a new initial calibration (and the reanalysis of sample extracts) is required. Calibration "verification" differs in concept and practice from "continuing calibration". In this latter technique, a standard is analyzed and new response factors are calculated, or a new calibration curve is drawn from the analysis of the continuing calibration standard. The former verifies compliance with the initial calibration curve, but does not overwrite the response factors used for the quantitation, nor allows re-sloping of the calibration curve. Calibration verification shall be used for all analytical methods, calculating a % Drift when the initial calibration is based on regression analysis, and a % Difference when the initial calibration is determined based upon % RSD values. Continuing calibration verification (CCV) typically involves the

analysis of a single primary source standard in the middle of the calibration range, between the concentrations of low-level and mid-level calibration standards. The frequencies of the CCV vary between methods, but are related to the type of detector used, and sample matrices analyzed. The analysis of more frequent CCVs is recommended for very sensitive detectors and when analyzing difficult matrices. This frequency is typically presented within SW-846 methods as (1) At the beginning of the analytical shift/sequence; (2) every 12 hours of analyses or every 10 to 20 samples; and may include (3) at the end of the analytical sequence. Refer to Section Tables 4-7 through 4-14 for details on requirements for CCV implementation and acceptance limits for the individual methods. If these QC criteria are not met, take corrective action to inspect the analytical system to determine the cause and perform instrument maintenance to correct the problem before analyzing a second CCV. If the second CCV is acceptable after system maintenance is performed, re-calibration is not required but all sample extracts analyzed after the last acceptable CCV must be reanalyzed. If however, the second CCV fails, a new initial calibration must be performed and all associated sample extracts reanalyzed. The CCVs do not have to be from the primary source standards.

4.9.5.1 Inorganic Analyses. A calibration verification pair of a CCB and CCV must be analyzed after every 10 samples (including batch QC samples) and at the end of the analytical sequence as outlined in Sections 4.9.4 and 4.9.5. Refer to Tables 4-7 through 4-8 or a summary of CCV implementation and QC requirements.

4.9.5.2 Organic Analyses. Calibration verification must be analyzed as outlined in Section 4.9.5, as summarized in Tables 4-9 through 4-14, in addition to the following:

- For certain organic analyses, additional CCVs at low- and high-level concentrations are recommended, due to the instability of their detectors (e.g., HECD, ECD). Method quality objectives (acceptance limits) for the high-level CCV should be in accordance with the mid-level CCV criteria. ***This criterion however, may not be achievable for the low-level CCV. Therefore, no method quality objectives for low-level CCV are included at this time, and should be identified within project documents based upon the data's use. For instance, if low-level detection is critical based on project action levels or decision levels, appropriate method quality objectives should be determined based on an acceptable level of error to support the data's use.***

- For methods that contain multi-component target compounds (e.g., PCBs), typically only a subset of these analytes would be used in the CCV.

- For GC/HPLC methods, concepts similar to that presented for initial calibrations apply. For the methods may possess limitations for certain target analytes which make the stated method acceptance criteria unattainable. Therefore, SW-846 has incorporated an allowance to evaluate the mean of the % Difference (%D) or %Drift values for all target analytes in the calibration verification standard that are less than the method acceptance criteria. To avoid the inclusion of target analytes showing gross method failure, this approach may be utilized as long as the target analytes do not exceed the criteria established for poor performers in the enclosed method-specific tables. ***In addition, the laboratory must communicate this information within the case narrative to the client. Provide a summary of all of the target analytes exceeding method acceptance criteria, the individual %D values for those compounds, and the mean %D calculated.***

- For GC/HPLC methods, compare the retention time of each analyte in the CCV with the absolute retention time windows established in Section 4.7.2. Each analyte must fall within its respective retention time window. If this criterion is not met, the chromatographic system must be adjusted to allow another CCV to meet the criterion, or a new initial calibration performed and new retention time windows established.

4.9.5.2.1 Method 8021. Due to the instability and potential drift of the electrolytic conductivity (HECD) detector, the following procedures are highly recommended. When analysis includes the halogenated volatile organic (HVO) target analytes, suggest alternating the mid-level CCV with high- and low-level CCVs as noted in Section 4.9.5.2.

4.9.5.2.2 Method 8081. Due to the instability and potential drift of the electron capture (ECD) detector, the following procedures are also highly recommended. Suggest alternating the mid-level CCV with high- and low-level CCVs as noted in Section 4.9.5.2, and also recommend incorporating periodic multi-component pesticide CCVs (i.e., toxaphene and chlordane), when applicable. Multi-component pesticide CCVs must be incorporated when identified as potential chemicals of concern (COPC).

4.9.5.2.3 Method 8082. When quantitating for PCBs as Aroclors, a mid-level CCV standard containing a mixture of Aroclors 1016 and 1260 (or Aroclors of interest) must be analyzed. When quantitating for individual PCB congeners, the CCV standard must contain all congener target compounds. Due to the instability and potential drift of the electron capture (ECD) detector, the following procedures are also highly recommended. Suggest alternating the mid-level CCV with high- and low-level CCVs as noted in Section 4.9.5.2.

4.9.5.2.4 Methods 8260 and 8270. Apply the principles as stated in Section 4.9.5.2, in addition to the items presented below. It is further recommended that a CCV be analyzed at the end of the analytical sequence.

- Evaluate the RFs of the SPCCs in the CCV. If the SPCCs do not satisfy the minimum response factor requirements specified by methods 8260/8270, take corrective action and re-inject the CCV. However, if CCV remains unacceptable, a new initial calibration must be performed.

- Evaluate the responses and retention times of the internal standards in the CCV as soon as possible. If the retention time for any internal standard changes by more than 30 seconds, or the EICP area changes by a factor of two (-50% to + 100%) from that of the mid-point standard of a current initial calibration, inspect the mass spectrometer for malfunctions and take corrective action. Reanalyze any affected samples if required.

- Evaluate the concentration of each target compounds and surrogate in the CCV. Verify the % Drift or % Difference for the CCCs (calibration check compounds) and all project-specified contaminants of concern are within ± 20% of its expected value. Evaluate remaining target compounds to assess instrument stability and survey the need for performing instrument maintenance.

4.10 General Laboratory Requirements

Per ER 1110-1-263, each laboratory performing work for the USACE shall comply with ISO/IEC Guide 25, General Requirements for the Competence of Calibration and Testing Laboratories, 1990 Edition and Updates. This may be accomplished by the application of the USACE laboratory validation as identified in ER 1110-1-263. Procedures for the laboratory validation process are described in EM 200-1-1. The following laboratory requirements are pursuant to meeting the standards established within the noted references. ***Individual project requirements may be more or less stringent than those listed below***. Having MRD validation does not preclude a laboratory or project from conducting project-specific audits. The QAPP shall specify the number and type of audits to be performed and specific certifications required for the project. The QAPP shall also provide an example audit checklist for review by the District Chemist. Laboratories performing non-routine analyses or analyses not validated by the USACE are required to meet minimum standards of quality and proficiency. These standards must be documented in the CDQMP.

4.10.1 Laboratory Quality System. A laboratory must establish, implement, and maintain a quality system appropriate for the type, range, and volume of analytical services it provides. The elements of this quality system shall be documented within a Laboratory Quality Management Plan or related documentation. Laboratory management is responsible for communicating the stated policies and practices to laboratory personnel, ensuring all information is clearly understood and implemented. The laboratory shall perform periodic audits of activities to verify compliance with the quality system. When deviations are discovered, the laboratory shall take immediate corrective action to remedy the situation or practice, notifying any client whose work may have been affected.

4.10.2 Laboratory Quality Management Plan. The laboratory shall prepare a written Quality Management Plan, which describes the general and specific procedures used within the laboratory to achieve scientifically valid and legally defensible data. ***This documentation requirement pertains exclusively to the laboratory, and is not considered equivalent to the Quality Assurance Project Plan (QAPP) which is an integral part of the project-related SAP.*** However, the laboratory may be required to submit this documentation as an appendix to the project-specific QAPP. ***When conflicting language exists between the project QAPP and the Laboratory Quality Management Plan, the project QAPP takes precedence over the LQMP.***

The Quality Management Plan should present the laboratory's policies, organization, objectives, functional guidelines, and specific QA and QC activities designed to achieve the data quality requirements when running performance-based methods, such as the SW-846 methods. Standard operating procedures pertaining to each element shall be included or referenced as part of this QA Management Plan and should describe the specific operational and analytical procedures as normally implemented by the laboratory. This plan should include, at a minimum, the following elements:

- QA policy, objectives, and commitments, any allowable departures from documented policies;

- Organization structure and personnel - include descriptions of key personnel, identify relationship between management, operations, support, and QA personnel;

- Facilities and equipment;

- Document control - notebook policy, sample tracking and custody procedures, LQMP and SOP organization and control;

- Scope of analytical methodologies provided - sample preparatory and determinative procedures available; Methods' implementation - calibration procedures and frequency, standards' preparation procedures, traceability of measurements and procedures employed, decision processes/procedures/responsibility for initiation of corrective action;

- Data generation - data collection procedures, data reduction procedures, data evaluation procedures, data reporting/authorization procedures;

- Quality control - solvent/reagent checks, reference material analysis, internal QC checks, retesting or corrective action implementation, verification of electronic data management systems;

- Quality assurance - Determination and monitoring of method QA performance, systems/internal audits, customer complaints' resolution, performance/external audits, interlaboratory comparisons and proficiency programs, corrective action procedures, and QA reporting procedures.

Submission of this Laboratory QA Management Plan for review, along with some or all of the standard operating procedures, may be required before sample testing can be initiated on any given project. These documents shall be amended should deficiencies be noted during review or whenever the fundamental elements described above are updated (i.e., annually).

4.10.3 Laboratory Organization, Management, and Analytical Personnel Responsibilities. The laboratory shall have sufficient personnel with appropriate education, current training, and experience to fulfill their assigned duties. The laboratory shall promote independence of judgement and integrity with well-defined responsibilities outlined for each individual within the laboratory organization. Personnel training records shall be maintained by the laboratory.

4.10.3.1 Laboratory Management. Laboratory management shall at a minimum have a technical director/manager responsible for overall technical operations. The technical director shall have a minimum of a Bachelor's degree in chemistry or any related scientific/engineering discipline, and a minimum of 2 years of laboratory experience. The laboratory management shall have sufficient authority and resources to fulfill their duties accordingly. Management staff shall be responsible for actively supporting the following at a minimum: (1) implementation of the policy and practices defined within the Laboratory Quality Management Plan, (2) maintaining accurate standard operating procedures and enforcing their use in the laboratory, (3) participation in interlaboratory comparisons and proficiency testing, (4) certifying that personnel performing all tests have proper education and training, (5) providing appropriate management and supervisory support to ensure adequate supervision of technical staff, (6) provide a contingency plan which identifies backup personnel for key laboratory positions (i.e., technical director/manager, QA officer/manager, etc.) in the event of personnel absence, (7) have policy and procedures in place which ensure protection of clients' confidential information and proprietary rights, and (8) maintaining a work environment that emphasizes the importance of data quality.

4.10.3.2 Laboratory Quality Assurance Officer. The laboratory shall at a minimum have a quality assurance (QA) officer/manager, responsible for the laboratory's quality system. The laboratory QA officer shall be responsible for maintaining the quality system and overseeing the quality assurance aspects of the data. The QA officer shall work independent of the laboratory's production management and have direct access to the highest level of management for decisions on laboratory policy and resources. In laboratories with limited staff (i.e., <10 technical personnel) the QA officer may also perform duties as the technical director or deputy technical director. QA officer shall at a minimum: (1) serve as a focal point for QA issues, (2) perform oversight and QA review for all nonconformance reports, (3) perform QA review for a percentage of laboratory analytical batches or project data packages, (4) evaluate data objectively, independent of laboratory management influence, (5) possess a general knowledge of the methods for which data review is performed, (6) conduct internal audits on the entire technical operation annually, and (7) monitor laboratory method performance by control charts/ranges evaluation, promoting method improvements as necessary. This individual should have a minimum of a Bachelor's degree in chemistry or any related scientific/engineering discipline and be familiar with all laboratory operations. A minimum of three years of laboratory experience, including at least one year of applied experience with quality assurance (QA) principles and practices in an analytical laboratory are required. In addition, a working knowledge of general statistical concepts is recommended to support data review and method performance monitoring responsibilities.

4.10.3.3 Organic Chemistry Section. If applicable, the laboratory shall maintain an Organic Chemistry Section with appropriate personnel, facilities, and instrumentation to conduct the work required. The following disciplines must be clearly represented and staffed as project testing dictates.

4.10.3.3.1 Organic Section Supervisor(s). The gas chromatograph/mass spectrometer (GC/MS), GC, or Sample Preparation Laboratory Supervisors are responsible for all technical efforts of their respective laboratories, providing sufficient oversight of activities to ensure data meet all terms and conditions expressed for the project. These individuals shall possess documentation which supports demonstration of performance for all areas which they

provide supervision. In addition, they should have a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline, and a minimum of three years of laboratory experience, including at least one year of supervisory experience.

4.10.3.3.2 GC/MS Analyst. Qualifications for these individuals should be at a minimum of one year of experience in operating and maintaining GC/MS/DS with a bachelor's degree in chemistry or in any related scientific/engineering discipline, or in lieu of the bachelor's degree, three years of experience in operating and maintaining the GC/MS and interpreting GC/MS data.

4.10.3.3.3 Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Analyst(s). Qualifications for these individuals should be at a minimum of one year of experience in operating and maintaining GC/HPLC equipment, respectively, with a bachelor's degree in chemistry or a related scientific/engineering discipline, or in lieu of the bachelor's degree, three years of experience in operating and maintaining the GC/HPLC and interpreting GC/HPLC data.

4.10.3.3.4 Extraction/Concentration Technician. Qualifications for these individuals should be at a minimum of a high school diploma and one year of college general chemistry. These individuals should also have a minimum of one year of experience in extraction/concentration.

4.10.3.4 Inorganic Chemistry Section. If applicable, the laboratory should maintain an Inorganic Chemistry Section with the appropriate personnel, facilities, and instrumentation to conduct the work required for the project. The following disciplines must be clearly represented and staffed as project testing dictates.

4.10.3.4.1 Inorganic Section Supervisor(s). The metals, wet chemistry, or sample preparation laboratory supervisor(s) is responsible for all technical efforts of their respective laboratories, providing sufficient oversight of activities to ensure data meet all terms and conditions for each project. These individuals shall possess documentation which supports demonstration of performance for all areas which they provide supervision. In addition, they should have a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline, and a minimum of three years of laboratory experience, including at least one year of supervisory experience.

4.10.3.4.2 ICP Analyst. Qualifications for these individuals should be at a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline with one year of experience in operating and maintaining ICP instrumentation, or, in lieu of the educational requirement, three additional years of experience in operating and maintaining ICP instrumentation.

4.10.3.4.3 Atomic Absorption (AA) Analyst. Qualifications of these individuals should be at a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline with one year of experience in operating and maintaining AA instrumentation for graphite furnace, flame, and cold vapor AA, or, in lieu of the educational requirement, three additional years of experience in operating and maintaining AA instrumentation, including graphite furnace, flame, and cold vapor techniques.

4.10.3.4.4 Inorganic Sample Preparation Technician. Qualifications for these individuals should be at a minimum of a high school diploma and a college level course in general chemistry or equivalent. These individuals should also have a minimum of one year of experience in sample preparation in an analytical laboratory.

4.10.3.5 Wet Chemistry Analyst. If applicable, qualifications of these individuals should be at a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline. These individuals should also have a

minimum of one year of experience with classical chemistry laboratory procedures, in conjunction with the education qualifications, or, in lieu of the educational requirement, 2 years of additional equivalent experience.

4.10.3.6 Radiochemical Techniques Analyst. Qualifications of these individuals should be at a minimum of a bachelor's degree in chemistry or any related scientific/engineering discipline with one year of experience in performing radiochemical analyses, or, in lieu of the educational requirement, three additional years of experience in operating and maintaining radiochemical instrumentation.

4.10.3.7 Technical Staff Backup. The laboratory should have a minimum of one chemist available at any time as a backup technical person for each analytical area to ensure continuous operations and accomplish the work required. These individuals should have similar education and experience requirements to the primary analyst.

4.10.3.8 Sample Custodian and Data Management. The laboratory should also maintain and staff support positions for Sample Custodian and Data Management personnel. Qualifications for these individuals should be at a minimum of a high school diploma, and appropriate on-the-job training.

4.10.4 Laboratory Facility and Equipment

4.10.4.1 Laboratory Facility Requirements. The laboratory shall provide a secure testing facility which can accommodate the proper performance for the type, range, and volume of analytical services it provides. Facility entries must be controlled, and monitored as necessary to assure restricted access is maintained, especially for areas affecting the quality of activities or data. The design must provide effective separation of incompatible testing activities; and adequate energy sources, lighting, heating/cooling and ventilation to ensure stability of voltage, temperature, humidity, or other pertinent environmental conditions. This may involve inclusion of an area under positive pressure for VOC analysis. Adequate monitoring of environmental conditions and general housekeeping should be maintained to avoid any influence on the testing activities performed.

4.10.4.2 Laboratory Equipment Requirements. The laboratory shall provide sufficient equipment, instruments, and related supplies for proper performance of work. All equipment used shall be reflective of the measurement accuracy necessary. The laboratory shall ensure that all equipment and supplies purchased are inspected, a unique identifier assigned to it and the equipment verified as compliant with all relevant requirements prior to their initial use. Records of all suppliers used to obtain support services and materials shall be maintained.

4.10.4.2.1 Equipment Preventive Maintenance. To minimize downtime and interruption of analytical work, preventive maintenance shall be routinely performed on each analytical instrument. Designated laboratory personnel should be trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, the equipment shall be taken out of service, repairs performed by either trained staff or trained service engineers, and an evaluation of the impact on previous calibrations or tests performed. It is generally recommended that maintenance contracts be maintained on all major analytical instruments. Detailed SOPs shall be on file or the information incorporated into method SOPs/Laboratory Quality Management Plan that describe preventive maintenance procedures and schedules. The laboratory shall maintain detailed logs for each instrument documenting the preventive maintenance and repairs performed.

4.10.4.2.2 Equipment Backup Capabilities. Backup instruments shall be designated in case of an extended breakdown for an analytical instrument. It is the laboratory's responsibility to have a backup plan in force such that all sample holding times can be met. This plan can include rental of backup instruments, or the use of another USACE validated laboratory for a given procedure. All equipment outside of the laboratory's permanent control shall be evaluated to ensure that all relevant requirements are met prior to their initial use. ***Before any subcontracting is performed, USACE must be informed and approval given, in writing.*** The laboratory shall

ensure, and be able to provide documentation, that all subcontractors employed are competent to perform the duties requested, and comply with all of the requirements established within this guidance and EM 200-1-1, as appropriate.

4.10.4.2.3 Laboratory Equipment Records. The laboratory shall maintain appropriate records or documentation for all instruments and support equipment to identify: (1) type of equipment, (2) manufacturers' name or equipment make, model, and any serial numbers or unique identifiers, (3) dates received and placed into service, (4) condition when purchased (new, used, etc.), (5) current location, (6) manufacturer instructions/manuals, (7) history of any damage, modification or repair, (8) instrument maintenance logs, and (9) calibration/calibration verification run logs.

4.10.5 Laboratory SOPs. Laboratories shall be required to maintain written, approved laboratory-specific standard operating procedures (SOPs) for all methods and general operations. Laboratory-specific SOPs that fully detail the actual procedures and documentation used to implement performance-based methods. Simply referencing a given method or method number is not sufficient. Overall, these SOPs should be based on the guidance as published by EPA (QA/G-6 Guidance for the preparation of Standard Operating Procedures (SOPs) for Quality -Related Documents, November 1995).

The SOP shall be a written narrative, stepwise description of laboratory operating procedures. The SOPs shall accurately describe the equipment, and actual procedures used in the laboratory. Copies of the SOPs shall be readily available to the appropriate laboratory personnel. Calculations that are performed external to an instrument or in its automation software shall be documented in the SOP. The SOP should also identify an appropriate estimation of uncertainty for all measurements by the designation of appropriate class/grade of equipment within the SOP, or by the number of significant figures recorded based upon the accuracy of the equipment used. The format for SOPs may vary depending upon the kind of activity for which they are prepared, however, at a minimum, the following sections shall be included: Title/Signature/Effective Date page; Scope and Application; Method Summary; Sample Preservation, Containers, Handling, and Storage; Interferences and Potential Problems; Equipment and Apparatus; Reagents and Solutions; Procedures; Calculations; Quality Assurance/Quality Control; Corrective Actions, Data Evaluation; MDL studies/Sensitivity Assessment; Health and Safety; Sample Disposal; References; and Example Forms. Laboratory SOPs shall be given unique ID numbers. These SOPs shall be controlled documents which are reviewed annually, or updated as necessary whenever procedure/method changes are made and a new version number assigned. Retired SOPs shall be maintained on file by the laboratory in case data quality questions arise later.

4.10.6 Document Control Procedures. The laboratory shall maintain records documenting all phases of sample handling from sample receipt to final analysis. Accountable documents used by laboratories include, but are not limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, instrument printout, and other documents relating to the sample or sample analysis. The laboratory shall use a document numbering and identification system for all documents/logs. All observations and results recorded by the laboratory shall be recorded on either preprinted laboratory forms, permanently bound laboratory logbooks, or entered into secure computer systems. Recommend observations include noting basis for any manual integrations performed. Pages in both the bound and unbound logbooks shall be sequentially numbered. Preprinted laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person(s) performing the activity at the time the activity was performed. Permanently bound laboratory logbooks shall be dated and signed by the person performing the activity at the time the activity was performed. All logbook entries shall be in chronological order. All entries shall be recorded in indelible ink. Unused portions of the logbooks shall be "z'd" out. Corrections to logbooks shall be made by drawing a single line through the error and entering the correct information. Corrections and additions shall be dated and initialed. Computer forms shall contain the name of the laboratory and be dated and signed by the person performing the activity at the time the form is printed. Computer systems must be established

to maintain the integrity of the data, i.e., verified to ensure accurate capture, processing, manipulation, recording, and reporting of data, configured to restrict access and provide for appropriate backups and audit trails, etc.

4.10.6.1 Standard Preparation Log. Standard preparation logs should document the preparation of all calibration standards and spiking standards associated with the respective analysis (e.g., the initial calibration, CCV, and ICV standards as well as the MS, LCS, surrogate, and PDS spiking standards). The laboratory shall maintain complete internal documentation for all standards and reagents used that allows traceability back to the original source. At a minimum, the standard preparation logs must clearly specify the following for all standards:

- Sources (e.g., manufacturer and lot number for commercial stock solutions),
- Composition (e.g., initial and final concentration of all target analytes, type and purity of standards)
- Preparation and expiration dates
- Unique ID number of the standard
- Reagents and solvents added to standards (including source and lot numbers)
- Name of preparer

When a standard is prepared via the dilution of a stock solution, the spiking volume and concentration of the stock solution, and the final volume and concentration of the diluted standard should be specified and documented accordingly. Manufacturer certificates for commercially purchased stock standards must be maintained. When the laboratory prepares its own stock solutions, calculations and conversion factors should be shown in the standard preparation log (e.g., a general formula or sample calculations).

4.10.6.2 Sample Preparation Log. Sample preparation logs should document all significant sample preparation activities. All reagents/standards used shall be clearly identified (e.g., with lot numbers) on the appropriate laboratory bench log sheets. The sample preparation logs must include the following information:

- Sample and batch ID numbers
- Matrix
- Preparatory method (method or laboratory SOP ID number)
- Date of sample preparation
- Initial volume or weight of the sample processed
- Final volume of the sample processed (after digestion, extraction or cleanup)
- Percent moisture (for solid samples)
- Reagents and solvents added to the samples (including source and lot numbers)

- Any pH and preservation checks and adjustments performed
- Spiking standards (ID number of the LCS, and MS spiking solutions, volume added, and the final spike concentration)
- Name of analyst

4.10.6.3 Instrument Run Log. Instrument run logs shall be maintained for each instrument to enable a complete reconstruction of the analytical run sequence. Run sequence logs must indicate the unique identifier appropriated for the instrument used to generate the data, the date of analysis and the aliquot volume of the sample analyzed (e.g., the injection volume for chromatographic methods). The time of analysis must be specified for chromatographic methods. The order in which field and QC samples are collected and presented should be consistent with the temporal order in which the analyses were performed. Run logs must clearly indicate which field and batch QC samples are associated with each initial calibration, ICV, and CCV. Instrumental analysis logs are particularly important since they provide the basic link between the sample analyses and QC data. Computer logs may be used if all of the preceding information is captured.

4.10.6.4 Computer/Instrument Outputs. Computer/instrument printouts or other independent information can be incorporated into logbooks if such printouts can be permanently affixed to the appropriate logbook.

4.10.6.5 Electronic Data Management. Electronic data management systems shall be verified by the laboratory to ensure accurate data transfer, data reduction, and reporting. All aspects of the data management system shall be fully documented as compliant with USEPA Good Automated Laboratory Practices (GALP) requirements.

4.10.7 Laboratory Quality Assurance Procedures. The laboratory shall ensure the quality of results by maintaining an integrated quality assurance system of activities involving the planning, implementation, assessment, reporting, and quality improvement of data. Refer to ISO/IEC Guide 25, General Requirements for the Competence of Calibration and Testing Laboratories and ANSI/ASQC E4, Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs for additional information. These activities are typically performed or facilitated by the Laboratory QA officer and include the (1) performance of periodic audits (system and technical); (2) participation in proficiency testing programs/interlaboratory comparisons, (3) routine analysis of certified reference materials or second source reference materials, and (4) monitor method performance (sensitivity, precision and bias) through an evaluation of the MDL study or MDL check sample, and batch QC sample (MB, LCS) control ranges/charts.

4.10.7.1 Laboratory Audits. Annual laboratory audits shall be conducted internally for each analytical area to verify the following at a minimum, (1) procedures are compliant with SOPs, (2) documentation practices are complete and traceable to a certified source(s), (3) data reviews are complete, well-documented, and effective, and (4) data reporting practices, including electronic or manual data transfer and client report generation are accurate and complete. All audit findings, any corrective actions, root cause determination, etc. shall be fully documented in QA reports to management. The QA officer shall document that all corrective actions necessary are verified complete within a reasonable time frame. Audits performed by external agencies or accrediting authorities shall not substitute for internally conducted laboratory audits.

4.10.7.2 Laboratory Method Performance Monitoring Using LCS. The laboratory shall generate in-house warning (2-sigma) and control (3-sigma) limits for all target analytes from LCSs. The LCSs are prepared from an interference-free aqueous and solid matrices in order to evaluate the quality of the method performance. These 'mean' control limits/charts are generated from bias measurements (e.g., LCS recoveries) to assess the method performance and data quality over an extended period of time. The 'warning' and 'control' limits for mean control

charts set at '2-sigma' and '3-sigma' approximate the 95% and 99% confidence intervals, respectively. A minimum of thirty points should be used to establish these control ranges or charts. In addition, data from all analyses (including method failures) should be used to generate the limits, so as not to diminish the ranges by biasing the data input. Outliers may be excluded from the data if proper QA procedures are employed such as using appropriate statistical tests (e.g., Dixon's Extreme Value test, Discordance test). It would not be necessary to maintain graphical control charts for all target analytes. Recommend a representative subset of target analytes for each method be chosen for control chart generation to observe method trends. These control ranges should be updated every six months, and reviewed by the QA officer annually at a minimum. Additionally, 'range' control charts may be used to evaluate precision between interbatch LCSs. Range control charts set the 95% and 99% confidence intervals at '2.456-sigma' and '3.268-sigma' for the 'warning' and 'control' limits, respectively. ***Because so many laboratories mistakenly apply the 2-sigma and 3-sigma factors to calculate precision control limits in lieu of the correct factors noted above, caution should be exercised when comparing control limits between different laboratories.***

Evaluate laboratory control limits against the method quality objectives presented in the project DQOs, the published reference method, or this guidance to survey the need for method evaluation, or modifications. Note the baseline method quality objectives summarized in Tables 4-7 through 4-14 are intended for evaluation of batch control acceptance and may not be reflective of a laboratory overall performance as depicted by their internal control limits. Evaluate the calculated mean for a general assessment of the method systematic bias, and review of representative control charts for evidence of analytical trends. Information gathered should be used to troubleshoot analytical problems associated with method implementation, offering suggestions for quality improvements and corrective action to tighten limits.

4.11 Laboratory Sample Handling Requirements.

4.11.1 Sample Receipt. The receiving laboratory's chain-of-custody, sample storage, and dispersement for analysis shall be documented per specific laboratory standard operating procedures (SOPs) and project requirements. ***Information on project custody, analysis, and data reporting requirements as noted in the SAP and highlighted on the Laboratory Notification Information Checklist (LNC) or similar, should be received by the laboratory prior to (or accompanying as with the LNC) the first shipment of incoming samples.*** Individual 'Cooler Receipt Forms' or similar, shall be used by the laboratory for each cooler to verify sample condition, including proper sample containers, volumes, preservation, etc. and document any problems noted. Corrective action will be required for any deficiencies identified. Refer to Chapter 3 (figures 3-4 and 3-3) of EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans for examples of the Laboratory Notification Sheet, and Cooler Receipt Form. ***It is required that all coolers contain at least one temperature blank.*** The temperature blank should be a 40-mL VOA vial filled with water and placed in a representative position inside the cooler. Multiple vials could be used, if needed. The laboratory should document when the temperature blank was positioned inappropriately or was not representative of the cooler temperature measurement. Sample login procedures shall follow the noted Cooler Receipt Form. The chain-of-custody form, any shipping documents, completed cooler receipt forms, telephone conversation record forms, and any corrective action forms will be maintained by the laboratory for each shipment and included in the reporting package when the results are submitted.

4.11.2 Sample Storage. The laboratory shall provide an adequate, contamination-free, and well-ventilated workspace for the receipt of samples. All samples and their associated extracts shall be stored under conditions that will ensure their integrity and preservation and are demonstrated to be free from all potential contaminants. Sufficient refrigerator space shall be provided for the proper storage of all samples and their associated extracts. Samples shall not be stored with standards. Samples designated for volatile organic analyses testing shall be segregated from other samples while samples suspected to contain high levels of volatile organic analyses (e.g., UST soil samples) should be further isolated from other volatile organic analyses samples. ***In the absence of project-specific criterion, samples and their associated extracts shall be stored under proper conditions for a minimum of***

sixty (60) days after receipt of the final data report for those samples. After that time, the laboratory is responsible for the disposal of the samples and their associated extracts in compliance with all federal, state, and local regulations unless arrangements have been made for the return of any unused sample portions back to the site.

4.11.3 Sample Security and Tracking. The laboratory shall maintain the integrity of the samples received, their associated extracts, and the data generated. Limited and controlled access to all laboratory areas shall be maintained. ***If required by the project, the laboratory should maintain sample and extract chain-of-custody within the laboratory at all times through the use of appropriate documentation and forms, otherwise strict internal chain-of-custody would not be required.***

4.11.4 Sample Holding Times. Extraction/digestion holding times shall be defined from the date/time of sample collection in the field to the date/time when the sample is first exposed to the extraction/digestion solvent. Analysis holding times shall be defined from the date/time of sample extraction to the date/time of sample analysis. It is required that laboratories maintain documentation that clearly show the dates (and times when applicable) for all sample handling/manipulation processes. Samples should be analyzed as soon as possible after sample collection. Published holding times are generally considered maximum times that samples may be held before analysis and still be considered compliant with method guidelines. Sufficient time should be allowed for the re-preparation or reanalysis of samples within holding times should calibration, method, or quality control failures occur. For meeting holding times, sample extraction is considered complete when the method analytes have been removed from the sample matrix.

TABLE 4-1
TARGET COMPOUND LIST FOR METHOD 8021 VOLATILE ORGANIC COMPOUNDS

Target Compound	CAS Registry No.
Benzene [2, 3]	71-43-2
Bromobenzene [1]	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane [1]	75-27-4
Bromoform [1]	75-25-2
Bromomethane [1, 5]	74-83-9
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon tetrachloride [1]	56-23-5
Chlorobenzene [1, 2]	108-90-7
Chloroethane [1, 5]	75-00-3
Chloroform [1]	67-66-3
Chloromethane [1, 5]	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Dibromochloromethane [1]	124-48-1
1,2-Dibromo-3-chloropropane [4]	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane [1]	74-95-3
1,2-Dichlorobenzene [1, 2]	95-50-1
1,3-Dichlorobenzene [1, 2]	541-73-1
1,4-Dichlorobenzene [1, 2]	106-46-7
Dichlorodifluoromethane [1, 5]	75-71-8

Target Compound	**CAS Registry No.**
1,1-Dichloroethane [1]	75-34-3
1,2-Dichloroethane [1]	107-06-2
1,1-Dichloroethene [1]	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene [1]	156-60-5
1,2-Dichloropropane [1]	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene [1]	10061-01-5
trans-1,3-Dichloropropene [1]	10061-02-6
Ethyl Benzene [2, 3]	100-41-4
Hexachlorobutadiene	87-68-3
Isopropylbenzene (Cumene)	98-82-8
p-Isoproplytoluene (p-Cumene)	99-87-6
Methylene chloride [1]	75-09-2
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane [1]	630-20-6
1,1,2,2-Tetrachloroethane [1]	79-34-5
Tetrachloroethene [1]	127-18-4
Toluene [2, 3]	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane [1]	71-55-6

Target Compound	**CAS Registry No.**
1,1,2-Trichloroethane [1]	79-00-5
Trichloroethene (trichloroethylene) [1]	79-01-6
Trichlorofluoromethane [1, 5]	75-69-4
1,2,3-Trichloropropane [1]	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl chloride [1, 5]	75-01-4
o-Xylene [2, 3]	95-47-6
m-Xylene [2, 3]	108-38-3
p-Xylene [2, 3]	106-42-3

[1] Halogenated Volatile Organic (HVO) target compounds
[2] Aromatic Volatile Organic (AVO) target compounds
[3] BTEX target compound list.
[4] Exhibits poor purging efficiency or instrumental response
[5] Gaseous target compound

TABLE 4-2
TARGET COMPOUND LIST FOR METHOD 8081 ORGANOCHLORINE PESTICIDES

Target Compound	**CAS Registry No.**
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Gamma-BHC (Lindane)	58-89-9
Delta-BHC	319-86-8
Alpha-Chlordane	5103-71-9
Gamma-Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2

TABLE 4-3A
TARGET COMPOUND LIST FOR METHOD 8082 PCBS AS AROCLORS

Target Compound	**CAS Registry No.**
Aroclor-1016	12674-11-2
Aroclor-1221	11104-28-2
Aroclor-1232	11141-16-5
Aroclor-1242	53469-21-9
Aroclor-1248	12672-29-6
Aroclor-1254	11097-69-1
Aroclor-1260	11096-82-5

TABLE 4-3B
TARGET COMPOUND LIST FOR METHOD 8082 PCB CONGENERS

Target Compound	CAS Registry No.
2-Chlorobiphenyl	2051-60-7
2,3-Dichlorobiphenyl	16605-91-7
2,2',5-Trichlorobiphenyl	37680-65-2
2,4',5-Trichlorobiphenyl	16606-02-3
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6
2,2',3,4,4',5, 5'-Heptachlorobiphenyl	35065-29-3
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9

TABLE 4-4
TARGET COMPOUND LIST FOR METHOD 8260 VOLATILE ORGANIC COMPOUNDS

Target Compound	**CAS Registry No.**
Acetone [1]	67-64-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane [1]	74-83-9
2-Butanone (methyl ethyl ketone) [1]	78-93-3
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon disulfide [1]	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane [1]	75-00-3
Chloroform	67-66-3
Chloromethane [1]	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Dibromochloromethane	124-48-1
1,2-Dibromo-3-chloropropane [1]	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1

Target Compound	**CAS Registry No.**
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dichlorodifluoromethane [1]	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Ethyl Benzene	100-41-4
Hexachlorobutadiene	87-68-3
2-Hexanone [1]	591-78-6
Iodomethane	74-88-4
Isoproplybenzene (Cumene)	98-82-8
p-Isoproplytoluene (p-Cumene)	99-87-6
Methylene chloride	75-09-2
4-Methyl-2-pentanone [1]	108-10-1
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6

Target Compound	**CAS Registry No.**
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene (trichloroethylene)	79-01-6
Trichlorofluoromethane [1]	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl chloride [1,2]	75-01-4
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3

[1] Denotes poor purging efficiency or poor response
[2] Gaseous target compound

TABLE 4-5A
TARGET COMPOUND LIST FOR METHOD 8270 FOR BASE/NEUTRAL FRACTION COMPOUNDS

Target Compound	CAS Registry No.
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Acetophenone	98-86-2
Aniline [1]	62-53-3
Anthracene	120-12-7
Benzidine [1]	92-87-5
Benzo(a)anthracene	56-55-3
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(g,h,i)perylene	191-24-2
Benzo(a)pyrene	50-32-8
Benzyl alcohol [1]	100-51-6
4-Bromophenyl phenyl ether	101-55-3
Butyl benzyl phthalate	85-68-7
4-Chloroaniline [1]	106-47-8
bis(2-Chloroethoxy)methane	111-91-1
bis(2-Chloroethyl) ether	111-44-4
bis(2-Chloroisopropyl) ether	108-60-1
2-Chloronaphthalene	91-58-7
4-Chlorophenyl phenyl ether	7005-72-3
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Dibenzofuran	132-64-9
Di-n-butyl phthalate	84-74-2

Target Compound	**CAS Registry No.**
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
3,3'-Dichlorobenzidine	91-94-1
Diethyl phthalate [1]	84-66-2
Dimethyl phthalate	131-11-3
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
Di-n-octyl phthalate	117-84-0
Diphenyl amine	122-39-4
1,2-Diphenylhydrazine	122-66-7
bis(2-Ethylhexyl) phthalate	117-81-7
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene [1]	77-47-4
Hexachloroethane	67-72-1
Hexachloropropene	1888-71-7
Indeno(1,2,3-cd)pyrene	193-39-5
Isophorone	78-59-1
2-Methylnaphthalene	91-57-6
Naphthalene	91-20-3
2-Naphthylamine	91-59-8
2-Nitroaniline [1]	88-74-4
3-Nitroaniline [1]	99-09-2

Target Compound	**CAS Registry No.**
4-Nitroaniline [1]	100-01-6
Nitrobenzene	98-95-3
N-Nitroso-dimethylamine [1]	62-75-9
N-Nitrosodiphenylamine [1],[2]	86-30-6
N-Nitroso-di-n-propylamine	621-64-7
N-Nitrosopyrrolidine	930-55-2
Phenanthrene	85-01-8
Pyrene	129-00-0
Pyridine	110-86-1
1,2,4,5-tetrachlorobenzene	95-94-3
1,2,4-Trichlorobenzene	120-82-1

[1] Denotes poor extraction efficiency, tendency to decompose, or poor chromatographic behavior
[2] N-Nitrosodiphenylamine co-elutes with, and cannot be differentiated from diphenylamine

TABLE 4-5B
TARGET COMPOUND LIST FOR METHOD 8270 FOR ACID FRACTION COMPOUNDS

Target Compound	**CAS Registry No.**
Benzoic Acid [1]	65-85-0
4-Chloro-3-methylphenol [1]	59-50-7
2-Chlorophenol	95-57-8
2,4-Dichlorophenol	120-83-2
2,6-Dichlorophenol	87-65-0
2,4-Dimethylphenol [1]	105-67-9
4,6-Dinitro-2-methylphenol [1]	534-52-1
2,4-Dinitrophenol [1]	51-28-5
2-Methylphenol [1] (o-cresol)	95-48-7
3-Methylphenol [1,2] (m-cresol) & 4-Methylphenol [1,2] (p-cresol)	108-39-4 & 106-44-5
2-Nitrophenol [1]	88-75-5
4-Nitrophenol [1]	100-02-7
Pentachlorophenol [1]	87-86-5
Phenol [1]	108-95-2
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2

[1] Denotes poor extraction efficiency, tendency to decompose, or poor chromatographic behavior
[2] 3-Methylphenol (m-cresol) co-elutes with 4-Methylphenol (p-cresol). Therefore, both are reported as isomeric pairs.

TABLE 4-6
TARGET COMPOUND LIST FOR METHOD 8330 EXPLOSIVES

Target Compound	**CAS Registry No.**
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4
1,3-Dinitrobenzene (1,3-DNB)	99-65-0
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8
Nitrobenzene (NB)	98-95-3
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	1946-51-0
2-Amino-4,6-dinitrotoluene (2-Am-DNT)	355-72-78-2
2,4-Dinitrotoluene (2,4-DNT)	121-14-2
2,6-Dinitrotoluene (2,6-DNT)	606-20-2
2-Nitrotoluene (2-NT)	88-72-2
3-Nitrotoluene (3-NT)	99-08-1
4-Nitrotoluene (4-NT)	99-99-0

TABLE 4-7
SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 6010
ICP METALS

Quality Control Element	**Description of Element**	**Frequency of Implementation**	**Acceptance Criteria**
Initial Calibration (4.9.2.1.1)	Option 1- 1 std and blank, and a low-level check standard at PQL Option 2- 3 stds and blank	Daily	Option 1- Low-level check standard ± 20% Option 2- r > 0.995
Instrumental Precision (4.9.2.1.1)	%RSD 3 integrations (exposures)	Each calibration and calibration verification standards (ICV/CCV)	%RSD < 5%
Initial Calibration Verification (ICV) (4.9.3)	Mid-level (2nd source) verification	After initial calibration	%Recovery ± 10%
Initial Calibration Blank (ICB) (4.9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL Check Sample (~2X MDL)
Interelement Check Standards (ICS) (4.8.1)	ICS-A - interferents only ICS-B - interferents and target analytes	Beginning of analytical sequence	%Recovery ± 20% for target analytes
Continuing Calibration Blank (CCB) (4.9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL Check Sample (~2X MDL)
Continuing Calibration Verification (CCV) (4.9.5 / 4.9.5.1)	Mid-level verification	Every 10 samples and at end of analytical sequence	%Recovery ± 10%
Method Blank (MB) (5.2.1.7.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < MDL Check Sample (~2X MDL)
Laboratory Control Sample (LCS) (5.2.1.7.4.2)	Interference-free matrix containing all target analytes	1 per sample batch	%Rec = 80% - 120%
Matrix Spike (MS)	Sample matrix spiked	1 per sample batch	%Rec = 75% - 125%

Quality Control Element	**Description of Element**	**Frequency of Implementation**	**Acceptance Criteria**
(5.2.1.7.4.3)	with all/subset of target analytes prior to digestion		
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (5.2.1.7.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD < 25%
Post Digestion Spike (PDS) (5.2.1.7.4.7.1)	Sample digestate spiked with all/subset of target analytes	As needed to confirm matrix effects	%Rec = 75% - 125%
Serial Dilution (SD) (5.2.1.7.4.7.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results ± 10%
Method of Standard Addition (MSA) (5.2.4.1.6.4.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	r > 0.995

[1] The number of Sporadic Marginal Failure (SMF) allowances depend upon the number of target analytes reported from the analysis. For instance, if between seven (7) to fifteen (15) metals are reported from the ICP analysis, one (1) SMF is allowed to the expanded criteria presented. If greater than 15 metals are reported from the ICP analysis, two (2) SMFs are allowed. Refer to Section 9.3 for additional information on the application of sporadic marginal failures.

TABLE 4-8
SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 7000 SERIES
GFAA/CVAA METALS

Quality Control Element	**Description of Element**	**Frequency of Implementation**	**Acceptance Criteria**
Initial Calibration (4.9.2.1.2)	3 stds and blank	Daily	r > 0.995
Instrumental Precision (4.9.2.1.2)	RPD of 2 injections	All standards, and ICV/CCV	RPD ± 10%
Initial Calibration Verification (ICV) (4.9.3)	Mid-level (2nd source) verification	After initial calibration	%Rec ± 10%
Initial Calibration Blank (ICB) (4.9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL Check Sample (~2X MDL)
Continuing Calibration Blank (CCB) (4.9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL Check Sample (~2X MDL)
Continuing Calibration Verification (CCV) (4.9.5 / 4.9.5.1)	Mid-level verification	Every 10 samples and at end of analytical sequence	%Rec ± 20%
Method Blank (MB) (5.2.1.7.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < MDL Check Sample (~2X MDL)
Laboratory Control Sample (LCS) (5.2.1.7.4.2)	Interference-free matrix containing target analytes	1 per sample batch	%Rec = 80% - 120%
Matrix Spike (MS) (5.2.1.7.4.3)	Sample matrix spiked with target analytes prior to digestion	1 per sample batch	%Rec = 80% - 120%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (5.2.1.7.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD < 20%
Post Digestion Spike (PDS) (5.2.1.7.4.7.1)	Sample digestate spiked with target analytes	As needed to confirm matrix effects	%Rec = 85% - 115%

Quality Control Element	**Description of Element**	**Frequency of Implementation**	**Acceptance Criteria**
Serial Dilution (SD) (5.2.1.7.4.7.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results ± 10%
Method of Standard Addition (MSA) (5.2.4.1.6.4.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	$r > 0.995$

TABLE 4-9
SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8021
VOLATILE ORGANIC COMPOUNDS

QC Element	Target Compound / Surrogate Acceptance Criteria
Initial Calibration (4.9.2.2.1)	Primary Evaluation: r = 0.995, %RSD < 20%, r^2 <0.990 Alternative Evaluation: Mean %RSD for all target Compounds < 20% Maximum allowable %RSD for each target compounds < 40%
ICV (4.9.3)	%Rec = 85% - 115%
CCV (4.9.5 / 4.9.5.2 / 4.9.5.2.1)	Primary Evaluation: %Drift < 15%, %D < 15% Alternative Evaluation: Mean %Drift/%D for all target analytes < 15% Maximum allowable %Drift/%D for each target compounds < 30%
MB (5.2.1.7.4.1)	Target Compounds: Compound < MDL Check Sample (~2X MDL) or ½ PQL Common Lab Contaminant Compounds < PQLs
LCS (5.2.1.7.4.2)	Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125%
MS (5.2.1.7.4.3)	%Rec = 70% - 130%
MSD/MD (5.2.1.7.4.4)	Water: RPD < 30% Solids: RPD < 40%
Surrogates (5.2.1.7.4.5)	LCS: Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125% Project Sample Matrix: %Rec = 70% - 130%
Target Compound Confirmation (5.2.3.4)	RPD < 40%

TABLE 4-10
SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8081
ORGANOCHLORINE PESTICIDES

QC Element	Target Compound/Surrogate
DDT/Endrin %Breakdown (4.8.2)	DDT & Endrin %Breakdown < 15% each
Initial Calibration (4.9.2.2.2)	Primary Evaluation: r = 0.995, %RSD < 20%, r^2 <0.990 Alternative Evaluation: Mean %RSD for all target Compound < 20% Maximum allowable %RSD for each target compound < 40%
ICV (4.9.3 / 4.9.3.1)	%Rec = 85% - 115%
CCV (4.9.5 / 4.9.5.2 / 4.9.5.2.2)	Primary Evaluation: %Drift < 15%, %D < 15% Alternative Evaluation: Mean %Drift/%D for all target compound < 15% Maximum allowable %Drift/%D for each target compound < 30%
MB (5.2.1.7.4.1)	Compounds < MDL Check Sample (~2X MDL) or ½ PQL
LCS (5.2.1.7.4.2)	Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130%
MS (5.2.1.7.4.3)	%Rec = 40% - 140%
MSD/MD (5.2.1.4.4)	RPD < 35%
Surrogates (5.2.1.4.5)	LCS: Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130% Project Sample Matrix: %Rec = 40% - 140%
Target Compound Confirmation (5.2.3.4)	RPD < 40%

TABLE 4-11
SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8082
PCBS

QC Element	Target Compound/Surrogate
Initial Calibration (4.9.2.2.3)	r > 0.995, %RSD < 20%, r^2 >0.990
ICV (4.9.3 / 9.3.2)	%Rec = 85% - 115%
CCV (9.5 / 9.5.2)	%Drift < 15%, %D < 15%
MB (5.2.1.7.4.1)	Analytes < MDL Check Sample (~2X MDL) or ½ PQL
LCS (5.2.1.7.4.2)	Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130%
MS (5.2.1.7.4.3)	%Rec = 40% - 140%
MSD/MD (5.2.1.7.4.4)	RPD = 35%
Surrogates (5.2.1.7.4.5)	LCS: Water: %Rec = 50% - 130% Solids: %Rec = 50% - 130% Project Sample Matrix: %Rec = 40% - 140%
Target Analyte Confirmation (5.2.3.4)	RPD < 40%

TABLE 4-12
SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8260
VOLATILE ORGANIC COMPOUNDS

QC Element	Target Compound / Surrogate
Initial Calibration (4.9.2.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %RSD < 30% Primary Evaluation: r = 0.995, %RSD < 20%, r^2 <0.990 Alternative Evaluation: Mean %RSD for all target Analytes < 15% Maximum allowable %RSD for each target analyte < 30%
ICV (4.9.3)	%Rec = 80% - 120%
CCV (4.9.5 /4.9.5.2 / 4.9.5.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %D < 30% Primary Evaluation (CCCs): %Drift < 20%, %D < 20% Qualitative, see text
MB (5.2.1.7.4.1)	Target Compounds: Compounds < MDL Check Sample (~2X MDL) or ½ PQL Common Lab Contaminant Compounds < PQLs
LCS (5.2.1.7.4.2)	Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125%
MS (5.2.1.7.4.3)	%Rec = 70% - 130%
MSD/MD (5.2.1.7.4.4)	Water: RPD < 25% Solids: RPD < 35%
Surrogates (5.2.1.7.4.5)	LCS: Water: %Rec = 80% - 120% Solids: %Rec = 75% - 125% Project Sample Matrix: %Rec = 70% - 130%

TABLE 4-13
SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8270
SEMIVOLATILE ORGANIC COMPOUNDS

QC Element	Target Compound/Surrogate
Initial Calibration (4.9.2.2.5)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %RSD < 30% Primary Evaluation (all target analytes) : r = 0.995, %RSD < 15%, r^2 <0.990 Alternative Evaluation: Mean %RSD for all target compounds < 15% Maximum allowable %RSD for each target compounds < 40%
ICV (4.9.3)	%Rec = 70% - 130%
CCV (4.9.5 / 4.9.5.2 / 4.9.5.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %D < 30% Primary Evaluation (CCCs): %Drift < 20%, %D < 20% Qualitative, see text
MB (5.2.1.7.4.1)	Target Compounds: Compounds < MDL Check Sample (~2X MDL) or ½ PQL Common Lab Contaminant Compounds < PQLs
LCS (5.2.1.7.4.2) Assuming Full List Spike	Water: %Rec = 60% - 120% (~20 compounds) = 45% - 135% (~30 compounds) = 20% - 150% (~45 compounds) Solids: %Rec = 60% - 120% (~20 compounds) = 45% - 135% (~30 compounds) = 30% - 150% (~45 compounds)
MS (5.2.1.7.4.3)	Water: %Rec = 45% - 135%

QC Element	Target Compound/Surrogate
	Solids: %Rec = 45% - 135%
MSD/MD (5.2.1.7.4.4)	Water: RPD < 35% Solids: RPD < 40%
Surrogates (5.2.1.7.4.5)	LCS: Water: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Solids: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Project Sample Matrix: Water: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds Solids: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds

[1] B = Base, N = Neutral, and A = Acid compounds (cmpds).

TABLE 4-14
SUMMARY OF METHOD QUALITY OBJECTIVES FOR METHOD 8330
EXPLOSIVES

QC Element	Target Compounds/Surrogate
Initial Calibration (4.9.2.2.6)	Primary Evaluation: r = 0.995, %RSD < 20%, r^2 <0.990 Alternative Evaluation: Mean %RSD for all target Compounds < 20% Maximum allowable %RSD for each target compounds < 40%
ICV (4.9.3)	%Rec = 85% - 115%
CCV (4.9.5 /4.9.5.2)	Primary Evaluation: %Drift < 15%, %D < 15% Alternative Evaluation: Mean %Drift/%D for all target compounds < 15% Maximum allowable %Drift/%D for each target compounds < 30%
MB (5.2.1.7.4.1)	Target Compounds: Compounds < MDL Check Sample (~2X MDL)
LCS (5.2.1.7.4.2)	Water: %Rec = 60% - 120%[2] Solids:%Rec = 60% - 120%[2]
MS (5.2.1.7.4.3)	%Rec = 50% - 140%[2]
MSD/MD (5.2.1.7.4.4)	RPD < 50%
Surrogates (5.2.1.7.4.5)	LCS: Water: %Rec = 60% - 140% Solids: %Rec = 50% - 150% Project Sample Matrix: %Rec = 50% - 150%
Target Compound Confirmation (5.2.3.4)	RPD < 40%

5.0 CHEMICAL DATA QUALITY MANAGEMENT PLAN

The CDQMP is intended to be an installation-wide document. The CDQMP covers both field and analytical requirements from a comprehensive perspective. It shall address the topics outlined in this section.

A. The Contractor shall submit a draft CDQMP for review and comment by the USACE Contracting Officer (CO). The plan shall be composed of a Field Sampling Plan and a Quality Assurance Project Plan. The CDQMP shall be prepared following the requirements of these specifications. The references listed in paragraph 2.1 and 2.2 will serve as useful adjuncts to these specifications. The format for the QAPP is taken directly from EPA QA/R-5. USACE EM 200-1-3 will be a useful reference for preparation of the FSP as well as providing material on implementation of three-phase control as applicable to sampling and analytical activities that must be described in the QAPP and FSP.

Note: While these references are provided as useful adjuncts to the requirements of this contract the requirements for document content that are described in these references may conflict. Only the requirements of this contract shall be operative with respect to document content and execution of aspects of work related to CDQM.

B. The CDQMP shall delineate the methods the Contractor intends to use to accomplish the chemical quality control items as indicated in these specifications to assure accurate, precise, representative, complete, legally defensible and comparable data. Qualifications and certification of the laboratory facilities projected for use on this contract shall be described in the CDQMP. The CDQMP shall represent the Contractor's corporate standards and procedures for execution of work related to sampling and analysis. As such, procedures applicable to sub-contractors associated with drilling, well installation, and laboratory analysis shall be explicitly and completely described in the text of the CDQMP. Please note that since the text of the CDQMP must represent the Contractor's corporate standards for execution of work inclusion of or reference to a laboratory QA manual or other subcontractor SOP's will be unacceptable to fulfill the requirements of these specifications as described below. The required material must be incorporated directly in the text of the CDQMP or a project specific SAP as applicable. The intent of these specifications is that the majority of the procedures that will be applicable for delivery orders for this contract will be described once at a high level of detail in the CDQMP. The CDQMP will also describe the format and type of project specific information to be included in delivery order-specific Sampling and Analysis Plans (SAPs). SAPs for this contract may reference specific paragraphs of the final CDQMP as applicable to describe procedures that are relevant for execution of delivery order tasks. Field and laboratory procedures not included in the CDQMP must be described in project-specific SAPs at the level of detail required for the original submittal of the CDQMP.

C. The CDQMP shall contain a statement of sampling procedures to include specifications of equipment and sample types. The plan shall address all levels of the investigation and all transportation and custody procedures. A level of detail shall be incorporated such that the document may be used as an audit guide for field and laboratory work.

D. While a certain degree of duplication between the FSP and the QAPP is inevitable the intent of these specifications is to minimize this duplication. In general the QAPP shall be the definitive document with respect to quality control procedures for field, laboratory, and general operations, analytical procedures, and reports to management. The FSP shall provide a comprehensive description of all aspects of field procedures to include sampling procedures and sample handling procedures. The Contractor will submit a draft CDQMP for USACE review and comment. After incorporation of all comments approved by the USACE CO the Contractor shall submit a revised text for USACE acceptance. Subsequent re-submittals may be required if comments are not resolved to the satisfaction of the USACE CO. All revisions of the text required to complete satisfactory

incorporation of comments will be performed at no additional cost to the Government. No sampling or analysis shall be performed without the acceptance of the CDQMP and project-specific SAPs by the CO. Additionally, in response to written comments from the Contracting Officer the final CDQMP shall be revised by the Contractor at annual intervals during the life of this contract. The CDQMP shall be prepared according to the following outline:

CHEMICAL DATA QUALITY MANAGEMENT PLAN/SAP (Section 5.0)

I. Title and Signature Page
II. Table of Contents
III. Executive Summary

QUALITY ASSURANCE PROJECT PLAN (Section 5.2)

Title Page
Table of Contents

1.0 PROGRAM MANAGEMENT
- 1.1 Program/Project Organization
- 1.2 Problem Definition/Background
- 1.3 Project Description
- 1.4 Data Quality Objectives
- 1.5 Documentation and Records

2.0 MEASUREMENT AND DATA ACQUISITION
- 2.1 Sampling Process Design
- 2.2 Sampling Methods Requirements
- 2.3 Sample Handling and Custody Requirements
- 2.4 Analytical Methods Requirements
- 2.5 Analytical Quality Control Requirements
- 2.6 Instrument Calibration and Frequency
- 2.7 Data Acquisition Requirements (Non-direct Measurements)

3.0 ASSESSMENT AND OVERSIGHT
- 3.1 Contractor Quality Control
- 3.1 Assessments and Response Actions
- 3.2 Reports to Management

4.0 DATA VALIDATION AND USABILITY
- 4.1 Data Review, Validation, and Verification Requirements
- 4.2 Validation and Verification Methods
- 4.3 Reconciliation with Data Quality Objectives

FIELD SAMPLING PLAN (Section 5.3)

Title Page
Table of Contents

1.0 Site Background
2.0 Sampling Objectives
3.0 Sample Types
4.0 Sample Location and Frequency
5.0 Field Documentation
6.0 Sampling Equipment and Procedures
7.0 Sample Handling Procedures
8.0 Investigative Derived Waste

9.0 Quality Control for Field Operations

E. A Sampling and Analysis Plan (SAP) shall be prepared for review and comment by the Contracting Officer for each delivery order authorized under this contract involving acquisition and analysis of environmental samples (soil, sediment, water, product, etc.). The site-specific SAP shall address project specific requirements for execution of a delivery order and may reference specific sections of the CDQMP for descriptions of field and laboratory procedures. Field or laboratory procedures not described in the CDQMP will be described in the site-specific SAP at a level of detail comparable to the CDQMP. The CDQMP shall describe the preparation of site-specific SAPs and shall include a discussion of all information required to be present in the site-specific SAP. The site-specific SAP shall be prepared according to the outline presented below:

SITE-SPECIFIC SAMPLING AND ANALYSIS PLAN (Section 5.4)
- Title and Signature Page
- Table of Contents
- Executive Summary
- 1.0 Problem Definition and Background
- 2.0 Project Description
- 3.0 Project Organization
- 4.0 Data Quality Objectives
- 5.0 Sampling Process Design
- 6.0 Sampling Methods Requirements
- 7.0 Analytical Methods Summary
- 8.0 Investigation Derived Wastes
- 9.0 Quality Control
- 10.0 References

To provide a complete document for regulatory review for project specific submittals inclusion of referenced sections of the CDQMP as appendices to the SAP may be required.

5.1 SAMPLING AND ANALYSIS PLAN

The CDQMP/SAP is considered a general guidance for the overall contract or for use during all base-wide or installation-wide activities. Site-specific activities requirements and data quality objectives will be outlined in subsequent project-specific SAPs. For long-term projects (>2 years) the QAPP portion of the SAP shall be revised on an annual basis.

5.1.1 Title & Signature Page

The title page shall identify the contract, project name; site location; applicable program; office locations; Contract Laboratory; and the Government QA Laboratory need to be specified in the SAP. The title page will be followed by a signature page, which shall include the signatures of principal personnel involved in development and execution of the CDQMP (Contractor Program Manager, Contractor Project Manager, Contractor Technical Professionals, and Contract Laboratory Director).

5.1.2 Table of Contents

The table of content will include a breakdown of each major section and appendices or attachments.

5.1.3 Executive Summary

The executive summary shall be composed of a brief description of the context of contract or project work, the goal of the proposed work, a general description of the work to be performed, and a brief statement describing the relevance of the work to be performed to the goal of the program, as applicable.

5.2 QUALITY ASSURANCE PROJECT PLAN

The QAPP is intended to outline the specific requirements for the analytical procedures.

5.2.1 Title Page and Table of Contents

The title page will include the title of the document, contract number, who the document was prepared for, the location of the base or project, designation regarding draft or final, the date published, and who prepared the document.

The table of content will include a breakdown of each major section and appendices or attachments, list of tables, list of figures, list of acronyms, and list of references.

5.2.2 Program Management

This section discusses the organizational structure for the program/project management, which includes the responsibilities of each project management team member, problem definition, and background, project description, data quality objectives, and documentation and records requirements.

5.2.2.1 Program and Project Organization

Program Organization. The CDQMP shall address all organizational items that are general for the contract but must include the overall QC organization and lines of authority to management. The CDQMP must address not only the prime Contractor, but any subcontractor and Contractor-subcontractor interactions known to be applicable. Key personnel must be identified along with their function and qualifications. The text shall include a chart showing lines of authority and communication among all program participants. The organization chart shall be realistic and practical and shall reflect only the actual lines of authority and communication for the program.

5.2.2.1.1 Quality Assurance Officer

Government employees acting as a QA Officer (QAO) on behalf of the USACE will direct the project during the initial planning stages of investigation and throughout its lifetime to help ensure the DQO requirements established in the CDQMP are met. Part of the QAO's responsibility is to review QAPPs, FSPs, revisions and addenda. The QAO and government project managers will be identified clearly in the organization chart and their responsibilities described in Section 1.0 of the QAPP. The QAO's signature block will be clearly indicated on the approval page of the document.

Comments provided by the USACE and regulatory agency QAO for QAPPs and FSP will be provided to the USACE and regulatory project managers. All responses to comments will likewise be exchanged to ensure all comments are satisfactorily addressed.

Once a QAPP, FSP, revision or addendum are approved by the USACE, government project managers may assist the DOD and regulatory QAO in the implementation of the approved documents by transferring custodial oversight responsibility to them. Under the direction and oversight of the QAO, project managers are fully responsible for understanding and ensuring that the work performed in the field and laboratory meet the DQOs set forth in the QAPP, FSP, and any revisions or addendum thereof. Regulatory and USACE QAOs retain all and full QA authority over the program within their respective agencies. The extent to which the custodial oversight responsibilities are transferred to the project manager will be documented in the corresponding QAPP.

Once a plan, revision, or addendum is approved and custodial oversight transferred, the project managers are responsible for providing oversight to ensure the success, or failure, of the project. Project managers are at the forefront of the activities occurring in the field and laboratories and are the parties most knowledgeable about the day to day activities and out of control events. They are the immediate and active deterrent to prevent deviation from QAPPs and FSPs. Because limited authority is provided to project managers, they must seek approval from the QAO

on issues arising in the field and laboratory which potentially impact data quality.

Where selection of a laboratory is a primary contractor's responsibility, the Primary contractor is responsible for ensuring that the laboratory can perform the data quality technical requirements identified in the QAPP or FSP. The primary contractor will also provide a copy of the QAPP or FSP to the laboratory to ensure that it has the necessary documentation to follow and reference. Both the primary contractor and laboratory will be responsible for ensuring that all data quality requirements are met as stipulated in the contract. This does not relieve project managers from the regulatory and USACE from performing their custodial oversight responsibilities to ensure that data is collected and analyzed as specified in the QAPP or FSP and that the overall work performed meets the DQO requirements of the project. It is suggested that project managers work closely with a chemist from their respective agencies to assist in the oversight of the laboratory, if necessary.

5.2.2.1.2 Program Chemist - As part of the project organization, the Contractor shall appoint a Program Chemist for sampling and analytical activities who is responsible to a senior company officer. The Program Chemist should have general knowledge of remedial process chemistry, fate and transport of organic compounds and inorganic analytes, knowledge of chemical quality control, experience in the sampling and analysis of toxic/hazardous chemicals and radiological contamination in environmental matrices. The Program Chemist will be required to have advanced expertise (senior level) in chemical data quality management of environmental analytical data. The Program Chemist will be appointed by senior corporate or project management to be principally responsible for oversight of all quality control operations for field and laboratory activities related to sampling and analysis.

The Program Chemist shall have, as a minimum, the following qualifications:

a. A 4-year college degree in Chemistry from an accredited post-secondary institution.
b. A minimum of 10 years of professional experience in Chemistry of which a minimum of seven years must be directly related to environmental investigations and/or remedial actions as a part of a Contractor management team (i.e. not primarily employed at a laboratory).
c. A minimum of two (2) years experience at the level of a commercial environmental analytical laboratory with expertise in standard analytical chemistry methods common for analyzing soil, water, air and other materials for chemical contamination assessment.

The Program Chemist will be expected to have a lead role in management of project tasks associated with sampling and analysis including preparation of the CDQMP, preparation of Sampling and Analysis Plans, instruction of field personnel in sampling and preservation requirements, general oversight of field personnel involved in sampling activities, coordination with the analytical laboratory to insure readiness to implement project specific requirements, participation in on site inspections of the Contract Laboratory, review of analytical data as it becomes available to insure conformance with quality standards, implementation of corrective actions in accordance with these specifications when review of data uncovers deficiencies, and serve as a general point of contact for the USACE CO for issues related to environmental chemistry. The Program Chemist shall be employed or subcontracted by the Contractor and shall not be employed by a laboratory performing analyses for this contract.

The program and project chemists will be proposed by the contractor and are subject to approval by the USACE District Chemist. Any proposed changes in chemistry staff shall be approved (prior to the change) by the District Chemist.

Project Organization. This section in the SAP shall address the specific personnel that will be responsible for execution of a delivery order. The SAP must address not only the prime Contractor, but any subcontractor and Contractor-subcontractor interactions applicable for a delivery order. Key personnel must be identified along with their function and qualifications. The text shall include a chart showing lines of authority and communication among all project participants. Include other data users who are outside of the organization generating data, but for whom the data are nevertheless

intended; e.g. modelers, risk assessors, design engineers, toxicologists, etc. Where direct contact between project managers and data users does not occur, the organization chart should show the route by which information is exchanged. The organization chart shall be realistic and practical and shall reflect only the actual lines of authority and communication for the project described.

5.2.2.1.3 Project Chemist - As part of the project organization, the Contractor shall appoint a Project Chemist. The Project Chemist must have knowledge of environmental analytical chemistry methodologies as described in EPA SW-846, and quality control procedures as applicable to environmental analytical chemistry.

The Project Chemist shall have, as a minimum, the following qualifications:

a. A minimum of a 4-year college degree in Chemistry from an accredited post-secondary institution.
b. A minimum of four years of combined professional experience at the level of a commercial environmental analytical laboratory or working as a part of a Contractor project management team of which a minimum of 1-2 years must be directly related to environmental investigations and/or remedial actions as a part of a Contractor management team (i.e. not primarily employed at a laboratory).

The Project Chemist will be expected to have a "hands on" role in management of project tasks associated with sampling and analysis including preparation of the CDQMP, preparation of Sampling and Analysis Plans, instruction of field personnel in sampling and preservation requirements, general oversight of field personnel involved in sampling activities, 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 these specifications when review of data uncovers deficiencies, and serve as a point of contact for the USACE CO for issues related to environmental chemistry. The Chemist shall conduct or oversee all onsite analytical testing including field-screening tests. The Project Chemist shall coordinate Government Quality Assurance testing that verifies the Contractor chemical data. The Chemist shall review and verify all chemical data for hazardous waste manifests. The Chemist shall also prepare all data validation reports or review for accuracy all data validation reports prepared by subcontractors. The Project Chemist will perform an inspection of the Contract Laboratory at or near the beginning of sample analyses for each delivery order to insure laboratory capability to implement method and contract specified aspects of work. Method specific checklists presented in USACE EM 200-1-1 or equivalent in conjunction with the contract specifications and the final CDQMP shall be used as the basis for this inspection. Findings of this inspection shall be delivered by memorandum to the USACE CO within 15 days of completion. Inspection checklists shall be included as an attachment to the memorandum of findings. This review of the Contract Laboratory may be conducted concurrently with a project kickoff meeting, preparatory, or initial inspection. The Project Chemist shall be employed or subcontracted by the Contractor and shall not be employed by a laboratory performing analyses for this contract.

The project chemist must be fully integrated into the project team from planning, work plan document writing, field execution, laboratory management and final report writing.

Note: If the same laboratory is used for multiple delivery orders a complete technical systems review will not be required for concurrent delivery orders. However, if new analyses are performed for which the Program or Project Chemist has not performed an assessment of laboratory capability to execute the requirements of the CDQMP a technical systems review will be required for those analyses. At a minimum a technical audit of the laboratory will be performed on an annual basis and whenever required as a function of deficiencies in laboratory performance.

5.2.2.2 Problem Definition and Background

The CDQMP will describe specific details that will be included in project-specific SAPs as described below. The SAP will include a project-specific discussion of all items that are described below.

A narrative describing the project shall be included that shall state the specific problem to be solved or the decision to be made. The goal of the investigation shall be clearly stated. The Contractor shall describe 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. The text shall include diagrams detailing areas to be sampled as relevant to the definition of the investigation goals. These sections shall also contain a summary of site geology/hydrogeology as known prepared to a level of detail such as to provide a comprehensive description of the site. The discussion must include enough information about the problem, the past history, any previous work or data, the regulatory or legal context, and any relevant ARAR's to present a clear description of the project objectives. The Contractor shall be responsible for researching all necessary references to accomplish this task and shall not rely upon the USACE Contracting Officer to provide relevant information regarding problem definition/background.

5.2.2.3 Project Description

The CDQMP will describe specific details that will be included in project specific SAPs, as described below. The SAP will include a project specific discussion of all items that are described below.

The text shall provide a description of the work to be performed. This discussion may not be lengthy or overly detailed but it shall give an overall picture of how the project will resolve the problem or questions described in the definition and background of the problem. A general description of the sampling to be carried out for this project shall be included. Anticipated project start and completion dates shall be included. Describe in general terms:

- Measurements that are expected during the course of the project and the approach that will be used.
- Applicable technical, regulatory, or program specific quality standards, criteria, or objectives.
- Any special personnel and equipment requirements that may indicate the complexity of the project.
- Assessment tools that will be employed for the project (program technical reviews, peer reviews, surveillances, technical audits, etc.)
- Project schedule or a sequence of milestones and their expected duration. If individual-sampling plans will be developed for discrete project phases include their preparation schedule.

5.2.2.4 Data Quality Objectives

The CDQMP will describe specific details that will be included in project-specific SAPs as described below. The SAP will include a project specific discussion of all items that are described below.

The text shall describe the general scope of work and background information as it relates to the acquisition of geological, geophysical, hydrogeological, and chemical data. The text shall 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 shall be used to obtain data of sufficient quality to meet or exceed all project objectives. The discussion of Data Quality Objectives (DQOs) shall follow the guidance contained in the EPA document "EPA Guidance for the Data Quality Objectives Process", EPA/600/R-96/055, Final, September 1994, EM 200-1-2, Technical Project Planning Guidance for HTRW Data Quality Design, and the requirements of these documents 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 section on DQOs will address the following topics in the specified order:

(1) Statement of the Problem. Summarize the problem that requires environmental data acquisition and identify the resources available to resolve the problem.
(2) Identification of Decisions. 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 shall be prioritized.
(3) Identify Inputs to Decisions. Identify the information needed to support the decision and specify the

inputs requiring environmental measurements.

(4) Definition of Study Boundaries. Specify the spatial and temporal aspects of the environmental media that the data must represent to support the decision.

(5) Development of Decision Rules. Develop a logical statement that defines the conditions that would cause the decision-maker to choose among alternative actions.

(6) 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.

(7) 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 DQOs.

Statements of the problem shall be defined quantitatively if possible. For example:

> UV Treatment of Contaminated Groundwater. "The purpose of this project is to demonstrate that the residual trichloroethylene concentration in the treated water is less than 0.5 ug/L at a confidence level of 95%."

Identification of decisions and descriptions of data use shall be described with text and supported with tables and lists that describe:

- Data needed. Measurement parameters, compounds, and sample matrices.
- The action levels or standards upon which decisions will be made, including the detection limits and data reporting units for relevant parameters.
- The summary statistic(s), e.g., mean maximum, range, etc., which specify the form the data will be in when compared against action levels or standards.
- The acceptable level of confidence in the data needed for the stated purposes; or the acceptable amount of uncertainty.

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

To generate data that will meet the project-specific requirements, it is necessary to define the types of decisions that will be made and to identify the purpose of the data. DQOs are an integrated set of specifications that define data quality requirements based on the intended use of the data. Project-specific DQOs are established to encompass both the field and laboratory operations. The DQO process leads to the specification of the following at a minimum: (1) sample handling procedures, (2) preparatory (extraction/digestion), cleanup, and determinative methods, (3) target analytes, (4) method quantitation or reporting limits, (5) field and laboratory quality control samples, (6) method quality objectives (QC acceptance limits) and data quality indicators (formerly PARCC parameters) performance objectives, (7) required corrective actions, and (8) data assessment procedures necessary to meet the intended use of the data. Special considerations which may also apply include: internal laboratory sample chain-of-custody, data confidentiality, data archival, or data retention requirements beyond those stated herein.

A. Assessment of Data Needs. As presented in EM 200-1-2, data needs are determined for the project based upon the decisions, which need to be made. At the same time, a determination of the data quality required for each piece of data (data need) must also be defined by the eventual data user. This information, whether given as a maximum allowable quantitative uncertainty or a qualitative statement of requirements, will help other technical planners (data implementors) to identify applicable sampling and analytical protocols to generate the required data. In order to accomplish this, all data needs should be compiled and grouped by location, matrix, and parameter. Once the

grouping is completed, the data quality requirements of these needs are assessed by analytical parameter (per matrix, per area). It is possible to have more than one data user requesting the same analytical parameter for a particular area's media. In those cases, the most stringent data user requirements are applied to ensure the suitability of these data by all requesting parties. This information is then used to decide the type of data necessary (screening or definitive), and the appropriate sampling and analytical methods to be proposed for collecting and generating the required data.

B. Assessment of Data Collection Options. Initially, the applicability of field analytical methods to the objectives of the project should be investigated. These may be used in conjunction with or without more rigorous analytical methods which the analytical error has been determined (i.e., definitive data). Field analytical methods include (1) qualitative or semi-quantitative field screening techniques *(e.g., photoionization detector/flame ionization detector (PID/FID), immunoassay, colorimetric, etc.)*, and (2) quantitative onsite techniques whose preparatory process and/or QC elements are typically less rigorous than those established for definitive data *(e.g., x-ray fluorescence (XRF), gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), etc.)*.

Standard analytical methods producing definitive data must also be reviewed for applicability to the project. Input necessary to determine applicable screening or definitive analytical techniques include at a minimum defining the (1) contaminants of concern, (2) the concentration range of interest, (3) sensitivity requirements for detection, and quantitation limits, (4) method quality objectives for precision, bias, and completeness, (5) the need and type of confirmation necessary, and (6) whether any physical, chemical, or logistical constraints are germane. The method may also be dictated by the data user (e.g., outlined by regulatory authority or ROD).

5.2.2.5 Documentation and Records

The text shall itemize the information and records, which will be included in a data report package for each delivery order, and specify the reporting format. Content of items such as well completion reports, boring logs, and data packages shall be described in detail. Examples of well completion reports and finished boring logs as relevant shall be included in appendices to the QAPP for USACE comment regarding content of these materials. Specifics regarding the QAPP content with respect to chemical data are included below.

- **Data Reduction, Validation, and Documentation:** The Contractor shall provide in the QAPP for each analytical method and major measurement parameter the following:
- **Calculations:** The QAPP shall provide, for each analytical method, details regarding the data analysis scheme including units and equations required to calculate concentrations or the value of the measured parameter.
- **Procedures to Ensure Data Integrity:** The QAPP shall identify the principal criteria used to assure data integrity during collection and reporting. The means of establishing these criteria must be identified as well as procedures implemented to provide corrective action when data or instrumentation that do not meet these criteria. Possible matrix interferences for laboratory analyses attributable to site characteristics shall be identified and methods for compensating for expected or unexpected interferences shall be detailed in the QAPP.
- **Treatment of Outliers:** The QAPP shall describe the specific mechanisms employed when outlier data are identified. Limits of data acceptability shall not be exceeded. Details provided shall include a description of the phase of the analytical process where these systems are employed, and the process by which subsequent decisions regarding the disposition of the data in question are made. Information justifying the poor recovery or precision shall be documented when limits are exceeded. The CO will then decide what further action, if any, need be taken. Personnel responsible for initiating and executing a corrective action shall be indicated in the protocol.
- **Data Management:** The Contractor shall provide detailed information regarding the handling of data, including the types and mechanisms of review processes and the qualifications of the various individuals

involved in this activity.

- **Data Archive:** The QAPP shall describe the specific procedures employed to archive data, including a description of any hardware involved (computers, etc.). Handling and storage procedures for all raw data shall also be described. Since the ultimate use of the data is not known the Contract Laboratory shall preserve all information regarding sample analyses (calibration records, etc.) such that the analytical process can be reconstructed at some future time. The Contract Laboratory shall maintain all data associated with delivery orders for this contract for a period of ten years following submission of the certificate of analysis including all relevant electronic media used for data storage.
- **Format for Comprehensive Certificates of Analysis:** The Contract Laboratory shall address the requirements in preparing comprehensive certificates of analysis outlined in Section 5.5.

Unless otherwise specified in delivery order specific scopes of work the comprehensive certificate of analysis shall be prepared for each group of samples submitted to the Contract Laboratory and shall be received by the CO no later than 21 days after sample acquisition in the field. This submittal is subject to review and comment by the CO. The Contractor will be directed to resubmit the comprehensive certificate of analysis at no additional charge to the Government if the conditions of these specifications are not executed by the Contractor.

Draft certificates of analysis containing analytical results and preliminary QC data only shall be submitted to the CO as soon as they are available (approximately 10 working days after sample shipment to the Contract Laboratory for each shipment of samples, not to be interpreted as a requirement for 10 day turn-around). Draft certificates of analysis do not have to satisfy all of the requirements of this section but should contain basic QC information such as MS/MSD analyses, LCS analyses, method blank results, chain of custody forms, and cooler receipt forms. The Contractor is encouraged to select a laboratory that has the capability for electronic transmission of data as this will greatly facilitate delivery of draft certificates of analysis.

5.2.3 Measurement and Data Acquisition

The following sections describe the requirements for sampling process design, sampling method requirements, sample handling and custody requirements, analytical method requirements, and analytical, statistical, and control parameters.

5.2.3.1Sampling Process Design

The CDQMP will describe the items to be contained in the SAP as described below. The SAP for each delivery order will provide a detailed project specific discussion of the requirements presented below.

Outline specifically the experimental design of the project including the sampling network design, types of samples required, sampling frequencies, sample matrices, and measurement parameters of interest. The rationale for the design shall be clearly stated. The rationale for the design shall be described for all sites where samples shall be obtained and will be supported with figures describing the specific points where samples shall be obtained. Measurement parameters to be described shall include geological, geophysical, hydrogeological, and chemical parameters as applicable. If cone penetrometer locations, hydropunch locations, or monitoring well locations are to be chosen on the basis of field observations the text shall clearly state the evaluation criteria that shall be used in the field for these determinations. Monitoring well design criteria (if applicable) shall be clearly described to include a description of field determinations for appropriate filter packs and well screens.

5.2.3.2 Sampling Methods Requirements

The CDQMP will describe the items to be contained in the SAP as described below. The SAP for each delivery order will provide a detailed project specific discussion of the requirements presented below.

Provide a general description of sample collection procedures. Detailed specific descriptions of these procedures shall be described in the FSP and the SAP shall reference the specific paragraphs applicable from the FSP. For each sampling

method identify any support facilities needed. The discussion shall focus on Contractor procedures for addressing failures in the sampling system and responsibilities for corrective action. The text shall include a table that describes bottle requirements, preservation, and holding times to extraction and/or analysis for all analytical parameters and matrices.

5.2.3.3 Sample Handling and Custody Requirements

Provide a general description of provisions for sample handling taking into account the nature of the samples and the maximum allowable holding time. Specific sample handling procedures will be described in the FSP. The text of the QAPP should focus on quality control for sample handling and custody procedures and discuss Contractor procedures for controlling common problems such as labeling errors, chain-of-custody errors, transcription errors, preservation failures, etc.

5.2.3.4 Analytical Methods Requirements

Section 4.0 of this specification outlines the requirements for specific methods. The following sections describe the requirements for screening and definitive level data. Screening level data are data generated by rapid, less precise methods of analysis with less rigorous sample preparation methods. Screening data provide analyte identification and quantification, although quantification may be relatively imprecise. At least 10 percent of the screening data must be confirmed using definitive data in order for the screening data to be of known quality. Definitive level data are data generated at the site or off site in analytical laboratories using rigorous analytical methods, such as approved EPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration, and analytical error or total error is determined.

A. **Screening Level Data**. The results of the analyses detailed in the approved CDQMP will provide the final determination as to the presence and extent of contamination (as verified by the appropriate QA/QC procedures); however, the Contractor may wish to investigate the availability and/or utility of colorimetric, immunoassay or other "field" analytical methods (screening level data). If appropriate, such methods could be used to provide a preliminary indication of contamination at sampling locations. The use of such a method is subject to review and approval by the CO, and must meet the requirements for a Contract Laboratory as outlined elsewhere in this document. In particular, work performed by a mobile laboratory or a temporary field unit mobilized to the site will be required to meet all of the requirements of the text of this specification as they relate to sample analysis performed by a fixed site laboratory. All field-screening methods shall be detailed in the QAPP or the SAP (as applicable) to the level of detail required for fixed site laboratory analyses described in these specifications.

B. **Definitive Level Data.** The Contractor is responsible for the quality of all data produced by the Contract Laboratory. All samples shall be prepared/analyzed per the referenced analytical methods specified in the approved CDQMP. Alternate or additional procedures must be pre-approved by the CO. The QAPP shall contain a brief description of each laboratory analytical method to be used for acquisition of chemical data. The text shall be specific including relevant aspects of laboratory procedures (general extraction method, analytical detector, instrumentation, etc.). The text must include a discussion of instrument preventive maintenance programs, specific instrument calibration procedures and frequency, method-specific data quality objectives, laboratory quality control criteria, laboratory corrective action, data reduction, and data validation. Corrective action descriptions shall be specific for each controlled parameter for each analytical method. Tabular presentation of Contract Laboratory quality control criteria and corrective action procedures is acceptable. Submission of Contract Laboratory SOPs, Contract Laboratory QA Manual, incorporation of SW-846 requirements by reference, or inclusion of excerpted sections of SW-846 and other standard analytical methods is unacceptable to fulfill the requirements of this specification.

5.2.3.4.1 Analytical/Statistical/Control Parameters

Measurement criteria shall be defined for the critical indicator parameters of data quality – which are precision, accuracy,

representativeness, completeness, and comparability (PARCC) – and the detection and quantitation limits. PARCC criteria will be specified for both screening and definitive level data. Measurement objectives for these indicator parameters will be developed based on the analytical methods, screening techniques, and the data quality objectives of the project.

As previously noted, QC procedures are employed during chemical analysis to support and document the attainment of established method quality objectives. Whether these QC procedures support an assessment of general batch control or matrix-specific application, documentation includes calculating data quality indicators to verify data usability and contract compliance. Data quality indicators were formerly referred to as the PARCC parameters and sensitivity. All laboratories conducting analytical work for the USACE must be aware of, and be in agreement with, the project DQOs, including the stated data quality indicators - method quality objectives. ***To avoid any misunderstandings concerning the level of quality required for the project chemical analyses, the SAP must very clearly delineate all method quality objectives for the method QC checks and data quality indicators (precision, bias, representativeness, comparability, completeness, and sensitivity) for each method applied.*** Tables 4-7 through 4-14 summarize the method quality objectives for eight (8) SW-846 methods. ***These tables may be applied directly to a project, or modified accordingly to define the method quality objectives for laboratory data quality indicators (precision (P) and bias (B)) of the LCS, MS, MD/MSD, etc.***

However, project requirements must still be defined for the remaining applicable data quality indicators within appropriate project documents (e.g., SOW, SAP). For example: (1) data quality indicator performance objectives of field QC samples (precision objective for field replicates, bias objective for field blanks, bias objective of double-blind PE samples, etc.); (2) data quality indicator performance objectives for matrix-specific sensitivity (per requisite methods); (3) data quality indicator performance objectives for project completeness (note whether field and lab completeness are assessed separately or combined); and (4) qualitative data quality indicator (representativeness, comparability).

5.2.3.4.1.1 Precision

Precision shall be evaluated through the collection and analysis of field and laboratory duplicate samples. Field duplicates (QC samples) shall be collected at a frequency of one duplicate for each ten samples of a given matrix. The identity of QC samples shall be held blind to the Contract Laboratory until after analyses have been completed.

The relative percent difference for field and laboratory duplicates shall be calculated and used as a measure of precision, however only laboratory duplicates will be included in the quantitative assessment of completeness. Results of field duplicates will be described in qualitative assessment of completeness.

Laboratory duplicates are defined as two aliquots obtained from the same sample which are extracted and analyzed for the purpose of determining matrix specific precision. Laboratory duplicates shall be performed for all metals analyses at a rate of one in twenty (one for each batch up to a maximum of twenty). Precision for organic analyses may be determined by the analysis of matrix spike/matrix spike duplicate samples.

Contract Laboratory quality control criteria for RPD for field and laboratory duplicates shall be specified in the QAPP. Laboratory duplicate samples not meeting quality control criteria shall be re-extracted/reanalyzed once. (For organic analyses failure of different matrix spike compounds to meet QC criteria on successive runs shall constitute failure and satisfy the requirement for reanalysis.) Quality control criteria are subject to approval by the CO. Failure of the Contract Laboratory to present QC criteria for precision (including corrective action) that are acceptable to USACE will result in directing of the Contractor by the CO to retain another laboratory for contract services.

A. Bias. Bias refers to the systematic or persistent distortion of a measurement process, which causes errors in one direction (above or below the true value or mean). Bias may be affected by errors made in field or laboratory handling

procedures. For example, procedural deviations in sample acquisition, or incomplete homogenization prior to subsampling , or incomplete extraction of contaminants from the matrix intensify bias. Bias is a term, which is related to but is not interchangeable with accuracy. ***Bias assessments are typically based upon the analysis of spiked reference materials or spiked samples (i.e., LCS, MS, MSD, surrogates).*** When the sample matrix is spiked, the result allows an assessment of the effect of the sample matrix on recoveries. The sources of error contributing to the bias of a measurement can be difficult to determine for an entire sample collection/analysis activity. Sources of error may include the loss (or addition) of contaminants from the sampling and analysis process (i.e., sample handling, field cross-contamination, improper sample preservation, sample manipulation during preparation and analysis), interferences present within the sample matrix, and measurement error (i.e., calibration error or drift). Bias values for the LCS represent quantitative limits beyond which data are unacceptable. Bias values are commonly expressed as percent recovery.

5.2.3.4.1.2 Accuracy

A. Organic Analyses. Accuracy shall be evaluated through the collection and analysis of matrix spike, matrix spike duplicate (MS/MSD) samples, laboratory control samples (LCS), and by spiking all samples with surrogate compounds where applicable. Only samples from this project will be used for MS/MSD procedures. Trip blanks and rinsate samples will not knowingly be used for MS/MSD analyses.

B. Matrix Spike and Matrix Spike Duplicate. For each shipment of samples that is sent to the Contract Laboratory one sample shall be provided in sufficient quantity such that a matrix spike and a matrix spike duplicate can be generated in addition to an aliquot reserved for actual sample analysis. (If more than 20 samples are shipped at any time one sample will be provided in quantities sufficient to generate a MS/MSD for each 20 samples.) This sample will include sufficient volume such that one re-extraction/reanalysis of the MS/MSD pair may be performed if necessary. Alternatively, with the concurrence of the USACE CO, the Contractor may coordinate with the Contract Laboratory such that the laboratory is continually aware of sample collection and delivery such that batch size may be maximized and alleviate the otherwise necessary requirements for collection of samples for MS/MSD procedures for each shipment. The sample that is chosen for matrix spiking purposes shall be representative of the other samples in the batch. For large investigations where samples are being collected at multiple sites, to the extent that it is practical, sample batching and matrix spiking should reflect the sampling at specific investigation sites. In this case the Contractor should select the sample to be used for matrix spiking and the samples that it should be batched with.

The matrix spike and matrix spike duplicate samples shall be fortified with a series of method target compounds, while a third aliquot of the sample shall be analyzed unfortified. Accuracy shall be measured in terms of percent recovery of each of the fortified components. MS/MSD analyses not meeting the laboratory quality control criteria specified in the QAPP shall be re-extracted/reanalyzed once at no additional cost to the government. Both the MS and the MSD must be compliant for both accuracy and precision for all spiked compounds for the MS/MSD pair to be considered acceptable. Both the MS and the MSD must be re-extracted/reanalyzed in the event of failure. Failure of different spike compounds on successive runs for methods with multiple spike compounds will be considered a reanalysis failure and will satisfy the requirement for reanalysis. (Note: This provision is meant to apply for a single analytical method. This language shall not be construed to indicate that failing QC results for one analytical method are applicable to another.) Failure of MS/MSD analyses to meet QC criteria shall initiate a review of the data for the corresponding analytical batch. A determination should be made as to whether the failing matrix spike result is representative of the sample that was spiked or is representative of the entire batch. Reanalysis and/or re-extraction and reanalysis of the batch may be required if trend analysis of the batch data indicates that the analytical system is out of control.

C Surrogate Standards. Analyses exhibiting out of control surrogate recoveries shall be re-extracted/reanalyzed once at no additional cost to the government. For GC/MS analyses of volatile and semivolatile organic

compounds the SW-846 QC acceptance criteria for surrogate recoveries shall be employed.

D. **LCS.** LCS analyses are matrix spikes on a blank matrix (DI water, reagent sand) to assess Contract Laboratory accuracy independent of matrix effects. Use of sodium sulfate and/or other approved matrices may be used with the prior approval of the USACE CO. Failure of the LCS to meet QC criteria will result in re-analysis of the LCS sample to determine if the failing result is representative of a transient instrumental condition. (Failing LCS samples for extractable parameters will not be re-extracted in an attempt to validate the results from initial extraction. If the laboratory employs a routine system of running dual LCS samples, both results must be acceptable for the batch to pass.) A second failure will result in mandatory re-extraction/reanalysis of the entire analytical batch. Upon failure, initial reanalysis of the LCS must occur in real time with respect to sample analyses, otherwise reanalysis (at a minimum) of the batch will be mandatory. In the event of batch re-analyses for GC/MS analyses all SPCC and CCC criteria must be met for the reanalysis to be valid.

The QAPP shall detail matrix, method, and compound-specific procedures and quality control criteria for all sample analyses used to determine Contract Laboratory accuracy. Laboratory generated criteria for accuracy may be utilized with the provision that they must comply with the guidelines specified below. If laboratory generated QC criteria do not fall within the specified ranges the criteria described below will be utilized by default. If the laboratory generated QC criteria exceed the requirements of these specifications the laboratory generated QC criteria shall be utilized. QC criteria for this project will not exceed the ranges described in these specifications.

E. **GC/MS.** QC criteria for GC/MS analyses shall conform to SW-846 criteria for surrogate recoveries and use full standard list of compounds to be spiked for MS/MSD analyses and QC criteria. LCS criteria for GC/MS analyses shall utilize the 65-135% criteria for MS/MSD analyses as a minimum standard. QC criteria for LCS recoveries should generally be more stringent relative to MS/MSD criteria. Laboratory methods used to generate QC criteria must include analyses that discard outlier data. Use of all historical data is discouraged as this may result in QC ranges that may exceed the limits of acceptability.

F. **Other Organic Methods.** QC criteria for GC, HPLC, and TRPH (418.1 & 413.2) analyses (surrogate recoveries, LCS recoveries, and MS/MSD recoveries as applicable) shall fall within a 65-135% range.

Quality control criteria are subject to approval by the CO. Failure of the Contract Laboratory to present QC criteria (including corrective action) that are acceptable to USACE will result in direction of the Contractor by the CO to retain another laboratory for contract services.

Under certain limited circumstances, such as the occurrence of gross chromatographic interference, it is reasonable to infer that reanalysis or re-extraction/reanalysis would produce the same result. Under these circumstances re-extraction and reanalysis as described in these specifications would not be required. However, if this argument is proposed by the laboratory the data package submitted must include chromatographs (and any other raw data necessary), presented at an attenuation where aspects of the chromatography are clearly visible, to substantiate assertions of this type. This language shall not be interpreted to indicate that all appropriate sample cleanups are not required or that failure to execute appropriate sample cleanups prior to concluding that matrix effects are operative will be acceptable to USACE. The final determination of the acceptability of the laboratories actions in deviating from the basic requirements of these specifications will be made by the USACE CO.

Note: Contract Laboratories are cautioned not to attempt to use the provisions of this paragraph in an effort to evade the basic requirements for re-extraction and/or reanalysis as described in this specification. Data found to be associated with laboratory failure to execute the basic requirements of this contract for re-extraction and reanalysis, in the absence of a bona fide explanation for not executing these requirements, will be rejected by the USACE CO. The Contractor/Contract Laboratory is encouraged to contact the USACE CO or their designated representatives at any time if there is a question

regarding the appropriate course of action to take in the event of QC failures for obvious causes not related to laboratory performance.

G. Inorganic and General Chemistry Analyses. Accuracy for inorganic analyses shall be evaluated through the collection and analysis of matrix spike samples and laboratory control samples (LCS). For each shipment of samples that is sent to the Contract Laboratory one sample shall be provided in sufficient quantity such that a matrix spike can be generated in addition to an aliquot reserved for actual sample analysis. (If more than 20 samples are shipped at any time one sample will be provided in quantities sufficient to generate a MS for each 20 samples.) This sample will include sufficient volume such that one re-extraction/reanalysis of the MS may be performed if necessary. Alternatively, with the concurrence of the USACE CO, the Contractor may coordinate with the Contract Laboratory such that the laboratory is continually aware of sample collection and delivery such that batch size may be maximized and alleviate the otherwise necessary requirements for collection of samples for MS procedures for each shipment. The sample that is chosen for matrix spiking purposes shall be representative of the other samples in the batch. For large investigations where samples are being collected at multiple sites, to the extent that it is practical, sample batching and matrix spiking should reflect the sampling at specific investigation sites. In this case the Contractor should select the sample to be used for matrix spiking and the samples that it should be batched with.

The matrix spike samples shall be fortified with the method target compounds, while an aliquot of the sample shall be analyzed unfortified. The matrix spike for inorganic analyses shall be an analytical spike, i.e. a spike of the solution being extracted prior to the extraction procedure. Accuracy shall be measured in terms of percent recovery of each of the fortified components. MS analyses not meeting the laboratory quality control criteria specified in the QAPP shall be re-prepared/reanalyzed once at no additional cost to the government. QC criteria for matrix spike analyses shall fall within a range of 75-125% for inorganic and general chemistry analyses.

LCS analyses are matrix spikes on a blank matrix (DI water, reagent sand) to assess Contract Laboratory accuracy independent of matrix effects. LCS analyses shall be performed for each batch of samples up to a maximum of 20. Failure of LCS analyses shall result in re-extraction and reanalysis of the corresponding analytical batch for the specific analyte failing QC criteria. QC criteria for LCS analyses shall fall within a range of 80-120% for inorganic and general chemistry analyses.

Failure of MS analyses to meet QC criteria shall initiate a review of the data for the corresponding analytical batch. If review indicates out-of-control data due to laboratory error the Contract Laboratory shall perform re-extraction/reanalysis of the batch to correct the out-of-control condition at no additional cost to the Government.

The interference tests specified by paragraph 8.5 of SW-846 Method 6010B and by paragraph 8.6 of SW-846 Method 7000A shall be performed on one "representative" sample from each analytical batch. The choice of samples for performance of interference tests shall be conservative such that the sample displaying characteristics most likely to result in interference shall be selected for the procedure. No corrective action is specified by Method 6010A however the results of the interference tests shall be documented in the narrative if they result in out of control results. The specific corrective action described by Method 7000A shall be executed by the laboratory on failure of interference tests for 7000 series methods. The laboratory may propose alternatives to the standard procedures described in SW-846 for 7000 series methods such as post digestion spikes for all samples. In this case corrective action for failing post digestion spikes should be consistent with the corrective action described in Method 7000A. Alternatives to the standard procedures specified by Method 7000A that may be proposed by the Contract Laboratory must be approved by the USACE CO.

The QAPP shall detail matrix, method, and analyte- or compound-specific procedures and quality control criteria for all sample analyses used to determine Contract Laboratory accuracy. QC criteria for metals analyses shall conform with standards specified by SW-846 and CLP (matrix spike recovery, duplicate RPD). Quality control criteria are subject to

approval by the CO. Failure of the Contract Laboratory to present QC criteria (including corrective action) that are acceptable to USACE will result in directing of the Contractor by the CO to retain another laboratory for contract services.

5.2.3.4.1.3 Representativeness

A. Field Procedures. For field sample collection, it is the responsibility of the Contractor to conduct the sampling activities such that primary samples, blind QC duplicates, and QA splits are representative of field conditions. All duplicate samples (QA and QC splits) for nonvolatile analyses shall be homogenized in the field prior to packaging. Errors in sampling and packaging that result in non-representative samples or rejection of data by regulatory elements will result in direction by the CO to resample at the Contractor's expense. The Contractor is cautioned to interact closely with the Contract Laboratory during shipment of samples to insure that requirements for preservation have been met. All sample results associated with out of control preservation conditions will be rejected with re-sampling and reanalysis at the Contractor's expense. Alternatively, the USACE CO may determine that re-sampling will not be required. In this case the Government will receive credit for all costs of sampling and analytical work for sample results associated with out of control preservation conditions. Specific Contractor procedures to ensure representativeness shall be detailed in the QAPP.

B. Laboratory Procedures. Laboratory procedures must be established to ensure that aliquots used for sample analysis are representative of the whole sample. Similarly, any such procedures employed at the laboratory level shall not interfere with the concentration or composition of the analytes in the sample. All non-VOC samples shall be homogenized prior to extracting an aliquot for sample analysis. Contract Laboratory procedures to ensure representativeness shall be detailed in the QAPP.

5.2.3.4.1.4 Completeness

Completeness shall be evaluated qualitatively and quantitatively. The qualitative evaluation of completeness shall be determined as a function of all events contributing to the sampling event including items such as correct handling of chain of custody forms, etc. The quantitative description of completeness shall be defined as the percentage of Contract Laboratory controlled QC parameters that are acceptable.

QC parameters that shall be assessed for quantitative determinations of completeness shall include initial calibrations, continuing calibrations, surrogate percent recovery for organic analyses, analysis of laboratory duplicates for Relative Percent Difference (RPD), analysis of matrix spike/matrix spike duplicate analyses for percent recovery and RPD, and analysis of LCS for percent recovery, and holding times. The requirement for the quantitative assessment of completeness shall be 90%. The 90% standard shall be applied to the entire list of parameters described above such that a minimum of 90% of the data for each analytical method is associated with acceptable quality control criteria as described above and in other sections of this document. The quantitative assessment of completeness shall be calculated for each analytical method as the ratio of acceptable sample results to all sample results. For multi-compound or analyte methods (organic analyses) each analysis of the aggregate of compounds or analyte shall be considered a single sample result.

The requirement for holding times shall be 100%. If any sample exceeds the holding time specified by EPA SW-846 (or other guidance documents for other analyses) that sample shall be re-sampled and reanalyzed at the expense of the Contractor.

Data shall be screened for contract compliance by the CO. Failure of the analytical data to meet the standards for completeness will result in rejection of data with re-sampling and re-extraction/reanalysis performed at the expense of the Contractor. Completeness requirements shall be applied to data for each Quality Control Summary Report, however this requirement shall also be applicable to individual data packages associated with a single chain of custody.

Non-conforming data as a result of well-substantiated matrix effects shall not be considered in assessing Contractor

compliance with respect to completeness. In the event of significant occurrence of non-conforming data the Contractor will present a summary of data to substantiate an argument for matrix effects to the CO. This data will be reviewed by USACE. The USACE CO will determine the validity of an argument for matrix effects and instruct the Contractor as to the necessity of re-sampling/re-extraction/reanalysis.

5.2.3.4.1.5 Comparability

A. Contract Laboratory Procedures. The Contract Laboratory shall make the necessary provisions to ensure the comparability of all data. These procedures include, but are not limited to, the use of standard approved methodologies, the use of standard units and report format, the use of calculations as referenced in the methodology for quantitation, and the use of standard measures of accuracy and precision for quality control samples. All provisions to ensure data comparability shall be detailed in the QAPP.

B. Comparison to QA Lab Results. Analysis of QA samples by an independent laboratory is considered an instrument of contract administration by USACE. Significant deviations between QA lab and Contract Laboratory results will result in direction from the CO to the Contractor/Contract Laboratory to investigate the suspect data as detailed in Section 5.9. If investigation reveals errors in sampling or analytical procedures by the Contract Laboratory, re-sampling/re-extraction/reanalysis at the expense of the Contractor will be directed by the CO.

5.2.3.4.1.6 Sensitivity

The term sensitivity is used broadly here to describe the contract method detection, quantitation, and reporting limits established to meet the DQOs; and not limited to the definition which describes the capability of a method or instrument to discriminate between measurement responses. Several limits have been established to describe sensitivity requirements (i.e., IDL, MDL, SQL, PQL, CRDL, CRQL, etc.). Normally, instrument detection limits (IDLs), and method detection limits (MDLs) reported are typically based upon a reagent water matrix or purified solid and ignore sample matrix interferences and the resulting effects on the limits. For this reason, published MDLs or IDLs are presumably not achievable for environmental samples. The CRDLs and CRQLs published within CLP methodologies are contractually based levels and may have nothing to do with what is instrumentally possible. Because of these inconsistencies, and to promote the generation of comparable data, the definitions described below shall be used if not superseded by project-specific requirements. ***Contract requirements for sensitivity should be achievable for the batch QC samples within a reagent water/purified solid matrix (method blanks, LCSs) and compliance should be verified.***

5.2.3.4.1.7 Method Detection Limit. The method detection limit (MDL) shall be as defined in 40 CFR 136 App. B. The practical quantitation limit (PQL) or method quantitation limit (MQL) will be set as a multiple of the MDL. Subject to laboratory and instrument capability MDLs shall be consistent with those specified by EPA SW-846. PQLs for TPH by EPA Method 8015B (purgeable and extractable, LUFT manual, etc.) shall be consistent with the recommendations of the Tri-Regional Board Recommendations (1990) or those specified during the DQO process and local regulations. MDLs and PQLs for all analyses and all analytes shall be detailed in the QAPP in tabular format for soil and water matrices. MDLs presented in the QAPP should represent the laboratories most recent MDL studies. MDLs are instrument specific. For the purpose of providing documentation for the QAPP it will be acceptable to provide MDL representing the least sensitive instrument (highest MDLs). However, PQLs are subject to the approval of the USACE CO and the PQL in the final approved QAPP will be considered Contract Required Quantitation Limits (CRQLs).

The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The laboratory shall perform MDL studies during initial method setup and whenever the basic chemistry of the procedures are changed. Since it is not practical to establish an MDL for each specific matrix received at any given

laboratory, MDLs shall be estimated in an interference-free matrix, typically reagent water for water methods and a purified solid matrix (e.g., sand) for soil/sediment methods. Method detection limits shall be estimated for each target analyte using the procedures presented in 40 CFR, Part 136, Appendix B. The MDLs shall be extraction/digestion method-specific and shall include any clean-up methods used. To ensure that reasonable MDL values are determined, the laboratory shall analyze an MDL check sample by spiking an interference free matrix with all method target analytes at about two times the determined MDL and taking this sample through the same process used initially to establish the MDL values. If any of the target analytes are not recovered, then the MDL study shall be repeated for the failed target analytes. The laboratory established MDL values shall be verified quarterly by analyzing the MDL check sample. When multiple instruments are used for the same method, separate MDL studies may not be needed. However, laboratories must demonstrate equivalent sensitivity through the analysis of the MDL check sample. This check would also apply to confirmation columns. The acceptance criteria to be applied to this check sample are to verify that all target analytes are detectable. If any of the target analytes are not reliably detected, then the MDL study must be repeated. The determination of method detection limits in site-specific matrices may be required for certain projects. The analysis of the MDL check sample may not be required if the lowest calibration standard is significantly higher than the estimated MDL values.

5.2.3.4.1.8 Practical Quantitation Limit. The PQL is the lowest calibration standard and should be no lower than ten times the standard deviation as determined from the MDL study. For MDLs established using the seven replicates as defined in 40 CFR, the corresponding PQLs are approximately three times the MDLs. The highest PQL may be used when multiple instruments and sample preparation procedures are available in a laboratory. In the absence of project-specific requirements, the lowest calibration standard used for initial calibration shall be set at or between three to ten times the MDL (at or slightly above the PQL) for each target analyte. The low standard shall not be set at a value that is lower than the PQL. For projects where the action levels are significantly above the PQL or where high native concentrations are expected, the low standard could be set at a value greater than ten times the MDL. Analyte values reported below the PQL (low standard concentration) must be flagged as an estimated quantity (i.e., J flagged).

5.2.3.4.1.9 Method Reporting Limit. The method reporting limit (MRL) is a threshold value below which the laboratory reports a result as "<" the reporting limit value. If the reporting limit is elevated due to dilution or other analytical requirements, this will be noted on the associated sample analytical result page. It may be based on a project-specific reporting limit, a regulatory action level, or two times the laboratory's MDL. The definition of MRLs used the laboratory shall be declared in each data package. MRLs would be adjusted based on the sample matrix and any necessary sample dilutions. The highest value that can be reported for a MRL must be less than any project specified action levels or concentrations of concern. The lowest value that can be reported by a laboratory as a non-detect (or '<' value) shall be no lower than the value of the corresponding MDL check sample (2 times the MDL). This is the point where the laboratory has demonstrated their ability to reliably detect target analytes. However, the laboratory shall not claim to reliably quantitate values below the low standard. Therefore, analyte values reported between the MDL and PQL (low standard concentration) must be flagged as an estimated quantity (i.e., J flagged). If dilution to bring the reported concentration of a single compound of interest results in non detect values for any other analytes with detected concentrations in the initial analysis the results of the original run and the dilution will be reported with the appropriate notations in the narrative.

PQLs are subject to approval by the CO. Failure of the Contract Laboratory to present PQLs that are acceptable to USACE will result in directing of the Contractor by the CO to retain another laboratory for contract services. Failure of the Contract Laboratory to achieve PQLs/CRQLs specified in the QAPP will result in rejection of data with re-sampling/reanalysis at the Contractor's expense.

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 Contractor compliance with the requirements for sensitivity. A detailed analysis of all failures to meet requirements for

sensitivity will be included in the narrative section of the certificate of analysis detailed in Section 5.4.

5.2.3.5 Target Analyte Identification, Quantitation, and Confirmation.

5.2.3.5.1 Target Analyte Identification. Employ procedures presented within the individual determinative methods for determining presence and identification of target analytes within samples. ***For GC/MS analyses and any samples containing extraneous peaks not associated with the calibration standards, a scan against a mass spectral library (typically ~75,000 compounds) may be performed for the purposes of tentative identification if warranted by project DQOs***. Based upon the degree of match, evidence of similar pattern, and analyst professional judgement, compounds may be reported as Tentatively Identified Compounds (TICs) and the analytical values estimated. ***The necessity to perform this will depend on project specific requirements. Recommend the use of TIC searches only in the early stages of site characterization on samples speculated as contaminated. Significant detections identified through TIC searches, should require the inclusion of these compounds as project-specific target analytes. Future analyses shall require that calibration standards include these target analytes for more accurate quantitative determination of their result.***

For TPH analyses the laboratory shall use the following hydrocarbon ranges as default identification ranges: gasoline C6-C12, diesel C10-C28, motor oil C20-C36, and JP4 C8-C13. The QAPP shall define the specific ranges used for identification and quantitation for each fuel determined and reference any State requirements or methods used for analysis, as applicable. The QAPP must clearly define the hydrocarbon ranges and standards used for quantitation. Use of clean up procedures, analytical methods, and supplemental analyses must also be clearly described in the QAPP. The quality control and data verification procedures must be clearly defined in the QAPP for natural attenuation and bioremediation parameters. Interpretation of pattern matching for petroleum hydrocarbons and forensic geochemistry for hydrocarbon fuel fingerprinting and age dating must be established in the laboratory standard operating procedures.

Pattern matching includes fuel type, ownership, and age. Historical information (process history, blending trends, and legislative records) will be used as tools for pattern matching. Other analytical tools include infrared spectrometer, X-ray fluoresence spectrometer (used for metals determination in fresh or used lube oil), ICP, atomic absorption spectrometer, and thin lay chromatography. GC resolution for the components of the TPH is essential to accurate identification and quantitation of fuel type. The laboratory shall maintain a library of chromatograms for typical fuels including (but not limited to) kerosene, thinner (stoddard solvent), diesel fuel, bunker fuel or heavy fuel oil, motor oil, gasoline, aviation gasoline, and jet fuel. The laboratory shall also be capable of analysis for oxygenates (ethers-MTBE, TAME, DIPE, and ETBE and alcohols-methanol, ethanol, and TBA).

Forensic environmental geochemistry is defined as a scientific methodology developed for identifying petroleum-related and other potentially hazardous environmental contaminants and for determining their sources and time of release. As a result of physical process, hydrocarbon fuel in the subsurface environment is distributed among the following major phases: fuel vapors as a component of soil gas, fuel sorbed to soil particles, liquid fuel in pore spaces of the soil particles, liquid fuel floating on the groundwater table, and fuel dissolved in groundwater. The following analytical techniques may be used for forsenic characterization of petroleum products: gaseous hydrocarbons (C1-C5) by ASTM Method D2820, TPH-GRO by EPA Method 8015B, BTEX by EPA Method 8021, alkyl lead speciation and lead scavengers by modified EPA Method 8081, oxygenated blending agents (ethers and alcohols) by ASTM Method D4815, dye additives by thin layer chromatography, total lead, organic lead, and trace metals (especially vanadium and nickel) by EPA Method 6010B, simulated distillation by ASTM Method D2887, n-alkanes in the C8-C35 range, along with branched-chain alkanes by ASTM Method D3328, alkylbenzenes, alkylcyclohexanes, polynuclear aromatic hydrocarbons (PAHs), and polycyclic saturated hydrocarbons (steranes and terpanes-biomarkers) by EPA Method 8270, and stable isotope ratios for carbon (13C/12C), hydrogen (D/H), and sulfur (34S/32S) by dual collecting isotope ratio mass spectrometry.

5.2.3.5.2 Target Analyte Quantitation. All samples shall be quantitated using the initial calibration curve, following procedures outlined within the determinative methods. Sample results that exceed the range of the initial calibration high standard must be diluted and reanalyzed, and sample analyte values reported below the PQL must be flagged as estimated quantities (i.e., J-flag). All dilutions must be applied to the sample results and reported accordingly. Solid samples are to be determined on a dry-weight basis. Sample target analyte values should be reported to three significant figures.

5.2.3.5.2.1 Inorganic Analyses. Quantitative results are calculated using the mean value from the set of duplicate injections for Method 7000 or the mean value from multiple exposures for Method 6010. Also recommend the laboratory review the RPDs for duplicate injections/multiple exposures of samples exhibiting quantifiable concentrations. If the %RPD/% RSD is consistently > 20% and highly variable for concentrations greater than the low-level calibration standard, corrective action should be taken. When matrix interference is suspected/confirmed, the use of Method of Standard Additions (MSA) must be used to calculate the sample result. The laboratory shall at a minimum use a series of three standard additions containing 50%, 100%, and 150% of the expected concentration. As outlined within the method, plot the absorbance of each solution at the concentration of the known standards. The concentration of the sample is then obtained from extrapolating the resulting line back to zero absorbance.

5.2.3.5.2.2 Organic Analyses. The laboratory should make a reasonable attempt to correct for any matrix interference encountered. Dilutions should not be routinely used in preference to cleanup methods to address matrix interference. When matrix interference is present, samples should be processed using at least one clean up method as outlined by the determinative method. Refer to Section 6.8.2.2 for information on recommended cleanup methods. ***If the cleanup and reanalysis do not reduce the matrix interference, discuss the impact on the data within the case narrative.***

5.2.3.5.2.2.1 Method 8081. In general, multiple-component analytes are quantitated (via external calibrations) by comparing the areas (or heights) for the characteristic peaks to the areas (or heights) for the corresponding calibration peaks of the same retention time and shape. Quantitation may be performed using a number (i.e., three to five) major peaks or the total peak area of the appropriate pattern as described in the method. For Chlordane, quantitate the peaks of alpha-Chlordane, gamma-Chlordane, and Heptachlor separately against the initial 3-point calibration curves and report the individual results. When the GC pattern of the residue resembles that of technical Chlordane, quantitate for this. Since commercial BHC (which consists of a mixture of six chemically distinct isomers and one or more heptachlorocyclohexanes and octachlorocyclohexanes) may exhibit a wide variance in the percentage of the individual isomers present, quantitate and report the alpha, beta, gamma, and delta-BHC isomers separately. For DDT, the 4,4'-isomers of DDT, DDE and DDD are the predominate pesticides in the environment and are the isomers normally regulated by USEPA. Therefore quantitate separately and report the pure 4,4'-isomers of DDT.

5.2.3.5.2.2.2 Method 8290. The laboratory must comply with the specifications in the method. MS/MSD samples are required for this analysis.

5.2.3.5.2.2.3 Method 8330. Due to the lack of resolution between 2,4-DNT and 2,6-DNT, and between 2-Am-DNT and 4-Am-DNT, quantitation of these compounds may be expressed as 'isomeric pairs'.

5.2.3.5.3 Target Analyte Confirmation. Chromatography is a technique that relies upon the comparison of retention times between standards and unknown peaks for qualitative identification. Unless mass spectrometry is used as the detector, tentative identification is based solely on the retention time of an unknown peak falling within the prescribed retention time window of a known standard. ***In the absence of project-specific criteria, to minimize the possibility of incorrect identification (or false positives), confirmation shall be required for all***

chromatographic methods involving the analysis of single component target analytes. Quantitative confirmation of results above the PQL is required for samples analyzed by GC or HPLC and shall be completed within the method-required holding times. Confirmation may be required for multi-component analytes even though identification is primarily achieved through pattern recognition (i.e., PCBs, gasoline, etc.). When available, it is recommended that confirmation techniques involve the use of (1) another analytical technique (i.e., GC/MS), or (2) a second dissimilar column. When the laboratory is using the second dissimilar column, it shall be calibrated in the same manner as the primary column. After the target analyte has been identified, compare the primary and confirmatory results for agreement according to a method-prescribed criterion. Analytical results would normally be reported from the primary column unless interferences were noted. If quantitative results are reported from the confirmation column, the documentation from the analysis of all appropriate QC samples on the confirmation column shall also be required within the data package. Section 5.2.3.6.4.8 also provides requirements for confirmation analyses. Designation of which column is considered primary and which considered confirmation must be documented in the laboratory method-specific SOPs for each appropriate analysis. Once column designation has been established, the laboratory analysts will apply this designation consistently for all samples.

5.2.3.6 Contract Laboratory Internal Quality Control Checks.
The basic unit for application of laboratory quality control is the batch. Samples shall be prepared, analyzed, and reported in batches and be traceable to their respective batches. Batch sizes are normally limited to twenty field samples of a similar matrix but can exceed this by incorporating additional QC samples. Each batch shall be uniquely identified within the laboratory. Samples prepared together would normally be analyzed together on a single instrument. Samples taken from the same site would normally be grouped together for batching purposes within the constraints imposed by the method holding times. However, laboratories may find it necessary to group multiple clients samples into a single batch. Under these circumstances, additional batch QC samples may be needed that evaluate the effect of the matrix from each site on method performance. Field QC samples, i.e., trip blanks, rinsates, etc., shall not knowingly be used for batch QC purposes.

5.2.3.6.1 Preparation Batch. The preparation batch shall be defined as samples of the same or similar matrix that is prepared together by the same person, or group of people within the same time period or within limited continuous time periods, which follow the same method, using the same type of equipment and same lots of reagents. The laboratory shall have sufficient quantities of extraction / digestion labware to meet these requirements. Each preparation batch shall contain the requisite number and type of calibration solutions, blanks, quality control samples, and regular analytical samples as defined by the analytical method. These requirements shall be completely defined in the laboratory SOPs and are summarized in part in the following sections. The use of clean-up methods would be included as part of the preparation batch. All field and batch specific QC samples within the batch should be subjected to all preparatory and clean-up procedures employed.

5.2.3.6.2 Analysis Sequence. The analysis sequence or instrument run sequence shall be defined as samples that are analyzed together within the same time period or in continuous time periods on one instrument under the control of one continuing calibration verification. Analysis sequences would be bracketed by the appropriate continuing calibration verification standards and other QC samples as defined by the analytical method. In general, if an instrument is not used for periods of time or shut down (e.g., overnight, etc.), then a new analysis sequence shall be initiated. Each analysis sequence shall contain the requisite number and type of calibration solutions, quality control samples, and regular analytical samples as defined by the analytical method. These requirements shall be completely defined in the laboratories SOPs and are summarized in part in the following sections.

For samples that are purged and then analyzed immediately, the preparation batch and analysis sequences are combined. For this situation, the batch would normally be defined by the loading of samples into the various purge tubes. This definition has been interpreted differently however. For instance, the loading of purge tubes may be

performed all at one time, or may continue throughout the day. In order to ensure ambient environmental conditions throughout the potential loading process, USACE requires a minimum of an MB run every four (4) hours, or twice a day when samples are loaded throughout the day.

5.2.3.6.3 Analytical Batch. The analytical batch is defined as a preparation batch. The analytical batch will not exceed 20 samples and is defined as a set of samples that are extracted/analyzed concurrently or sequentially. Significant gaps (greater than two hours) in the analytical sequence will result in the termination of the previous sequence and the initiation of a new analytical sequence. The analytical batch shall be analyzed sequentially on a single instrument. The practice of "holding a batch open" and performing a single set of batch QC samples for all analyses performed during that period is unacceptable relative to the requirements of these specifications. Data reported by the Contract Laboratory that is found to be associated with batch QC samples that were not extracted concurrently or were not analyzed in the same sequence on the same instrument relative to the primary sample results shall be rejected. If the batch size is found to exceed 20 samples the data will be rejected. Once the analysis of a batch has started, all batch samples must be analyzed concurrently within 16 hours.

5.2.3.6.4 Batch QC Samples. The Contract Laboratory shall, as a minimum, analyze internal QC samples at the frequency specified by the method and in these specifications for all analytical methods. These QC samples for each analytical batch shall include method blanks (MB), MS/MSD analyses (laboratory duplicates and MS for inorganic analyses), and laboratory control samples (LCS). Definitions for the QC samples described are provided in Chapter One, Update I to EPA SW-846. The matrix used for LCS analyses shall be reagent grade water for aqueous analyses and reagent sand for soil/sediment matrices. Failure to include either matrix spikes or LCS samples with each analytical batch will result in credit to the government for one-third of the cost of the associated analyses. Failure to incorporate both a matrix spike and a LCS sample will result in rejection of data. Failure to incorporate a method blank with each analytical batch will result in rejection of data. A summary of the minimum required QC samples for each preparation batch are presented in the following subsections.. All calibrations and QC samples analyzed shall be uniquely identified and traceable to that unique sample preparation batch. Additional QC samples may be required for other batch types based upon project-DQOs.

A. QC Checks of Known Composition Samples. General batch QC may be viewed as those QC procedures applied to an interference-free matrix or a matrix of known composition (i.e., blanks, laboratory control samples, PE samples, standard reference materials (SRM), calibration verification standards, etc.). They ensure the analytical method is being performed in an in-control mode of operation. These QC checks provide no information on how well the method is performing with respect to the project sample matrix, however. Document clearly within the case narrative the QC checks that exceed method quality objectives along with corrective actions taken. ***It is recommended that contract nonpayment clauses be limited to QC sample results of interference-free or known composition matrices only. An example of a contract nonpayment clause which may be included within project contract documents is given below:***

> "The Contractor shall perform chemical analyses in accordance with the requirements established within the specified method and this document. When QC checks of an interference-free or known compositions do not meet these standards/requirements, corrective action must be taken through proper application of the inspection and services clause. Corrective action may include resampling, repreparation, and/or reanalyses of the affected samples at no additional cost to the government. If the Contractor fails to promptly perform the required corrective actions, or when the failure cannot be corrected by reperformance, the Government may reduce the contract price or fee payable under the contract to reflect the reduced value of services performed. Continued failure to perform chemical analyses in accordance with these standards/requirements may result in termination of the contract for default."

B. QC Checks of Matrix-Specific Samples. Matrix-specific (matrix-sensitive) QC procedures should be incorporated into the laboratory analysis to provide information on the precision and bias of the analyses on project samples. These procedures include analyses of field samples in association with surrogate compounds, matrix spikes (MS), matrix spike duplicates (MSD), or matrix duplicates (MD). *Matrix-specific procedures performed on other field samples at the laboratory not associated with the project samples are of limited value, for they do not provide information on the matrix under observation*. It should be noted that MS/MSD/MD analyses may require the submittal of an additional replicate sample to enable the laboratory to perform the requisite analysis. ***For this reason, the project requirements of minimum sample volumes necessary to accommodate the matrix-specific QC samples must be addressed very clearly within the SAP.***

Exceedances of method quality objective for these types of QC checks may be problematic due to matrix effect (signal enhancement or suppression) on the analysis and should not be viewed as an indicator of poor laboratory performance. For this reason, ***contract nonpayment clauses should not be associated with matrix-specific QC samples.*** However, the laboratory should not use this as an 'excuse' to avoid employing proper analytical techniques. The laboratory should make a reasonable effort to overcome matrix interference as noted below. Necessary corrective actions will vary depending on the type of interference, and are subject to analyst professional judgement. ***When these excursions indicate a potential for false negatives, lack of sensitivity, or an inability to accurately detect the target analytes, communication between the laboratory and data user should be pursued to identify alternatives available.*** For instance, procedures to decrease the matrix effect may include implementing cleanup procedures, dilution techniques, smaller sample size processed, etc. However, consequences to the data (i.e., higher detection limits, less representative aliquot, etc.) should also be assessed against project objectives.

5.2.3.6.4.1 Method Blank Samples. One method blank sample shall be analyzed for each analytical batch (one every 12 hours for GC/MS analyses). Contamination in method blanks (as well as reagent blanks, instrument blanks, extraction blanks for elutriations, initial calibration blanks, and continuing calibration blanks) above the MDL will not be allowed. Data found to be associated with blanks containing target analytes at or above the MDL may be rejected with re-sampling and/or re-extraction and reanalysis at the expense of the Contractor. The USACE will evaluate the data based on the level detected in the associated samples. Chronic systematic method blank contamination will not be accepted.

Method blanks are analyzed to assess background interference or contamination that exists in the analytical system that might lead to the reporting of elevated concentration levels or false positive data. The method blank is defined as an interference-free blank matrix similar to the sample matrix to which all reagents are added in the same volumes or proportions as used in sample preparation and carried through the complete sample preparation, cleanup, and determinative procedures. For aqueous analyses, analyte-free reagent water would typically be used. For soil analyses, a purified solid matrix (e.g., sand) would typically be used, except for metals analyses. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. Refer to Sections 5.2.3.11 and 5.2.3.8.1 for method quality objectives/corrective action scenarios for the method blank. Sample results shall not be corrected for blank contamination.

5.2.3.6.4.2 Laboratory Control Sample. The LCS is analyzed to assess general method performance by the ability of the laboratory to successfully recover the target analytes from a control matrix. The LCS is similar in composition to the method blank. For aqueous analyses use analyte-free reagent water. For soil analyses, a purified solid matrix (e.g., Ottawa sand, sodium sulfate, or other purified solid) would typically be used. However, due to the difficulty in obtaining a solid matrix, which is metals-free, analyte-free reagent water is taken through the appropriate digestion procedures for metals analyses. The LCS is spiked with all single-component target analytes (the complete target compound or analyte list) before it is carried through the preparation, cleanup, and determinative procedures. The laboratory will perform corrective action based on failure of any analyte in the spiking list. ***A subset of the***

(single-component) target analytes containing the specific analytes of interest can be substituted for the full list of target analytes if specified in project-specific contracts or work plans. When multi-component target analytes are reported, a separate LCS may be necessary if specified by project documents. For Method 8082, the LCS must be spiked with at least one PCB (e.g., 1016/1260 mixture), any project-specified PCBs, or all congeners to support the LCS evaluation. The use of solid standard reference materials (SRMs) as the LCS is discouraged for they do not typically include all target analytes, and the acceptance limits associated with them are wide -- due to the heterogeneity of the spiked matrix. Suggest instead the use of an interference-free matrix (e.g., purified solid, or sodium sulfate). When samples are not subjected to a separate preparatory procedure (i.e., purge and trap VOC analyses, or aqueous Hg analysis), the CCV may be used as the LCS, provided the CCV acceptance limits are used for evaluation. ***The spiking levels for the LCS would normally be set at the project-specific action limits assuming that the low standard used for the initial calibration was below this limit. If the low standard used was at this limit or if the site action levels were unknown, then the spiking levels would be set between the low and mid-level standards.*** The results of the LCS are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. Refer to Section 5.2.3.11 for method quality objectives/corrective action scenarios for the LCS. The laboratory shall also maintain control charts, or tables for these samples to monitor the precision and bias for the method as outlined in Section 5.2.3.8.1 and 5.2.3.11. The precision may be evaluated by comparing the results of the LCS from batch to batch, or by duplicate LCSs. Duplicate LCSs within the same batch are not required, but recommended by the USACE.

5.2.3.6.4.3 Matrix Spikes. The matrix spike (MS) is used to assess the performance of the method as applied to a particular project matrix. A MS is an environmental sample to which known concentrations of certain target analytes have been added before sample manipulation from the preparation, cleanup, and determinative procedures have been implemented. ***Reference project-specific documents for the contaminants of concern, guidance presented below, or the preparatory and determinative methods to determine target analytes to include within the MS spiking solution.*** If no information is available, the entire target analyte list will be spiked within the MS. The laboratory will perform corrective action based on failure of any analyte in the spiking list. The spike concentrations of the target analytes would normally be set at the same level as the LCS. ***If target analytes were known to be present in samples from a given site, then the spiking level should be adjusted to a concentration that is approximately two to four times the concentrations of the original target analytes.*** For solid samples, care should be taken to ensure that the original field sample is properly divided into homogeneous fractions when allowed by the method. ***Aqueous samples require the submittal of an additional sample for several chemical parameters, especially organic analyses. Therefore, the sample to be used for the MS should be based on project-specific DQOs and specified in the field to ensure that sufficient sample is available to perform the test.*** From the laboratory perspective, preparation batches require MS frequency at one per preparation batch. The merging of these MS frequencies is often difficult for the laboratory to implement. For instance, batches consisting of samples from multiple sites may require additional MSs to meet project requirements of evaluating the samples within the batch. For a MS from one site cannot be used to evaluate the matrix effects on samples from other sites. ***Projects must consider the method(s) employed, previous knowledge of the matrix, and other matrix-specific QC samples to help decide an appropriate frequency for MSs for a given project. As a consequence, a MS may not be included with each shipment of samples submitted to the laboratory. Communication between project and laboratory personnel is essential.*** The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis. Refer to Section 5.2.3.11 and 5.2.3.6 for method quality objectives/corrective action scenarios for the MS. ***When critical decisions are based on the MS sample recoveries, control charts could be maintained for these samples to monitor the bias of the method for each particular matrix***. Sample results shall not be corrected for MS QC excursions.

5.2.3.6.4.3.1 Method 6010. ***Unless superseded by project DQOs, it is not necessary to perform matrix spikes for Na, K, Ca, and Mg for aqueous samples; or Na, K, Ca, Mg, Fe, Mn, and Al for soil samples.*** The native concentrations of these low-toxicity metals are usually relatively high.

5.2.3.6.4.3.2 Method 8081. The MS should be prepared all single-component pesticides. ***Multi-component pesticides need not be included within the MS, unless required by project DQOs.***

5.2.3.6.4.4 Matrix Duplicates or Matrix Spike Duplicates. The matrix duplicate (MD) or matrix spike duplicate (MSD) is used to assess the performance of the method as applied to a particular matrix and to provide information on the homogeneity of the matrix. A MSD is a duplicate of the MS as previously described. A MD is an environmental sample that is either divided into two separate aliquots by the laboratory, or requires the submittal of an additional sample. When applicable, care should be taken to ensure that the sample is properly divided into homogeneous fractions. Both the MD and MSD are carried through the complete sample preparation, cleanup, and determinative procedures. ***The requirements for the frequency of MDs or MSDs would normally be specified in the project-specific DQOs.*** The normal use of these QC samples would follow the same requirements as described for the MS. ***In the absence of project-specific DQOs, a MD would normally be included with each preparation batch of samples processed where target analytes were expected to be present (e.g., inorganic methods). An MSD would normally be included with each preparation batch of samples processed where target analytes were not expected to be present (e.g., organic methods).*** The results of the MD or MSD are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the precision of the analysis. Refer to Section 5.2.3.9.3.4 and 5.2.4.1.2B for method quality objectives/corrective action scenarios for the MD or MSD. Control charts can be maintained for these samples to monitor the precision of the method for each particular matrix if required by the project.

5.2.3.6.4.5 Surrogates Standards. Surrogates are analyzed to assess the ability of the method to successfully recover these specific non-target compounds from an actual matrix. Surrogates are organic compounds that are similar to the compounds of interest in chemical behavior, but are not normally found in environmental samples. Surrogates to use are identified within the determinative methods. Other compounds may be chosen and used as surrogates, depending on the analysis requirements, whether they are representative of the compounds being analyzed, and whether they cover the chromatographic range of interest. These compounds should be spiked into all samples and accompanying QC samples requiring GC, LC, or GC/MS analysis prior to any sample manipulation. As a result, the surrogates are used in much the same way that MSs are used, but cannot replace the function of the MS. The results of the surrogates are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the individual sample determinations. Refer to Section 5.2.3.11 for method quality objectives/corrective action scenarios for surrogates. Control charts, or tables, shall be maintained for surrogates contained within the LCS or MB to monitor the accuracy of the method for each particular matrix. Sample results shall not be corrected for surrogate excursions.

Explosives analysis by Method 8330 is an exception, in that the surrogate used is actually a target compound. Care should be exercised by the laboratory with the choice of surrogate used, for the potential remains for co-elution with target analytes present within the samples. If 3,4-DNT is used as the surrogate, it must not co-elute with TNT. If it is not possible to obtain adequate resolution between 3,4-DNT and TNT, another surrogate should be chosen (e.g., 1,2-DNB).

5.2.3.6.4.6 Standard Reference Materials. The laboratory is encouraged to analyze additional natural matrix standard reference materials (SRMs) and participate in external performance evaluation (PE) programs. Sections 5.2.3.5.4 and 5.2.4.3.1 describe the requirements for PE samples.

5.2.3.6.4.7 Analysis Sequence QC Samples. Certain inorganic analyses (metals by ICP and GFAA) incorporate the following additional QC samples to assess method performance without the influence of the preparatory procedures.

5.2.3.6.4.7.1 Post Digestion Spikes (PDS). PDSs are incorporated into an analytical sequence to assess matrix effects based upon (1) the occurrence of new and unusual matrices included within the batch, or (2) contingency analysis based upon serial dilution (SD) or matrix spike (MS) failures. Duplicate injections of each environmental sample may be avoided if a post-digestion spike (PDS) is performed for each sample. PDSs are prepared by the addition of the primary source standard to the digestate for the same metals and at approximately the same concentration as is used for the MS. Refer to Section 5.2.3.11 for method quality objectives / corrective action scenarios for PDSs.

5.2.3.6.4.7.2 Serial Dilutions (SD). A 5X (1:4) serial dilution test may be performed for an analyte to evaluate matrix interference if the analyte concentration in the original (undiluted) sample is at least 50 times the MDL. SD - Matrix effects are suspected if the RPD between the undiluted and diluted result > 10%. If this criterion is not met, further confirmation of the interference via implementation of PDS is necessary when matrix interference is suspected, and the calculation of the result through the use of MSA when matrix interference is suspected/confirmed.

NOTE: When serial dilutions are used to address matrix interference, only "best" diluted results (i.e., the lowest dilution which yielded acceptable results) need be reported. However, the reported result must be qualified (i.e., D-flag) and the dilution factor specified. The associated PQLs or MRLs must also be adjusted based on the dilution factor.

5.2.3.6.4.8 Second Column Confirmation. Second column confirmation for all GC sample analyses involving identification of discrete peaks with detected concentrations will be required at no additional charge to the government. Second column confirmation is not required for concentrations reported between the MDL and the PQL. Section 5.2.4.1.6.4.2 presents more detailed information regarding compound confirmation requirements.

5.2.3.7 Instrument Calibration and Frequency

The text shall identify all tools, gauges, instruments, and other sampling, measuring, and test equipment used for data collection activities affecting quality that must be controlled and, at specified periods, calibrated to maintain accuracy within specified limits. The text shall discuss how calibration shall be conducted using certified equipment and/or standards with known valid relationships to nationally recognized performance standards. If no such nationally recognized standards exist, document the basis for the calibration. Identify the certified equipment and/or standards used for calibration. Indicate how documentation of calibration shall be maintained and be traceable to the instrument for both field and laboratory procedures. A list of field and laboratory instrumentation (include details on manufacturer, models, accessories, etc.) procedures used for calibration and frequency of checks shall be required in the QAPP. The instrumentation and calibrations shall be consistent with the requirements of the contract and EPA-approved analytical method requirements. For laboratory methods the text shall present for each analytical method a tabular summary describing calibration procedures, the acceptance standard, and the required corrective action on failure. Field instruments shall be calibrated daily or immediately before use. Failure to meet method or contract specified requirements for initial or continuing calibrations will result in rejection of data. Once a compound has been designated as linear or nonlinear by the laboratory analyst, the designation must be specified in the method SOP. All analysts must apply this standard consistently for the analyses.

Additional information regarding calibration requirements are provided in Section 4.9.2.2.

5.2.3.7.1 GC Analyses. For GC analyses the initial response factors for all calibration levels shall exhibit a relative standard deviation less than or equal to 20%. Additionally, a continuing calibration check must be performed at the beginning of a run of analyses and again after every ten analyses or at the end of the run if the run consists of fewer than ten analyses. (Please note that this requirement applies to every ten analyses not to include initial/continuing calibration checks or instrument blanks.) All analytes in the calibration check samples (ICV and CCV) must meet a

standard of +/- 15% from the initial calibration. All samples must be bracketed by passing calibration check samples. Continuing calibration check samples will be compared to the response from the initial calibration and not to the response from the first calibration check of the day and must meet a standard of +/- 15%. Failure to bracket all samples with acceptable calibration checks will result in the reanalysis of affected samples. Analytical results not bracketed with passing calibration checks for all compounds will be rejected with re-sampling and reanalysis at the expense of the Contractor.

5.2.3.7.2 GC/MS Analyses. The initial calibration requirements for GC/MS analyses are well described by the methods in SW-846. Initial calibration requirements for GC/MS methods must be met for all CCCs and SPCCs. Data found to be associated with out of control calibrations will be rejected. Quantitation for GC/MS analyses (VOCs and semivolatiles) will be performed using the average response factor from the initial calibration. The continuing calibration check will only be used for comparison to the initial calibration curve. Sample results quantitated from the continuing calibration check will be rejected. Failure to bracket all samples with acceptable calibration checks will result in the reanalysis of affected samples.

5.2.3.7.3 Inorganic and General Chemistry Analyses. Calibration procedures for ICP analyses shall follow the specific protocols described in SW-846 (Update I) Method 6010A. Calibration procedures for Method 7000 series analyses shall conform to the requirements of Method 7000A with the exception that both initial and continuing calibration checks shall meet a standard of +/- 10%. Initial calibrations for inorganic and general chemistry analyses must demonstrate a correlation coefficient of 0.995. The analytical run sequence for general chemistry analyses shall generally follow the same procedures as metals analyses with an initial calibration check followed by a continuing calibration blank and continuing calibration checks. All sample results must be bracketed by passing continuing calibration checks. Data found not to be in conformance with these standards will be rejected.

5.2.3.8 Quality Control Requirements

For laboratory specific procedures the text shall present for each analytical method and matrix detailed tabular summaries of all controlled QC parameters, the acceptance limit, and the appropriate corrective action to be followed on exceedance of control limits. The text shall describe the procedures to be used to calculate each of the QC statistics, including the QC checks described in the preceding paragraph as well as precision and accuracy (bias). Copies of the formulas are acceptable as long as the accompanying narrative or explanation specifies clearly how the calculations will address difficult situations such as missing data values and "less than" or "greater than" values. The text will also include a description of completeness requirements as described in Section 5.2.3.4.1.4.

5.2.3.8.1 Control Limits and Acceptance Criteria. Appropriate mechanisms, including the definition of laboratory control limits for each of these elements, shall be established to ensure that control is maintained. A specific system detailing the protocols to be followed (corrective action) in the event that any internal QC check sample does not meet laboratory acceptance criteria shall be implemented. The QAPP shall detail specific corrective action for each controlled QC parameter (i.e., failure of surrogate recoveries for sample analyses, failure of method blanks, failure of MS/MSD or LCS for recoveries or RPD) for each analytical method and matrix. This system shall include the mechanism by which corrective action taken in the event of any non-conformance event is documented and assurance provided that the system in question remains in control. The details of this system shall be described in the QAPP.

Quality control criteria (including corrective action) are subject to approval by the CO. Failure of the Contract Laboratory to present QC criteria that are acceptable to USACE will result in directing of the Contractor by the CO to retain another laboratory for contract services.

5.2.3.8.2 Preventative Maintenance. A preventive maintenance program for all facilities and instrumentation used by the Contractor for sampling and analyses shall be presented in the QAPP. Specific details of preventive maintenance for analytical methods used for this project shall be included in the QAPP.

5.2.3.8.3 Record-Keeping. The Contract Laboratory shall maintain a bound logbook for each analytical instrument. This book serves as a permanent record documenting any routine preventive maintenance performed, as well as any service performed by external individuals such as manufacturer's service representatives. In any case, any maintenance activities must be performed by individuals qualified to perform the particular task involved. All records shall be made available for the CO's inspection upon request.

5.2.3.8.4 Performance Evaluation Samples. In addition to any performance audit samples submitted by the CO during this project, the Contract Laboratory shall be a participant in performance evaluation programs offered through agencies such as the EPA on the federal level, and other such programs offered or mandated at the state level. Performance evaluation program participation shall be detailed in the QAPP. Additionally, for each delivery order at the option of the USACE CO, blind PE samples for each matrix and analysis (PE sample to be provided by USACE) may be inserted into the sample stream. The Contractor will be required to furnish sample containers identical to those used for the field samples for this activity.

PESs are used to assess routine performance levels of laboratories. PES will be used in routine QA oversight as well as in the investigation of laboratory fraud. The use of PES sends a message to a laboratory that the client wants to assess the performance of the laboratory. The strongest message will be communicated through the use of double blind PES. The value of the incorporation of PES in the sampling effort will be determined based on DQOs during the planning stages of the project. Use of PES will be designated in the SAP based on DQOs.

A double blind PES is a sample submitted to evaluate the performance of a laboratory to perform analysis on a sample of known concentration and identity (i.e., known only to the parties submitting the PES to the laboratory). The concentration and identity of the double blind PES will not be known by the laboratory. Double blind PES labeling, packaging and chemical composition will mimic those of the routine samples to mask the identity of the sample to the laboratory. Double blind PES submitted concurrently with site samples are useful in increasing the overall level of confidence in the defensibility of data when the results submitted by laboratories fall within acceptance ranges.

The PES supplier will have a documented quality system, such as that required by ISO 9001 or equivalent. If appropriate PES is not commercially available, prepared PES will be validated with a reliable reference laboratory. Successful completion of a PES can build confidence in the use of a particular laboratory. Continuing success assures the data users of the reliability of the laboratory. Conversely, a laboratory's repeated failure with more than one contaminant and with more than one type of PES, brings into question the reliability of the laboratory. Repeated poor PES results may be a good reason for awarding the analytical contact to another laboratory when the existing contract ends.

5.2.3.9 Data Acquisition Requirements (Non-Direct Measurements)
Identify the type of data acquired from non-measurement sources such as computer data bases, spreadsheets, and programs, and literature files. Define acceptance criteria for the use of the data in this project. Discuss any limitations on the use of the data based on uncertainty in the quality of the data and discuss the nature of that uncertainty.

5.2.3.10 Laboratory Quality Control Procedures
Laboratory overall method performance shall be monitored by the inclusion of various internal quality control checks which allow an evaluation of method control (batch QC), and the effect of the sample matrix on the data being generated (matrix-specific QC). Batch QC is based on the analysis of a laboratory control sample to generate accuracy (precision and bias) data and method blank data to assess the potential for cross contamination. Matrix-specific QC shall be based on the use of an actual environmental sample for precision and bias determinations from the analysis of matrix spikes, matrix spike duplicates, matrix duplicates, and surrogate spikes, etc. Site-specific PE

samples could also be used, if available. The overall quality objectives are to implement procedures for laboratory analysis and reporting of data that are indicative of the degree of quality consistent with their intended use. ***Method quality objectives, given as QC sample acceptance limits and ranges may be default values established within this guidance, or may be based upon project DQOs.*** Laboratory generated control ranges are also used for an internal evaluation of method performance and control. ***Variances from any of these target ranges, would result in the implementation of appropriate corrective measures and an assessment of the impact on the usability of the data in the decision making process.***

5.2.3.11 Method Quality Objectives and Corrective Actions

When errors, deficiencies, or out-of-control situations exist, the laboratory's QA program shall include a system of QC activities that measure the system performance to verify that they meet stated requirements and objectives. When the analytical system performance does not meet defined standards, the laboratory shall employ systematic procedures, called 'corrective actions', to resolve problems and restore proper functioning to the analytical system(s). Laboratory personnel are alerted that corrective actions are necessary when: (1) QC data are outside the method quality objectives for precision and bias; (2) blanks or laboratory control samples contain contaminants above acceptable levels; (3) undesirable trends are detected in spike recoveries or RPD between duplicates; (4) there are unusual changes in method detection limits; (5) deficiencies are detected by the QA department during internal or external audits or from the results of PE samples; or (6) inquiries concerning data quality are received from a project manager. Corrective actions are often handled at the bench level by the analyst, who reviews the sample preparation procedures for possible errors, checks the instrument calibration, spike, and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager, or QA department for further investigation. ***Poor performance by the laboratory may result in payment penalties or work being repeated at the contractor's expense. Once resolved, full documentation of the corrective action procedure shall be filed with the project-specific records.*** The following identifies method quality objectives and the corrective actions necessary. When qualification of data is necessary (e.g., flagging), refer to Section 5.2.5 for details on flagging conventions. The following shall be required in the absence of project-specific requirements:

5.2.3.11.1 Incoming Samples. Problems noted during sample receipt shall be documented on an appropriate form (the 'Cooler Receipt Form'). ***The project manager or appropriate technical personnel, shall be contacted immediately for problem resolution. Minor temperature nonconformances may be allowed for samples transported short distances (less than 4 hours) from the project site to the laboratory. These criteria will be stipulated in the project QAPP.***

5.2.3.11.1.1 Sample Holding Times. ***If samples cannot be prepared or analyzed within the method required holding times, the project manager or appropriate technical personnel, shall be immediately notified, such that an appropriate corrective action plan can be generated. If holding times are exceeded and results reported, the resulting data shall be flagged, and a discussion of the impact included within the case narrative.***

5.2.3.11.2 Instrument Calibration. Sample analysis shall not be allowed until all initial calibrations, initial calibration verifications, and instrument blanks meet the appropriate requirements. All continuing calibration verifications that do not meet method requirements shall result in a review of the calibration, rerun of the appropriate calibration standard for the failed analytes, and, if necessary, reanalysis of all samples affected back to the previous acceptable continuing calibration verification check for the target analytes that failed. Continued failure of the CCV shall result in the construction of a new initial calibration curve followed by the reanalysis of all samples affected. ***If results are reported when a calibration criterion has been exceeded, then all results reported shall be flagged, and a discussion of the impact included within the case narrative.*** Instrument blanks should be implemented as outlined in the prescribed method.

5.2.3.11.3 Method QC Samples. Each preparatory batch and analysis sequence must include the appropriate batch and matrix-specific QC samples and standards: i.e., method blanks, laboratory control samples, matrix spikes, matrix duplicates, matrix spike duplicates, surrogate spikes, and other method specified QC. ***All QC shall meet the appropriate project-specific method quality objectives and associated corrective actions.*** In the absence of such criteria or actions, the corrective actions as described below shall be required. Failure of method QC shall result in the review of all affected data. If no errors can be noted, the affected sample(s) may need to be reanalyzed or re-prepared and reanalyzed within method holding times, if possible. ***All re-preparation and reanalysis necessary due to method failure shall be performed at no cost to the government. If the situation is not corrected, and results reported, then the corresponding data shall be flagged, and a discussion of the impact included within the case narrative. The project manager or appropriate technical personnel, shall be notified as soon as possible to discuss possible corrective actions should unusually difficult sample matrices are encountered.***

5.2.3.11.3.1 Method Blanks. The following criteria shall be used to evaluate the acceptability of the method blank data if project DQOs do not specify otherwise: The concentration of all target analytes shall be below the MDL check sample (approximately two times MDL) concentration for each target analyte, or less than 5 percent of the sample result for the same analyte, whichever is greater for the MB to be acceptable. When this criterion is exceeded, corrective action should be taken to find/reduce/eliminate the source of this contamination in the method blank. However, sample corrective action may be limited to qualification for blank contamination (i.e., B-flag). When the concentrations of any target analytes within the MB are above the MDL check sample for the majority of target analytes or above the PQL for target analytes known to be common laboratory contaminants, assess the effect this may have had on the samples. If an analyte is found only in the method blank, but not in any batch samples, any further corrective action may not be necessary. Steps shall be taken to find/reduce/eliminate the source of this contamination in the method blank. The case narrative should also discuss the situation. If an analyte is found in the method blank and some, or all, of the other batch samples, additional corrective action is required to reanalyze the method blank, and any samples containing the same contaminant. If the contamination remains, the contaminated samples of the batch would be re-prepared and reanalyzed with a new method blank and batch specific QC samples. Sporadic cases of contamination may be difficult to control, however, daily contamination would not be acceptable.

5.2.3.11.3.2 Laboratory Control Samples. ***The LCS is evaluated by comparing the percent recovery for all of the target analytes to the recovery method quality objectives as determined by project-specific DQOs, or the default ranges established in this guidance.*** If target analytes are outside the acceptance windows, corrective action is required. Project DQOS will dictate the corrective actions necessary. Initially, the effect the QC failure has on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it. The case narrative shall discuss the corrective action taken and any other information. Typically, the LCS would be reanalyzed for the failed analytes only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be re-prepared and reanalyzed for the failed analytes only. ***If sufficient sample is not available for re-preparation and reanalysis or if the corrective action is ineffective, the sample results reported within that batch shall be flagged accordingly (R-flag), and a discussion of the impact included within the case narrative.*** When there are multiple (>5) target analytes reported, the acceptance criteria may allow for the sporadic marginal failure of a few target analytes included within the LCS without requiring reanalysis of the entire batch. Reference Section 4.9.3 and Tables 4-7 through 4-14 for information on the number of sporadic failures allowed and the method-specific marginally-expanded acceptance criteria to be applied.

5.2.3.11.3.3 Matrix Spike Samples. ***The MS is evaluated by comparing the recovery for target analytes to the recovery windows established within project documents, or those established in Tables 4-7 through 4-14.*** MS data evaluation is more complex than method blank or LCS data evaluation since MSs measure matrix effects in addition to sample preparation and analysis errors. The heterogeneity of soil, grab samples, and sequentially collected water samples further complicate the evaluation since matrix-specific bias assumes that the native

concentrations in the duplicate analyses are constant. In addition concentrations of the target analytes in the sample can also far exceed the spike amounts added, lending the resulting recoveries invalid. MSs that fail to meet the appropriate acceptance criteria would indicate that a potential matrix effect is present. 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 not be significant, making the bias measures unrepresentative of the true method and matrix performance. ***For this reason, if the native concentration is two or more times the spiking level, corrective actions would be based on project DQOS.*** Regardless, steps should be taken to find the cause failure and corrective actions taken to remedy it. If possible, respike the sample as outlined below at a higher level (e.g., at two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. A review of the MSD result, if available, may confirm the matrix effect, if it is the same direction and same order of magnitude. If the native concentration is low, and the MS/MSD recoveries confirm matrix interference, reanalyze the MS/MSD sample/extract after employing cleanup procedures (organic analyses) or dilution techniques to minimize matrix interference. ***If the matrix effect cannot be resolved, discuss the impact on the data within the case narrative.***

5.2.3.11.3.3.1 Inorganic Analyses. Corrective action for unacceptable MS recoveries for ICP and GFAA analyses shall include implementation of a PDS from the same sample that the MS was prepared. In that way, information is obtained to identify whether matrix interference is occurring during the digestion or analytical procedures. Refer to Section X for guidance on the evaluation of MS in conjunction with the PDS.

5.2.3.11.3.3.2 Organic Analyses. When there are multiple (>5) target analytes reported, the acceptance criteria may allow for the sporadic marginal failure of a few target analytes included within the MS without requiring reanalysis. When only a subset of target analytes is included in the MS, allow only one (1) sporadic marginal failure. Reference Section 4.9.3 and Tables 4-7 through 4-14 for information on the number of sporadic failures allowed and the expanded acceptance criteria to be applied.

5.2.3.11.3.4 Matrix Duplicate and Matrix Spike Duplicate Samples. The MSD is evaluated using the same bias criteria as described for the MS. ***The MD or MSD is evaluated by comparing the precision for all target analytes to the windows as determined by project-specific DQOs, or as stated herein.*** These criteria should only be applied to concentrations of target analytes that are above each analyte's PQL. MDs or MSDs that fail to meet the appropriate acceptance criteria would indicate that a potential matrix effect is present. Corrective actions shall be performed as described for the MS.

5.2.3.11.3.5 Surrogates Standards. ***A surrogate is evaluated by comparing its recovery in each sample to the windows as determined by project-specific DQOs, or as stated within Tables 4-9 through 4-14***. Surrogate spikes in matrix-specific samples that fail to meet the appropriate acceptance criteria would indicate that a potential matrix effect is present. If significant non-target interference occurs, corrective action shall include implementing additional cleanup procedures, and re-analyses. ***If this does not reduce the interference, discuss the impact on the data within the case narrative. Recommendations to the client may include method modifications, such as re-preparation and reanalysis with smaller sample aliquots to reduce the effects of the matrix.*** The consequences to detection limits must also be considered in this instance. Surrogate failures in method blanks or laboratory control samples are indicative of a general method failure and should be thoroughly investigated as noted in Sections 5.2.3.9.3.5 and 5.2.4.1.2C, respectively.

5.2.3.11.3.6 Post Digestion Spike Samples. Default recovery control limits for the PDS is noted on Tables 4-7 through 4-8. Similar to the MS, if historic data or information on native sample concentrations is available, the MS or PDS should be spiked at a concentration at least twice the native sample concentration for the following evaluation to be considered valid. Professional judgement should be used to determine the corrective action necessary when the MS recovery for as analyte fails but the PDS recovery passes. ***For instance, when the MS***

recovery fails because it falls below the lower control limit but the PDS recovery passes, confirmatory re-digestion and reanalysis may not be required if allowed by project DQOs. When both the MS and PDS indicate matrix interference is present, the laboratory must attempt to correct for the interference by the use of method of standard additions, an internal standard technique for ICP (e.g., with yttrium), use of a different matrix modifier for GFAA, or different digestion or analytical procedure to achieve a representative result, before qualifying the sample for matrix interference. This does not apply to sporadic failures but rather to target analytes exhibiting out of control recoveries on consecutive batches. Also, verify overall batch control for the analysis by evaluation of the LCS.

5.2.3.11.4 Calculation Errors. Reports shall be reissued if calculation or reporting errors are noted with any given data package. The case narrative shall clearly state the reason(s) for re-issuance of the report.

5.2.3.11.5 On-site Audits. A corrective actions report shall be required that addresses any deficiencies noted during audits conducted. ***If corrective actions are needed for major deficiencies that would affect data quality, the laboratory should notify the USACE of other projects that may be affected.***

5.2.4 Assessment and Oversight

The following sections describe the assessment and oversight requirements for USACE HTRW projects.

5.2.4.1 Contractor Quality Control. The text shall address the responsibilities of all project personnel as they relate to the quality management function and describe the integration of the corporate quality assurance program into the execution of quality control operations for project asks related to sampling and analysis. Key personnel must be identified along with their function and qualifications. The text shall address specific Contractor procedures for control of the quality of work of subcontractors utilized for drilling, well installation, geophysics, etc. In particular, the text should address Contractor control mechanisms in relation to the quality of work performed by the Contract Laboratory. The text shall acknowledge and describe implementation of the three-phase control system for all aspects of the work specified as applicable to sampling and analysis. (Appendix H of EM 200-1-3 contains a discussion of the implementation of the three-phase control system for environmental sampling and analytical projects.) The discussion of Quality Control in the SAP should focus on field procedures while the discussion presented in the QAPP should focus on field, laboratory, and general CQC. The QAPP shall describe QC procedures that should be associated with each sampling, analysis, or measurement technique. At a minimum for field procedures the text shall include the checklists detailing quality control procedures associated with the three-phase control system. Applicable checklists for laboratory and field audit procedures shall be incorporated directly into the SAP in Contractor QC section. Examples of this type of checklist can be found in Appendix H of EM 200-1-3. If the discussion of CQC in the CDQMP references sections presented in the general CQC Plan the relevance to activities related to sampling and analysis presented in the general plan must be clear and unambiguous. The sections describing Contractor Quality Control (CQC) procedures shall address the following topics:

A description of the quality control organization including acknowledgment that the CQC staff shall implement the three phase control system for all aspects of the work specified. The staff shall include a Program Chemist who shall report to the overall contract manager, Program Contractor Quality Control Systems Manager, or someone higher in the Contractor's organization. Contract manager in this context shall mean the individual with responsibility for the overall management of this contract including quality and production. Delivery order specific Sampling and Analysis Plans should include the designation of the Project Quality Control Systems Manager, Project Chemist, and all other personnel involved in the quality control organization as implemented for work on specific delivery orders.

Note: In aspects of work related to sampling the Project Chemist shall have equal responsibilities for the quality assurance function relative to the Project QC Systems Manager. In aspects of work related to sample analyses the Project Chemist shall have lead responsibility for the quality assurance function. The Program Chemist shall provide quality assurance oversight on the work of the Project Chemist as well as having overall responsibility for implementation of the chemical data quality management program for this contract.

The name, qualifications, duties, responsibilities, and authorities of each person assigned a CQC function related to sampling and analysis. The text shall include resumes for all non-laboratory Contractor personnel to include the Contractor Program Manager, Contractor Project Manager, Contractor Quality Control Systems Manager, Contractor Program Chemist, Contractor Project Chemist, and Contractor Technical Professionals directly involved in execution of work for this project. If staff changes are necessary during the execution of this work resumes shall be submitted for new personnel as well as a description of their responsibilities in a technical memorandum to the USACE CO. Changes in the responsibilities of existing staff (if any) will also be described in technical memoranda prepared for this purpose.

A copy of the letter to the Program Chemist for activities related to sampling and analysis signed by an authorized official of the firm which describes the responsibilities and delegates sufficient authorities to adequately perform the functions of the Program Chemist, including authority to stop work related to sampling and analysis which is not in compliance with the contract will be included in the text of the CDQMP. For specific delivery orders the Program Chemist shall issue letters of direction to the Project Chemist(s), including authority to stop work related to sampling and analysis which is not in compliance with the contract. Copies of these letters shall be included in the CDQMP and SAP as applicable.

Procedures for scheduling, reviewing, certifying, and managing submittals. Submittals in this context refers to all project specific work plans, sampling and analysis plans, final investigation reports, data submittals, quality control summary reports, etc. The text of the CDQMP shall describe the organization and documentation required by the Contractors internal quality control review process. At any time the USACE CO may request copies of documentation (internal review comments as well as the review ladder) of the Contractors internal quality control review process for project specific submittals.

Procedures for tracking preparatory, initial, and follow-up control phases and control verification.

Procedures for tracking field and laboratory deficiencies from identification through acceptable corrective action. These procedures will establish verification that identified deficiencies have been corrected.

A list of the definable features of work shall be provided. A definable feature of work is a task, which is separate and distinct from other tasks and has separate control requirements. It could be identified by different trades or disciplines, or it could be work by the same trade in a different environment. The three-phase quality control system shall be implemented for each definable feature of work.

5.2.4.2 Three-Phase Quality Control. The three phase control system, and all attendant reports will be implemented by the Contractor and by major sub-contractors including the Contract Laboratory. Minutes of preparatory, initial, and follow up inspections and meetings held at the Contract Laboratory will be delivered to the USACE CO as well as minutes of meetings held at field sites. The minutes of initial, preparatory, and follow up inspections will be signed by all participating personnel. The Project Chemist and other Contractor personnel may participate in meetings held at the Contract Laboratory by teleconferencing. The Project Chemist is required to participate in preparatory and initial meetings at the Contract Laboratory. Follow-up inspections may be conducted by Contract Laboratory personnel with involvement of Contractor personnel as required. This requirement will be acknowledged in the QAPP and in the SAP.

5.2.4.2.1 Preparatory Phase. This phase shall be performed prior to beginning work on each definable feature of work and shall include:

a. A review of each paragraph of applicable specifications from the contract specifications, FSP, QAPP, and SAP.
b. A review of the site diagrams detailing locations where samples are expected to be obtained.
c. A check to assure that all materials and/or equipment are acceptable for use.

d. A check to assure that provisions have been made to provide required control inspection and testing.
e. Examination of the work area to assure that any required preliminary work has been completed and is in compliance with the SOW.
f. A review of the appropriate activity hazard analysis or Site Specific Health and Safety Plan to assure safety requirements are met.
g. Discussion of procedures for execution of work including repetitive deficiencies. Document performance standards for that phase of work.
h. The Government shall be notified at least 72 hours in advance of beginning any of the required action of the preparatory phase. This phase shall include a meeting conducted by the CQC System Manager and attended by the Project Chemist, Project Manager, and other CQC personnel (as applicable). The results of the preparatory phase actions shall be documented by separate minutes prepared by the QC System Manager and attached to the Daily Quality Control Report. The Contractor shall instruct applicable workers as to the acceptable level of performance required in order to meet the requirements of the contract specifications.

These requirements will be detailed in the QAPP and in the SAP in Section 5.4.11, as applicable to sampling and analysis operations. This information must be reviewed by each new field crew on site during execution of the work.

5.2.4.2.2 Initial Phase. This phase shall be accomplished at the beginning of a definable feature of work. The following shall be accomplished:

a. A check of preliminary work to ensure that it is in compliance with SOW, FSP, and QAPP requirements. Review minutes of the preparatory meeting.
b. Verification of full contract compliance. Verify required control inspection and testing.
c. Establish levels of performance and verify compliance with minimum acceptable performance standards.
d. Resolve all differences.
e. Check safety to include compliance with and upgrading of the safety plan and activity hazard analysis. Review the activity analysis with each worker.
f. The Government shall be notified at least 72 hours in advance of beginning any of the required action of the initial phase. This phase shall include a meeting conducted by the CQC System Manager and attended by the Project Chemist, Project Manager, and other CQC personnel (as applicable). The results of the initial phase actions shall be documented by separate minutes prepared by the QC System Manager and attached to the Daily Quality Control Report. The Contractor shall instruct applicable workers as to the acceptable level of performance required in order to meet the requirements of the contract specifications.
g. The initial phase should be repeated for each new crew to work on-site, any time acceptable specified quality standards are not being met, or when modifications to the SOW impact existing Contractor procedures.

These requirements will be detailed in the QAPP and in the SAP in Section 5.4.11 as applicable to sampling and analysis operations.

5.2.4.2.3 Follow-up Phase. Daily checks shall be performed to assure continuing compliance with contract requirements until completion of the particular feature of work. The checks shall be made a matter of record in the CQC documentation. Final follow-up checks shall be conducted and all deficiencies corrected prior to the start of additional features of work, which may be affected by the deficient work.

These requirements will be detailed in the QAPP and in the SAP in Section 5.4.11 as applicable to sampling and analysis

operations.

5.2.4.2.4 Additional Preparatory and Initial Phases. As determined by the Government, additional preparatory and initial phases may be required at no additional expense to the government on the same definable features of work if the quality of on-going work is unacceptable, if there are changes in the applicable CQC staff, on-site supervision or work crew, if work on a definable feature is resumed after a substantial period of inactivity, or if other problems develop.

These requirements will be detailed in the QAPP and in the SAP in Section 5.4.11, Contractor Quality Control, as applicable to sampling and analysis operations.

5.2.4.3 Monitoring Laboratory Performance Over time Using Follow-up Audits

After passing the pre-award on-site audit the project laboratories must be managed by the Primary Contractor. If laboratory work is subcontracted by the primary contractor for the project, the Contract Laboratory subcontract specifications will be developed that explicitly include the execution of primary contractor quality control oversight activities.

If the pre-award on-site audit revealed significant laboratory deficiencies, follow-up audits will be performed at the discretion of USACE and the Primary Contractor to ensure that corrective measures have taken place to sufficiently address the deficiencies and to ensure data quality requirements are being met. Follow-up audits will focus heavily on project specific data. They will incorporate the review and tracking of raw data from the original measurements through the generation of a final report. Audits normally will require some regeneration of raw data from electronic files to verify the integrity of this process. If significant problems are found through periodic audits, a stop work order or contract cancellation could result.

5.2.4.3.1 Use of Phased Audits for Monitoring Laboratory Performance

The implementation of a two-phase audit and check system is a method for oversight of contract laboratory operations. A two-phase check involves a system of pre-award on-site audit and follow-up inspections with attendant documentation for control over data quality and processes relevant to contract requirements. Audits planned for project activities shall be addressed in the project-specific SAP. MRD validation and agency audits do not preclude the need for project-specific audits.

5.2.4.3.1.1 Pre-Award On-Site Audit

These audits include review of project initiation systems, laboratory sample handling and tracking procedure, sample analysis procedures, routine quality control checks, data handling and reduction, data and report review systems, data storage, electronic data handling, reporting, and storage, personnel qualifications and training, corrective action systems, standards control, document control, waste handling and disposal, and the laboratory ethics training. Highly specific project requirements, such as calibration criteria, sensitivity check samples, matrix spiking levels, data validation criteria, and PE sample acceptance criteria will be in place prior to the start of the project these criteria shall be documented in the project-specific SAP.

A thorough systems audit will be performed covering project specific analyses, prior to the approval of the laboratory service agreement. In a thorough audit there will normally be some areas identified that need improvement. If pervasive problems are found during a pre-award audit, the laboratory will not be awarded a project until their systems have been brought up to the standards required by the lead agency or primary contractor. Professional judgement has been applied when determining whether the laboratory will be contracted using historical information. Laboratory performance history, age, source of information, and analyses performed have been considered when using existing reports. The existing reports and pre-screening information reviewed will be documented and provided in the CDQMP.

5.2.4.3.1.2 Audit Reports

A report summarizing audit findings will be generated by the Primary Contractor following each audit. Laboratories will be expected to respond promptly to all audit findings (less than four weeks). The audit reports shall contain audit criteria, areas evaluated, and specific findings. The laboratory's audit response shall contain the audit findings, their response to the audit finding, schedule implementation (as necessary), and backup information (as necessary). The audit process from site visit to final resolution should not be allowed to progress over an extensive period of time. Also, the contractor shall provide a schedule for follow up to verify resolution and determine the laboratory's capability to meet the project requirements to the USACE.

5.2.4.3.1.3 Using Follow-up Audits

After passing the pre-award on-site audit the project laboratories must be managed by Primary Contractor. If laboratory work is subcontracted by a primary contractor for the USACE, the laboratory subcontract specifications will be developed that explicitly include the execution of primary contractor quality control oversight activities.

If the pre-award on-site audit revealed significant laboratory deficiencies, follow-up audits will be performed at the discretion of USACE and the Primary Contractor to ensure that corrective measures have taken place to sufficiently address the deficiencies and to ensure data quality requirements are being met. Follow-up audits will focus heavily on project specific data. They will incorporate the review and tracking of raw data from the original measurements through the generation of a final report. Audits normally will require some regeneration of raw data from electronic files to verify the integrity of this process. If significant problems are found through periodic audits, a stop work order or contract cancellation could result.

5.2.4.4 Assessments and Response Actions.

The text of the QAPP shall identify the number, frequency, and type of assessment activities to be executed for this project. Assessments to be conducted by the Contractor during the execution of delivery orders shall include but are not limited to surveillance, peer review, management systems review, readiness review, technical systems audit, performance evaluation, audit of data quality, and data quality assessment. The text shall discuss the information expected from the assessment and success criteria (i.e., goals, performance objectives, acceptance criteria specifications, etc.) for each type of assessment required. For each assessment, list the approximate schedule of activities, and discuss the information expected from the assessment and the criteria for success. For any planned self assessments (utilizing personnel from within the project groups), identify the participants and their exact relationship within the project organization. For independent assessments, identify the organization and person(s) that will perform the assessments. Discuss how and to whom the results of the assessments will be reported. Define the authorities of the assessors. For example, if the assessors should order a work suspension upon finding a significant condition, this section delineates clearly their authority to do so. Define explicitly the unsatisfactory conditions under which the assessors are authorized to act. Recognizing that assessments may be needed at any time during the project, provide a schedule for the assessments to be performed. Discuss how response actions to non-conforming conditions will be addressed and by whom. Identify who is responsible for implementing the response action. Describe how response actions will be verified, validated, and documented.

5.2.4.5 Electronic Data and Table Audits

Data tracking audits will be preformed by the contractor on a routine basis, specified in the CDQMP. These audits focus on project-specific data. They incorporate the review and tracking of raw data from the original measurements through the generation of a final report. Regeneration of raw data from electronic files is required to verify the integrity of this process. If significant problems are found through periodic audits, a stop work order or contract cancellation could result.

5.2.4.5.1 Laboratory Internal Tape Audits

Project laboratories periodically audit their electronic data to verify that the procedures are being followed. In cases

where problems are indicated from other quality assurance measures, such as systems audits or PES, electronic data audits will target the areas of concern. The audit will result in a report that includes description of the tapes inspected, the date of the audit, the person performing the audit, any findings or problems observed, recommended corrective actions, and recommended frequency of future audits ("Good Automated Laboratory Practices"). Any findings that may affect data quality or data integrity will be reported to the laboratory management. Any findings that are verified to affect data quality or data integrity will be reported to USACE and the Primary Contractor.

5.2.4.5.2 Independent On-site Audits

During any pre-award or follow-up audit, an independent on-site audit will be performed. While it cannot be as detailed as either an internal audit program or an off-site external audit program, it is important as a QA tool to verify that the laboratory's internal program is effective.

As a first step, laboratory auditors will review the information from the internal electronic data audit program. Once this is complete, the auditor will choose some data packages and enlist the laboratory's assistance in finding the associated logbooks. The logbooks will be reviewed to see if any files were documented to require manual changes to the original results. If so, these files will be reviewed to verify that the manual changes were based on technically sound judgement, and that the results in the electronic file are the same as the results on the hard copy report or the hard copy files. A number of files that are not documented as requiring manual changes will also be inspected. The laboratory personnel will be asked to regenerate the original data. It will be inspected for manual changes, and be compared to the hard copy report or files.

This kind of on-site audit cannot verify fraud, nor would it detect certain types of inappropriate data manipulation, but it can only help to assess the effectiveness of the laboratory's internal electronic data audit program. Significant discrepancies found during this process would indicate that either the laboratory's program is weak or that there may be a more pervasive data integrity problem. In a pre-award audit, either conclusion will be sufficient to eliminate the laboratory from further consideration, and in a follow-up audit, it could result in a stop work order or contract cancellation.

This type of on-site audit will also encourage the development of strong internal audit programs throughout the laboratory industry. If the ability to acquire work is dependent upon an effective internal electronic audit program, then these programs will become a priority for laboratory managers.

5.2.4.5.3 Independent Off-Site Electronic Data Audits

Off-Site electronic audits will be performed whenever other measures indicate the need based on laboratories practices. Independent off-site electronic data audits are by far the most rigorous form of electronic data audits. These audits can be a definitive tool in identifying gas chromatography (GC) and gas chromatography/mass spectrometry computer fraud. These have been the most frequently detected categories of laboratory data fraud. Off-site electronic data audits have been the tool of choice to definitively identify computer data fraud, and have been crucial as evidence in convicting laboratories of computer fraud. They have been used to detect fraudulent reporting of DFTPP and BFB tuning compound results, calibrations, surrogate recoveries, internal standard areas, and under reporting of target compound concentrations where the laboratory was required to dilute and re-analyze highly contaminated samples. While these audits require considerable expertise on the part of the auditor, they can detect a wide variety of inappropriate data manipulations. When data fraud is suspected, electronic data audits will be required to determine the extent to which the data fraud affected data quality. In doing this, the electronic data audits can salvage critical environmental data.

An independent off-site electronic data audit program can help to deter computer fraud. Laboratory managers who are aware that their data is likely to undergo this level of scrutiny will be more likely to institute effective internal

data handling procedures and an internal audit program. However, any questionable practices revealed through this type of audit have already affected some quantity of environmental data. The laboratory internal audits and the on-site pre-award audits can detect the potential for data fraud before a contract has been signed and before any samples have been collected. It is important, therefore, to use all three of these tools in conjunction with each other. A laboratory will not be put under contract unless it has an internal program that is verified to be effective through an on-site audit. The independent off-site audits may be used as a periodic oversight tool and in cases where inappropriate data handling is suspected.

5.2.4.6 Reports to Management

The text of the QAPP shall identify the frequency, content, and distribution of reports issued to inform management of the status of the project, results of performance evaluations and system audits, results of periodic data quality assessments, and significant quality assurance problems and recommended solutions. The text shall identify the responsible persons that will prepare the reports and the recipients of the reports. Reports to management required by these specifications include but shall not be limited to minutes of preparatory, initial, and follow up inspection reports associated with three phase control, reports of assessment activities described in 5.2.4.6, Daily Quality Assurance Reports, Quality Control Summary Reports, Non-Routine Occurrences Reports, and the Data Report to the QA Lab.

5.2.5 Data Validation and Usability

The data validation strategy must be established at the beginning of the project and documented in the QAPP, and be consistent with project DQOs. The criteria for data validation must be specified for analytical parameters including screening level data, natural attenuation parameters and bioremediation parameters. Twenty percent (10% of primary and all QA data) will be validated by an independent third party, i.e., someone unassociated and without any interest with the laboratory. Data validation is a systematic process for reviewing a body of data against a pre-established set of quality control "acceptance" criteria to determine whether it is within the criteria windows to determine the quality of the data. Where data do not meet the "acceptance" criteria, they are flagged with a qualifier identifying the associated problem. Data validation will occur as soon as the data is received by the Primary Contractor. The most critical analyses have been scheduled for accelerated turn-around-time from the laboratory. This will allow for corrective actions to take place early in the analytical process. After validation, the data is assessed to determine if it is adequate for its intended purpose and the data user will have data of known quality. Data Validation is covered in Section 5.2.5.2. The flagging conventions are presented in Tables 5-1 through 5-4.

5.2.5.1 Data Review, Validation, and Verification Requirements

In all cases field and laboratory data shall be reviewed for quality, accuracy, and completeness. As applicable to the circumstance and as in specified in specific delivery orders varying levels of data review/validation may be required. The minimum level of data review/validation for analytical chemistry data that will be required in all cases is described in Section 5.8. Requirements for more comprehensive review and validation of data may be described in individual delivery orders. This may include preparation of CLP type data validation reports. The Contractor shall be prepared to execute all data review and validation requirements necessary within this range and shall describe capabilities and applicable procedures consistent with the requirements of these specifications to accomplish this activity in the CDQMP. For specific delivery orders the Contractor may be required to sub-contract data review/validation such that this activity is performed independently relative to the organization that is generating the data. The text of the QAPP shall describe the criteria used to review and validate data in an objective and consistent manner. The text shall present criteria applicable to field and laboratory data. Examples of any forms or checklists to be used by the Contractor for this purpose shall be included. The text shall describe any calculations that will be needed to prove or disprove the project objectives.

Note: If data review/validation is sub-contracted the name and qualifications of the selected sub-contractor shall be submitted to the USACE Contracting Officer for approval. At any time, the CO may direct the Contractor to select an alternate sub-contractor if the initial designee is found to be unacceptable. No additional cost over the

amounts negotiated in the delivery order will be paid by the Government if the initially selected sub-contractor is rejected due to performance failure.

5.2.5.2 Validation and Verification Methods

The text shall describe the process to be used for validating and verifying data, including the chain of custody for data throughout the life cycle of the project or task. Methods for validation of field and laboratory data shall be included and shall be consistent with the National Functional Guidelines for Organic and Inorganic Data Review as applicable to SW-846 analyses. These methods shall be presented as tabular summaries providing explicit descriptions of the data validation criteria, methods, qualifier flags to be used (J, R, etc.), and bias designation. The text shall also provide an explicit description of the processes used for verification of information contained in boring logs, field notes, and field sampling records as applicable. Discuss how issues shall be resolved and the authorities for resolving such issues. The text shall describe how the results are conveyed to data users. At a minimum the review of field data shall include checks of the following as applicable: transcription errors, transmittal errors, QC data, detection limits, instrument calibration, accuracy of sampling records such as groundwater sampling field data sheets, performance evaluations, technical systems audits, contract compliance issues (e.g., holding times), and statistical data treatments such as tests for outliers.

It is important to note the following flagging conventions: 1) data is not qualified for QC failures when the spiking standards are diluted out of the sample to quantitate detectable concentrations, 2) if the PQL check standard fails to meet the 20% criteria, the reporting limit for the associated samples should be evaluated and whether the DQOs were achieved should also be evaluated. Analytical bias must also be discussed in the data validation reports.

5.2.5.3 Reconciliation with Data Quality Objectives:

The QAPP shall describe how the results obtained from the project or task will be reconciled with the Data Quality Objectives. The text shall describe how issues will be resolved and provide a discussion as to how limitations on the use of the data will be reported to decision-makers. The text shall identify the specific mechanism by which that will be used to convey assessments of precision, accuracy, and completeness for the project data.

5.3 FIELD SAMPLING PLAN

The FSP shall contain detailed comprehensive information regarding program field activities and sampling methods and requirements for the contract. The FSP will serve as a tool for reference in the site-specific SAPs.

5.3.1 Title Page and Table of Contents

Refer to Section 5.1.1 and 5.1.2.

5.3.2 Site Background

Refer to Section 5.2.2.2 and 5.2.2.3.

5.3.3 Sampling Objectives

Using the guidance referenced in Section 2.0 and the DQOs (Section 5.2.2.4), the contractor will specifically outline the various objectives in detail for this program and provide guidance for the site-specific SAPs.

5.3.4 Sample Location and Frequency

The contractor will describe the sample locations and frequency in general for the program. The requirements for collection of QA/QC and PE samples will be specified. The site-specific SAPs will present details regarding the site-specific sampling locations and frequency.

5.3.4.1 Sample Types

The FSP will contain a description of the types and purpose of the sample types described below.

5.3.4.1.1 Trip Blanks. Trip blanks are composed of purged DI water added to a clean preserved VOA vial. The trip blank accompanies sample containers from the laboratory to the field and back again to the laboratory. Trip blanks shall be prepared and submitted to the Contract Laboratory (and the QA laboratory) for each shipment of environmental samples for VOC analyses (every cooler containing VOC samples shall contain a trip blank that shall be analyzed by the Contract Laboratory). Trip blanks shall be analyzed for all VOC analyses (including 8015B as gasoline) specified for samples in the corresponding cooler with the exception that if samples are to be analyzed for multiple VOC analyses covering the same analyte list the trip blanks shall be analyzed only for the method incorporating the lowest PQL.

5.3.4.1.2 Quality Control (QC) Samples. Quality Control samples (duplicates, rinsates, source water, etc.) are submitted to the Contract Laboratory for the purpose of assessing Contract Laboratory precision. QC field duplicate samples will be collected as 10% of the total sampling effort. Generally QC duplicates shall be collected for the first sample and every tenth sample thereafter. If information regarding areas of particular interest at a site is available (i.e. highly contaminated areas) the distribution of QC samples may be placed at the discretion of field personnel with the concurrence of the USACE CO. QC duplicate samples shall be analyzed for the same parameters as the corresponding primary sample. Other QC samples shall be collected at a frequency based on the field sampling procedures and DQOs for the project. The frequency will be stipulated in the CDQMP and site-specific SAPs.

5.3.4.1.3 Quality Assurance (QA) Samples. QA samples are co-located split samples that are submitted to a designated QA laboratory. The QA Laboratory is a government laboratory. Results of these analyses compared to Contract Laboratory data shall be used in preparation of the Chemical Quality Assurance Report by the SPK District. QA split samples shall be generally collected as 10% of the total sampling effort. Generally, QA samples shall be collected for the first sample and every tenth sample thereafter. If information regarding areas of particular interest at a site is available (i.e. highly contaminated areas) the distribution of QA samples may be placed at the discretion of field personnel with the concurrence of the USACE CO. QA duplicate samples shall be analyzed for the same parameters as the corresponding primary sample. The specific rate of QA samples and the laboratories that QA samples shall be sent to will be directed in individual delivery orders and shall be stipulated in the site-specific SAP. Sections 5.3.2.6 and 5.2.4.1.3 describe requirements for QA samples.

5.3.4.1.4 Rinsate Samples. One rinsate sample shall be collected for each day of sampling and for each crew performing groundwater sampling during field operations. Rinsate samples shall be analyzed for all analytical methods that the primary samples are analyzed. Rinsate samples shall be performed daily for groundwater sampling activities if reusable bailers are used. If disposable bailers are utilized for sampling rinsate samples will not be required. For soil sampling the Consultant shall propose a minimum rate of rinsate sampling. Daily rinsate samples for soil sampling will not be required for this investigation.

5.3.4.1.5 Source Water and Field Blank Samples. One source water sample shall be obtained for each lot (5 gallon container, lot #, etc.) of water that is used for rinsing. If source water is obtained from an on-site source water outlet, the frequency will be generally be once per field effort. For estimating purposes this will be assumed to be one per day of field activities involving sampling. Field blanks shall only be performed for groundwater sampling activities involving VOC analyses. When disposable bailers are used, a bailer blank sample will be collected for each lot of bailers used.

5.3.4.1.6 External QA/QC Samples. QA/QC samples are field splits and duplicates. Duplicate/split samples to be analyzed by both the Contract Laboratory and the QA Laboratory. These samples are generally collected at a frequency of 10 percent for each matrix. The QA splits and field duplicate samples will not be performed on IDW samples. The frequency of these samples and analyses required shall be stipulated in the site-specific SAP. The Consultant shall be responsible for the collection, labeling, packing, and shipping of QA samples to the QA Laboratory. Assignment of a District QA laboratory will be arranged through the project USACE TM. The Consultant shall notify the Environmental Group Coordinator at the assigned QA Laboratory by telephone at least two weeks in advance of sample shipment (for large numbers of samples, greater than 20) and again on the day that samples are forwarded to the QA Lab. QA samples

that are obtained on Friday or during the weekend shall be held in the field under appropriate preservation until delivery for the next business day can be arranged.

Samples arriving at the QA Laboratory without appropriate preservation or packaging will be rejected with re-sampling cost at the expense of the Consultant. Additionally the Consultant will be required to resample the corresponding primary sample(s) and resubmit to the Contract Laboratory.

5.3.5 Field Documentation

All field documentation shall be maintained in bound sequentially paginated notebooks. Loose leaf forms must be not be used without being bound or added to the field notebooks, as described below. Indelible ink must be used for all field documentation. Errors shall be crossed out (so as not to obliterate the original entry) with the initials and date of the person making the correction. If computer printouts or loose leaf forms are used, the pages are taped into the logbook, taped with clear permanent tape, initialed and dated by the person responsible for the entry.

5.3.5.1 Sample Information Documentation. All information pertinent to the environmental samples, including specific field collection data, names of sampling personnel, and laboratory observations shall be recorded in permanently bound notebooks. Sample identifications shall be linked to the site where the sample originated. The Contract Laboratory shall also employ a specific information management system to assist in tracking the progress of each sample through the analytical process. The FSP shall detail procedures for documentation of field and laboratory information that are consistent with the requirements of these specifications.

A sample collection tracking table shall be generated by the contractor which contains at a minimum of the following information: field sample identification, sample type, date of collection, laboratory SDG (as available), analyses requested, laboratory, date received by laboratory, date the data packages are due, and date the data packages are received (as available). These tracking tables shall be updated daily and provided to the USACE Project Chemist in a timely manner (daily or weekly, as necessary based on the project schedule).

5.3.5.2 Preparation of Field Logbooks. The field logbook shall be bound with serially numbered pages, and assigned to a specific person who is responsible for entry of information into the logbook. The logbook will be signed and dated by this person prior to initiation of field work. All entries into the logbook will be executed by this designated person. 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 shall be made by crossing a line through the entry and entering the correct information. The correction shall 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 shall 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. Field logs will become part of the project records and will be delivered to the USACE CO at the end of the project.

5.3.5.3 Photographs. When samples are being collected, photographs shall be taken to support the written description of sampling activities. In all cases when a photograph is taken the date, time, weather conditions (if applicable), subject, purpose for photographs being taken, number of photograph and identifying number from roll, and the name of the person taking the photograph shall be recorded. When photographs are developed the information in the field logbook will be transferred to the back of the photograph. All photographs will become part of the project file and subject to all standard document controls. All photographs will be delivered to the USACE CO at the end of the project. The compass orientation shall be documented on the photograph.

5.3.6 Sampling Equipment and Procedures:

All sampling activities shall be performed according to protocols, specific to each parameter of interest, promulgated by the U.S. Environmental Protection Agency (EPA) and by USACE. Where such protocols have not been established by the EPA or the USACE, protocols established by some other recognized authority (ASTM, Cal EPA) should be utilized. At a minimum the FSP shall fully describe the following procedures related to sample acquisition:

Hollow stem auger drilling procedures
Mud Rotary drilling procedures
Air Rotary drilling procedures
Sonic drilling procedures
CPT drilling and sampling procedures (soil)
Drilling equipment decontamination
Lithologic logging
Borehole abandonment
Monitoring well construction methods
Filter pack and well screen slot size determinations (in-field procedure)
Monitoring well development procedures
Monitoring well abandonment procedures
Temporary well installation procedures (shallow)
Volatile Organic Sampling Procedures for water and soil (SW-5035)
Split spoon sampling procedures
Wire-line coring sampling system (soil and rock)
Shallow hand auger sampling procedures
Grab sampling procedures
Stockpile sampling procedures
Groundwater sampling procedures (monitoring well)
Hydropunch groundwater sampling procedures
Low Flow sampling procedures
Soil vapor sample collection procedures
Surface water sampling procedures
Drum sampling procedures (concentrated waste - multiple phases)
Field analytical procedures (pH, conductivity, temperature, organic vapor, water levels, turbidity)
Composite sampling procedures

5.3.6.1 Sampling Procedures. The FSP shall detail all information relative to the sampling process, including equipment to be used, sample volume, and sampling technique. The sampling procedures presented in the following sections will form the basis of the sampling procedures that will be executed by the Contractor during execution of work for this contract. Sampling procedures presented in the FSP shall be consistent with the procedures described in these specifications as applicable. All sampling equipment will be made of Teflon ®, or stainless steel, which has been decontaminated. Materials such as polyvinyl chloride and other plastics will not be used. All sampling procedures are subject to the approval of the CO.

The FSP shall contain sufficient detail to understand the sampling area, sampling grid placement, sampling points, and well placement (i.e., up gradient, down gradient, confined, unconfined, etc.). The Contractor shall use EPA, USACE, and industry guidance to establish minimum standards and explain field sampling requirements based on the DQOs. EPA and ASTM methods must be referenced in the appropriate SOPs. Precision and accuracy requirements must also be stipulated for each method. Groundwater and split spoon sampling procedures are outlined in these specifications below as examples of the requirements for field samples for USACE projects. The level of detail for the SOPs must include similar information, sufficient detail, and are subject to approval by the USACE.

5.3.6.1.1 Groundwater Sampling Procedures

Prior to groundwater sampling operations the sampling team shall examine each well for signs of tampering or well deterioration. Any observations will be noted in the field notebook. After the well has been opened the air in the well head area will be tested for organic vapors with the PID and for explosive atmospheres with the oxygen/combustible gas indicator. Results of these observations shall be recorded in the field notebook. A plastic sheet shall be placed around the well head beneath all sampling equipment to prevent contamination of surficial soils during purging and sampling. The depth to standing water in each of the wells, the presence and thickness of floating product (if any), and total depth of the well to the bottom of the screened interval shall then be determined using an oil/water interface detector and recorded in the field notebook. This information is required to calculate the volume of stagnant water in the well and to provide a check on the integrity of the well. The top of the casing shall serve as a permanent reference point from which water level measurements shall be taken.

Using information on the diameter, total depth, and depth to water for the well, three casing and filter pack volumes shall be calculated and that amount of water shall be purged from the well. The pH, temperature, conductivity, dissolved oxygen, redox potential, and turbidity of the water will be monitored as well. The meters will be calibrated prior to use at each well using ASTM traceable standards rather than "auto-calibrated". The calibration will be checked after measurements for all samples have been completed to insure that the field instruments have remained in calibration throughout the process. Results of calibrations and final calibration checks will be recorded in the field notebook. If after three well volumes these three parameters have stabilized the well will be sampled. Stabilization criteria shall be three consecutive measurements for which the pH is within +/- 0.1 units, temperature is within +/- 0.5 degrees, conductivity is within 10%, dissolved oxygen is within 10%, turbidity is within 10% (0.10 NTU), and redox potential is between –400 mV and +800mV. Modifications to these criteria must be stipulated in the FSP and are subject to approval by the USACE Technical Manager and Geologist. At least six measurements will be obtained (one for each half-casing volume). Measurements for well parameters will also be obtained after sampling is completed with the results recorded in the field notes. If these three parameters have not stabilized after three volumes the purging will continue to a maximum of five volumes before sampling commences. Turbidity will be monitored with results recorded in the field notes may not be used as a stabilization parameter depending on the site. If purging is accomplished using a submersible pump the pump will be set just below water level so that all standing water is removed from the well. Placement of the pump for purging should take into consideration the anticipated depth to which water will be drawn down during pumping. The volume of water purged and the withdrawal rates will be recorded. Purge rates will be sustainable and executed at a rate such that draw-down is minimized to prevent cascading of water into the well. Alternatively, the wells may be purged by bailing. During the evacuation period, the appearance of the discharge water will be noted and periodic entries will be made in the sampling notebook. Use of a well purging data sheet for recording the information described above is acceptable.

A complete set of sampling pre-preserved containers and associated trip blank samples will be prepared for each sample in advance of the sampling event. Containers will be labeled with the date, sample number, project name, samplers name or initials, parameters for analysis (method numbers where possible), and preservation. All samples will be collected within the screened interval in each well to ensure that the sample is representative of formation water. The bailer will be carefully lowered beneath the top of the screened interval after purging of the well. A water sample is collected. The water from the bailer is then carefully transferred to sample containers using a bottom valve-discharging device. Pouring from the top of the bailer will not be allowed. Volatile water samples will be taken with a bottom valve-emptying device so that no air passes through the sample (to prevent volatile organic compounds from being stripped from the samples); the bottles will be filled by inserting the spout from the bailer to the bottom of the VOA vial with discharge of the bailer contents into the vial such that the tip of the spout is kept beneath the surface of the liquid in the vial as it is filled until there is a convex meniscus over the neck of the bottle. The Teflon side of septum (in cap) will be positioned against the meniscus, and the cap screwed on tightly; the sample will be inverted, and the bottle tapped lightly. The absence of an air bubble indicates a successful seal; if a bubble is evident the sample will be discarded. Refilling of VOA vials will not be allowed.

All sample bottles and equipment will be kept away from fuels and solvents. Gasoline (used in generators) shall be transported in a different vehicle from bailers, sample bottles, purging pumps, etc. If possible, one person should be designated to handle samples, and another person should work generators and the gas truck. Disposable gloves will be worn for each separate activity and then disposed of. Care will be taken not to spill any fuels on clothing.

To collect VOCs samples using the Encore ® sample collection system for low level (>1 ug/kg) analyses of soils, the contractor shall follow the procedure outlined below. Remover the sampler and cap from package and attach T-handle to sampler body. The technician quickly pushes the sampler into a freshly exposed surface of soil until the sampler is full. A paper towel is used to quickly wipe the sampler head so that the cap can be tightly attached. Three five-gram samples are necessary for each sample point (primary sample, backup, and percent moisture determination). An additional 25-gram sample should be collected for screening and or high level analysis. The containers must be shipped to the laboratory within 24-hours of sample collection. The contractor SOP must contain details regarding acid preservation sampling for low level analyses, testing for effervescing capacity of soils, testing buffering capacity of soils, Encore ® sampling for high level (>200 ug/kg) analyses, and methanol preservation. Documentation, collection of QA/QC samples, and holding time requirements must also be addressed in the SOP.

5.3.6.1.2 Split-Spoon Soil Sampling Procedures

Split-spoon sampling procedures shall be executed in accordance with ASTM D 1586-84, Standard Method for Penetration Test and Split-Barrel Sampling of Soils. The sampler will be fitted with three stainless steel sleeves. The sampler will be driven 18" or to refusal with a 140 lb. hammer dropping 30" repeatedly. The number of blows required to drive the sampler each six inches will be recorded. Refusal shall be defined as requiring 50 blows with the hammer to advance the sampler six inches or less. All equipment that comes into contact with the soil shall be fully decontaminated in accordance with paragraph Equipment Decontamination Procedures prior to the boring.

As the sample tubes are disassembled an organic vapor monitor (PID or FID) probe will be inserted into the gap between two sample liners and the liner exhibiting the highest reading will be selected for analysis. In general the middle liner will be collected for laboratory analysis. In addition, 10% of the bottom liners will be collected for QA/QC testing. One half of the soil in the top liner will be placed into a re-sealable plastic bag and left in the sun for approximately fifteen (15) minutes to allow any VOCs to volatilize. The soil vapor in the plastic bag will then be monitored by taking a reading of the headspace. Background VOCs in the bag will be determined by monitoring the air in an empty bag. Results of the organic vapor monitoring shall be recorded on the boring logs. Soils in the sample sleeves should be logged before they are sealed if VOCs are not contaminants of concern. Small portions of soil at the ends of the sleeves can be scraped off for classification.

The sleeves collected for laboratory analysis will be covered at both ends with Teflon sheets, capped, and taped with Teflon tape. Use of adhesive tape will not be allowed. Labels shall be affixed to the liners bearing job designation, time, boring number, sample depth interval, sample number, date sampled, and the initials of the sampler. The samples will then be enclosed in a plastic bag and stored in a cooler maintained at 4 degrees Celsius prior to shipment.

Each drilling rig that is operating in the field will be continuously inspected by a Geologist with a minimum of three years experience in environmental drilling and sampling. Continuous inspection is essential to insure that the intent of the drilling program is being followed and to provide knowledgeable direction to the field crew when conditions dictate variance from the original plan. Boring logs will be prepared using USACE Engineering Form 1836R. At a minimum boring logs will contain lithologic descriptions of soil strata, depth to groundwater, sample ID's, blow counts, and PID readings for headspace analyses. All drilling operations and/or well installation work for this project will be conducted under the supervision of a Registered Geologist. All boring and well installation logs will be signed by the field Geologist and by the supervising Registered Geologist.

5.3.6.1.3Equipment Decontamination Procedures.
During sampling activities, appropriate decontamination measures shall be taken to minimize sample contamination from sources such as sampling equipment or sample containers. These procedures shall consistent with those outlined in "Test Methods for Evaluating Solid Waste-Physical/Chemical Methods" (U.S. EPA SW-846, 3rd ed.). Steam cleaning will be acceptable for drill rigs and drilling rods. The decontamination procedure for sampling equipment shall incorporate a non-phosphate detergent wash, tap water rinse, rinse with pesticide grade methanol, rinse with de-ionized water, and a final rinse with type II reagent grade water with verified no detectable concentrations of the site chemicals of concern above the project specified PQLs. As an alternative to use of reagent grade water the Contractor may submit analytical data to indicate that de-ionized water to be used for the final rinse is free of the contaminants of concern for this project above the level of detection for the relevant analyses. If this option is chosen by the Contractor this data must be submitted prior to the initiation of field work to the USACE CO. The sampling program established for this project shall include provisions for generating the appropriate field QA/QC samples to monitor the effectiveness of the specific procedures employed in controlling contamination of samples as a function of field procedures or ambient conditions. The FSP shall detail all measures used to avoid sample contamination.

5.3.7 Sample Handling Procedures

5.3.7.1 Sample Containers. The types of containers and procedures used for cleaning these containers shall consistent with EPA and USACE requirements for the specific parameters of interest. The sample container label must include location, time and date of sampling, grab or composite, analyses to be performed, and sampler's signature. Sample containers planned for use shall be described in the FSP.

5.3.7.2 Sample Preservation. All samples collected shall be preserved according to EPA and/or USACE protocols established for the parameters of interest. Appropriate measures shall be taken to ensure that storage requirements with respect to temperature are maintained in the field, during transport to the laboratory, and during storage at the laboratory. Temperature blanks shall be used for all coolers containing samples requiring preservation at reduced temperature. The CDQMP shall detail sample preservation methods for all analyses to be used for this project.

5.3.7.3 Sample Transportation. Environmental samples shall be transported to the Contract Laboratory and QA laboratory via the most rapid means. Samples shall be packaged and transported according to health and safety requirements covered by the EPA, USACE, and DOT regulations. The FSP shall describe the planned mode of sample transport with detailed packing procedures. SUMMA canisters shall be shipped by express ground transportation and not by airplane.

5.3.7.4 Chain of Custody Procedures. Samples shall be collected, transported, and received under strict chain of custody protocols consistent with procedures established by the EPA for litigation-related materials. On receiving samples at the Contract Laboratory the air temperature inside the cooler and of the temperature blank shall be measured immediately after the cooler is opened with the results recorded on the Cooler Receipt Form. Water samples requiring acidic or basic preservation will also be checked for pH on arrival at the Contract Laboratory. Contractor and Contract Laboratory chain of custody procedures shall be detailed in the QAPP & FSP. Copies of chain of custody forms shall be provided to the CO whenever samples are shipped from the field site (facsimile transmission). Upon receipt at the laboratory, the laboratory shall provide a specific mechanism through which the disposition and custody of the samples are accurately documented during each phase of the analytical process. Cooler Receipt Forms shall be used to document the condition of samples on arrival at the laboratory. The results of all checks for preservation of samples shall be recorded on the Cooler Receipt Form. Examples of chain of custody forms and cooler receipt forms shall be provided in the CDQMP. An example of an acceptable Cooler Receipt Form can be obtained from the USACE CO.

5.3.8 Investigation Derived Waste
The text of the FSP shall describe the installation-wide provisions that will be made for the proper handling and disposal

of wastes generated through the various field operations.

5.3.9 Quality Control for Field Operations

Overall requirements described in Section 5.2.4 (including subsections) will be addressed. The text of the FSP shall substantially reflect the specific procedures as they apply to three-phase control with specific reference to the execution of field operations related to sampling and analysis. Checklists that are developed for implementation of three phase control shall be included in the text. Examples of these types of checklists are included in Appendix H of EM 200-1-3.

5.4 GUIDANCE FOR THE PREPARATION OF DELIVERY-ORDER/SITE-SPECIFIC SAMPLING AND ANALYSIS PLANS

5.4.1 Title Page, Signature Page, and Table of Contents

Refer to Section 5.1.1 and 5.1.2.

5.4.2 Executive Summary

The executive summary shall be composed of a brief description of the context of contract or project work, the goal of the proposed investigative work, a general description of the work to be performed, and a brief statement describing the relevance of the work to be performed to the goal of the investigation as applicable. This information is project specific

5.4.3 Problem Definition and Background

A narrative describing the project shall be included that shall state the specific problem to be solved or the decision to be made. The goal of the investigation shall be clearly stated.

- Describe 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. Include diagrams detailing areas to be sampled as relevant to the definition of the investigation goals.
- Summarize the site geology/hydrogeology as known prepared to a level of detail such as to provide a comprehensive description of the site.
- Include enough information about the problem, the past history, any previous work or data, the regulatory or legal context, and any relevant ARAR's to present a clear description of the project objectives.

The information provided in this section should be detailed enough to provide background information for USACE and other reviews.

5.4.4 Project Description

Describe what work will be performed. Give an overall picture of how the project will resolve the problem or questions described in the Problem Definition/Background Section above. Include the following elements:

- Applicable technical, regulatory, or program specific quality standards, criteria, or objectives.
- General description of the sampling approach and measurements to be conducted for the project. Include a table that lists the feature of work and the responsible organization for each (contractor, contract lab, sub-contractor etc.)
- Required special project specific personnel or equipment that may add to the complexity of the project.
- Assessment tools that will be employed for the project (program technical reviews, peer reviews, surveillances, technical audits, etc.)
- Project schedule including start time, milestones and expected completion date. If individual sampling plans will be developed for discrete project phases include their preparation schedule.
- List of deliverables.

5.4.5 Project Organization

This section in the SAP shall address the specific personnel that will be responsible for execution of a delivery order. The SAP must address not only the Contractor personnel but any subcontractor interactions applicable for a delivery order.

- Specific personnel must be identified along with their function and qualifications.
- Specify lines of authority and communication among all project participants. Include other data users who are outside of the organization generating data, but for whom the data are nevertheless intended; e.g. modelers, risk assessors, design engineers, toxicologists, etc. Where direct contact between project managers and data users does not occur, the organization chart should show the route by which information is exchanged.
- Include subcontractor personnel as appropriate, including the contract laboratory.

5.4.6 Data Quality Objectives

State the general scope of work and 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 shall be used to obtain data of sufficient quality to meet or exceed all project objectives. The discussion of Data Quality Objectives (DQOs) shall follow the guidance contained in the EPA document Guidance for the Data Quality Objectives Process (EPA QA/G4) and the requirements of this document are included by reference. The section on DQOs 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.
- Identification of Decisions. 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 shall be prioritized.
- Identify Inputs to Decisions. Identify the information needed to support the decision and specify the inputs requiring environmental measurements.
- Definition of Study Boundaries. Specify the spatial and temporal aspects of the environmental media that the data must represent to support the decision.
- 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 DQOs.

Statements of the problem shall be defined quantitatively if possible. Example:

> UV Treatment of Contaminated Groundwater. "The purpose of this project is to demonstrate that the residual trichloroethylene concentration in the treated water is less than 0.5 ug/L at a confidence level of 95%."

Identification of decisions and descriptions of data use shall be described with text and supported with tables and lists that describe:

- Data needed. Measurement parameters, compounds, and sample matrices.
- The action levels or standards upon which decisions will be made, including the detection limits and data reporting units for relevant parameters.
- The summary statistic(s), e.g., mean maximum, range, etc., which specify the form the data will be in when compared against action levels or standards.

- The acceptable level of confidence in the data needed for the stated purposes; or the acceptable amount of uncertainty.

Describe in quantitative terms the sensitivity, precision, accuracy, and completeness goals for each major measurement parameter and for each matrix to be sampled. Define different types of sensitivity (e.g. quantitative, qualitative, screening) for each major measurement parameter as applicable. A qualitative discussion shall be presented regarding representativeness and comparability.

5.4.7 Sampling Process Design

Describe the experimental design of the project including:

- Sampling network design
- Sample types and matrices
- Sampling frequencies
- Measurement parameters of interest (compounds, elements or other analytical parameters).

In the sampling design discussion, the rationale for the design shall be clearly stated and described for all sites where samples will be collected. Include figures describing the specific sampling points. Measurement parameters to be described include geological, geophysical, hydrogeological, and chemical parameters as applicable. If cone penetrometer locations, hydropunch locations, or monitoring well locations are to be chosen on the basis of field observations, state the evaluation criteria that will be used in the field for these determinations. Monitoring well design criteria (if applicable) shall be clearly described to include a description of field determinations for appropriate filter packs and well screens.

Provide a table (e.g. the Planned Sample Table) that shows the estimated number of samples by location for:

- Field samples
- QC split samples
- QA split samples
- Field blanks
- Rinsate blanks
- Trip blanks

5.4.8 Sampling Methods Requirements

Provide a general description of sample collection procedures to be used for the project. Cite the CDQMP SOPs if applicable. If SOPs are not available detailed specific SOPs of these procedures shall be included in the SAP as an appendix. Where SOPs allow for alternate approaches or equipment depending on the sampling task, specify which approaches will be used for the project. Focus on Contractor procedures for addressing failures in the sampling system and responsibilities for corrective action. Provide a project specific table that describes bottle requirements, preservation, and holding times to extraction and/or analysis for all analytical parameters and matrices.

5.4.9 Analytical Methods Summary

Provide tabular summaries of preparation and analytical methods required for each site. Specify analytes and PQLs for each analytical method, especially if analytes are added to standard lists. Specify any method modifications necessary for the project (e.g. 25 ml purge for VOA analysis). Provide or cite laboratory QC requirements unless described in section 9.0, below.

5.4.10 Investigation Derived Waste

The text of the SAP shall describe the provisions that will be made for the proper handling and disposal of wastes generated on site.

5.4.11 Quality Control

Project-specific requirements described in Section 5.2.4 (including subsections) will be addressed. The text of the SAP shall substantially reflect the specific procedures described in the CDQMP as they apply to three-phase control with specific reference to the execution of field operations related to sampling and analysis. Checklists that are developed for implementation of three phase control shall be included in the text. Examples of these types of checklists are included in Appendix H of EM 200-1-3.

5.4.12 References

All references and guidance documents used to determine the content and format of this document shall be cited with appropriate titles, authors, and dates of publication.

5.5 ANALYTICAL DATA PACKAGE REPORT

The following sections describe the requirements for analytical data packages.

5.5.1 Format for the Comprehensive Certificates of Analysis

A. The "Cooler Receipt Form" shall be completed by the Contract Laboratory documenting sample conditions on arrival at the laboratory. Original copies of cooler receipt forms as well as original copies of chain of custody forms shall be provided with certificates of analysis. Examples of both forms shall be provided in the QAPP.

B. For each analytical method the Contract Laboratory shall report all analytes as a detected concentration or as less than the PQL. All samples with out of control spike recoveries being attributed to matrix interference will be designated as such. All soil samples will be reported on a dry weight basis with the percent moisture reported for each sample. Dilution factors, date of extraction, date of analysis, and practical quantitation limits shall be reported for each analyte and method.

C. Reports of method blanks shall include all analytes for each analytical method. Analytical results for each sample shall be clearly associated with a particular method blank. Any detected concentration found in method blanks shall be reported. Reports of concentrations below the PQL are necessary to evaluate low level determinations of target compounds in samples.

D. Surrogate spike recoveries shall be reported for all applicable methods. The report shall also specify the control limits for surrogate recoveries. Any out-of-control recoveries shall result in the sample being rerun once. If subsequent analyses result in out of control recoveries both results shall be reported and the data flagged.

E. MS/MSD recoveries shall be reported for all analyses. All sample results shall be designated as corresponding to a particular set of MS/MSD analyses. MS/MSD analyses not meeting quality control criteria specified in the QAPP shall be rerun once. If subsequent analyses result in out of control recoveries both results shall be reported and the data flagged. Only samples from this project shall be used for MS/MSD analyses. (The Contract Laboratory shall not use samples from other projects for MS/MSD analyses.) The report shall also specify control limits for spike recoveries and RPD for each spiked analyte.

F. Results for laboratory duplicates shall be reported with RPD limits for duplicate analyses.

G. LCS results shall be reported with control limits for LCS analyses. Analytical results for each sample shall be clearly associated with a particular LCS sample.

H. Results of initial and continuing calibration analyses for all analyses shall be included in the data package. Continuing calibration results shall be organized such that sample results shall be clearly correlated with the calibration check samples that bracket the sample results. Injection records for all sample analyses shall be included with the calibration data. Summaries of calibration data should be provided as a CLP Form VI and VII or equivalent for organic analyses and Form II modified for SW-846 analyses for inorganic analyses. (Note: Copied pages of handwritten laboratory notebooks will be unacceptable to fulfill the requirements of these specifications.)

I. The Contract Laboratory shall prepare a summary of all samples with detected concentrations of target compounds indexed by method and by sample ID.

J. The Contract Laboratory shall prepare a summary of all surrogate recoveries for organic analyses for each applicable method with the acceptable recovery range clearly indicated. This summary shall be performed for all samples for each analytical method involving surrogate spikes.

K. The Contract Laboratory shall prepare a summary of all Matrix Spike/Matrix Spike Duplicate analyses for each applicable method indicating acceptable recovery ranges and QC acceptance criteria for RPD.

L. The Contract Laboratory shall prepare a summary of all laboratory and field duplicates with QC acceptance criteria for RPD clearly indicated.

M. The Contractor/Contract Laboratory shall prepare a table identifying all QA samples and the corresponding primary samples for use by the QA Lab in preparation of the Chemical Quality Assurance Report (CQAR). This summary shall be delivered to the QA laboratory as described in Section 5.10.

N. The comprehensive certificate of analysis shall contain a narrative section identifying samples not meeting quality control criteria and any other out of control condition. The narrative shall describe the corrective action taken. If "matrix effects" are invoked as a cause for out of control recoveries a subsection of the narrative shall present a detailed justification for this assertion to include a summary of all relevant quality control data.

O. Chromatographs for all fuels analyses (detects and non-detects) presented at an attenuation where features of the chromatography are clearly visible shall be submitted for all projects involving fuels analyses by gas chromatography. Chromatographs of standards used for identification of fuels must also be included in the data package.

P. All data for analyses during the period covered by the comprehensive certificate of analysis shall be included as an appendix to the comprehensive report. This data shall be presented on numbered pages with an index or table of contents describing the contents of the appendix.

5.5.2 RAW DATA PACKAGES

Requirements for submittal: Raw data packages shall be submitted to USACE for 10% of all samples analyzed by the Contract Laboratory. The Contractor shall select samples for raw data packages to include all analyses and matrices, to provide temporal representation, to provide data in particular areas of interest, and to provide data at periods of maximum loading of the Contract Laboratory. The Contractor should notify the USACE CO of the samples that have been selected for submittal as raw data packages and the CO will have the option of directing the Contractor to select specific samples (other than those proposed by the Contractor) for reporting in this manner. The Contract Laboratory shall not be notified

of the samples for which raw data packages will be required until after the analytical process has been initiated. Raw data packages shall be delivered in place of the Comprehensive Certificate of Analysis. Raw data packages shall be delivered to the CO within 28 days of the time of sample acquisition in the field.

5.5.2.1 Organic Analyses

The raw data package for organic analyses shall 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 organic raw data package are provided in the following sections.

5.5.2.1.1 Case Narrative. The case narrative shall be written on laboratory letterhead and the laboratory manager or his/her designee shall authorize the release of data. 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.

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

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

5.5.2.1.4 Summary of QA/QC Results. The following QA/QC results shall be presented in summary form. Details specified in Section 5.5.2.1 (Organic Analysis) shall also be included for the summary of QA/QC results. Acceptance limits for all categories of QC criteria shall be provided with the data. All summaries will be presented on standard forms. Use of CLP standard forms is not necessary, however submission of standard instrument output alone is unacceptable to satisfy the requirements for raw data packages.

A. Initial Calibration. The concentrations of the standards used for analysis and the date and time of analysis. The response factor, percent relative standard deviation (%RSD), and retention time for each compound (as applicable, GC and GC/MS analyses) shall be included in initial calibration summaries. A statement should also be made regarding the samples or dates for which a single initial calibration applies.

B. Daily Calibration and Mid-level Standard: The concentration of the calibration standard used for daily calibration and/or the mid-level calibration check shall be reported. The response factor, percent difference, and retention time for each compound shall be reported (GC and GC/MS). Daily calibration information shall be linked to sample analyses by summary or by daily injection or analysis logs. Tuning information for GC/MS shall also be included with the calibration.

C. Method Blank Analyses: The concentrations of any compounds found in method blanks shall be reported. The environmental samples and QA/QC analyses associated with each method blank shall be stated.

D. Surrogate Standard Recovery: The name and concentration of each surrogate compound added shall be detailed. The percent recovery of each surrogate compound in the samples, method blanks, matrix spike / matrix spike duplicates and other QA/QC analyses shall be summarized with sample ID's such

that the information can be linked to sample and QA/QC analyses.

E. Internal Standard Recovery: The name and concentration of each internal compound added shall be detailed (retention time and area counts). The percent recovery of each internal compound in the samples, method blanks, matrix spike/matrix spike duplicates and other QA/QC analyses shall be summarized with sample ID's such that the information can be linked to sample and QA/QC analyses.

F Precision and Accuracy: For matrix spike / matrix spike duplicate analyses the sample results, spiked sample results, percent recovery, and RPD with the associated control limits shall be detailed. For laboratory duplicate analyses the RPD between duplicate analyses shall be reported as applicable. For laboratory QC Check and/or LCS analyses the percent recovery and acceptable control limits for each analyte shall be reported. All batch QC information shall be linked to the corresponding sample groups.

G Retention Time Windows (GC, GC/MS): The retention time window for each compound for both primary and confirmation analyses shall be reported. Retention time windows are to be updated daily per EPA SW-846.

H Compound Identification (GC, GC/MS): the retention times and the concentrations of each compound detected in environmental and QA/QC samples shall be reported for both primary and confirmation analyses.

I Method Detection Limits: Results of the most current detection limit study shall be provided in the raw data package.

J Injection Record: Injection logs for all instruments used for analysis of project samples shall 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.).

5.5.2.1.5 Raw Data. Legible copies of all raw data shall be organized systematically on numbered pages. The raw data for compound identification and quantitation must be sufficient to support all results presented in other sections of the raw data package. All raw data will be presented on standard forms and accompanied by the instrument output. Use of CLP standard forms is not necessary, however submission of standard instrument output alone is unacceptable to satisfy the requirements for raw data packages.

A. GC Analyses: This section of the data package shall 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 confirmations if applicable. The raw data for each analysis shall include chromatograms (preferably with target compound, internal standard and surrogate compounds labeled by name) with a quantitation report and/or areas print out.

B. GC/MS Analyses: This section of the data package shall 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 shall include chromatograms (preferably with target compound, internal standard, and surrogate compounds labeled by name) and enhanced spectra of target compounds and/or tentatively identified compounds with the associated best matched spectra. Quantitation reports for all analyses shall be included in the data package.

5.5.2.2 Inorganic Analyses. The raw data package for inorganic analyses shall 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 inorganic analyses raw data package are provided in the following sections.

5.5.2.2.1 Case Narrative. The case narrative shall be written on laboratory letterhead and the laboratory manager or his/her designee shall authorize the release of data. 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. The case narrative shall be sufficiently detailed such that the process of analysis can be reconstructed (i.e. if samples are diluted to bring results into the linear dynamic range, or re-extracted for QC failures the course of analysis shall be detailed in the case narrative.)

5.5.2.2.2 Chain-of-Custody Documentation. Legible copies of Chain-of-Custody forms for each sample shall be maintained in the data package. The date of receipt must be described on the Cooler log-in sheets shall be associated with the corresponding Chain-of-Custody form. Any internal laboratory-tracking document shall be included.

5.5.2.2.3 Summary of Environmental Results. For each environmental sample analysis the raw data package should include field identification and corresponding laboratory identification number, sample matrix, date of sample digestion (as applicable), date and time of analysis, identification of the instrument used for analysis, instrument specifications, weight or volume of sample used for analysis/digestion, dilution or concentration factor used for the sample extract, percentage of moisture in the sample, method detection limit or sample quantitation limit, definitions of any data qualifiers used, and analytical results.

5.5.2.2.4 Summary of QA/QC Results. The following QA/QC results shall be presented in summary form. Details specified in Section 5.10 (Inorganic Analysis) shall also be included for the summary of QA/QC results. All summaries will be presented on standard forms. Use of CLP standard forms is not necessary, however submission of standard instrument output alone is unacceptable to satisfy the requirements for raw data packages.

A. Instrument Calibration: The order of reporting of calibrations for each analyte must follow the temporal order in which standards were analyzed.
B. Initial Calibration: The source of the calibration standards, true value concentrations, found concentrations, the percent recovery for each element analyzed, and the date and time of analysis shall be reported.
C. Continuing Calibration Verification: The source of the calibration standards, true value concentrations, found concentrations, the percent recovery for each element analyzed, and the date and time of analysis shall be reported.
D. Method Blank Analyses: The concentrations of any analytes found in initial calibration blanks, continuing calibration blank, and in the preparation blank shall be reported. The date and time of analysis shall also be reported.
E. Interference Check Sample: The source of the interference check sample, true value concentrations, found concentrations, the percent recovery for each element analyzed, and the date and time of analysis shall be reported.
F. Precision and Accuracy - Matrix Spikes and Duplicates: For matrix spike analyses the sample results, spiked sample results, percent recovery, the spiking solution used, and the control range for each element shall be detailed. For post digestion spikes the concentration of the spiked sample, the sample result, the spiking solution added, percent recovery and control limits shall be detailed. For laboratory duplicates the original concentration, duplicate concentration, relative percent difference, and control limits shall be detailed. Date and time for all analyses shall be recorded.
G. Precision and Accuracy - Laboratory Control Samples: The source of the laboratory control sample, true value concentrations, found concentrations, the percent recovery for each element analyzed, and the date and time of analysis shall be reported.

H. Method of Standard Additions (MSA): This summary must be included when MSA analyses are required. The absorbance values and the corresponding concentration values, the final analyte concentrations, and correlation coefficients shall be reported for all analyses. Date and time of analysis shall be recorded for all analyses.

I. ICP Serial Dilution: The initial and serial dilution results with percent difference shall be reported.

J. 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 shall be reported.

K. ICP Inter-element Correction Factors: For each instrument and wavelength used the date on which correction factors were determined shall be detailed. Specific correction factors for Al, Ca, Fe, Mg, and any other element and the analytes to which they are applied shall be detailed. These elements must be quantitated accurately (within the quantitation range) to apply the correction factor for each sample, regardless of whether these are specified analytes required for analysis.

L. Instrument Detection Limits: Results of the most current detection limit study shall be provided in the raw data package.

M. Analysis Record: Analysis logs for all instruments used for analysis of project samples shall 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.).

5.5.2.2.5 Raw Data. Legible copies of all raw data shall be organized systematically on numbered pages. The raw data for compound identification and quantitation must be sufficient to support all results presented in other sections of the raw data package. This section of the data package shall 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 are particularly important since they provide the basic link between all sample analyses and QC information. (calibration standards, matrix spike, etc.) Instrument analysis logs for all instruments used for sample analyses for this project shall be provided for all days on which analysis was performed. The raw data for each analysis shall include measurement print outs and quantitation reports for each instrument used. Records of absorbance, titrimetric, or other measurements for wet chemical analysis shall be recorded. All raw data will be presented on standard forms and accompanied by the instrument output. Use of CLP standard forms is not necessary, however submission of standard instrument output alone is unacceptable to satisfy the requirements for raw data packages.

5.5.3 Electronic Data Deliverables and Electronic Data Validation

Use of electronic data deliverables and electronic data validation, will promote objectivity, substantially reduce costs, and facilitate data exchange. This will also allow data validators to focus and spend more time on inspection of raw data. The electronic data deliverables requirements and electronic data validation procedures will be specified in the CDQMP. The laboratory is encouraged to perform a QC check of the electronic data file for accuracy. The laboratory has not met its obligation of submission of the EDDs if errors are identified. The required turnaround time is for complete and accurate EDD deliverables.

5.6 DATA VALIDATION REPORTS

Unless directed by the CO, an independent data review specialist will review 100% of the data generated for the program. Approximately, 10% of the data will be validated to equivalent of EPA Level 4 (raw data packages) and 90% will be validated to equivalent of EPA Level 3 (definitive data with QC summaries). The CDQMP will specify the data validation procedures. For each sample delivery group, a data validation report shall be generated. The content and format shall confirm to the specifications outlined in this section.

5.6.1 Project Scope Summary

This section shall reference the guidance documents used to review the data, USACE contract number, the site(s), the sampling event or field program, he contractors responsible for the work (i.e., the primary contractor, the laboratory, and

data validator), review date, sample delivery group identification, sample numbers, matrix, and collection date (s).

5.6.2 Data Validation Requirements

This section shall reference the data validation requirements, documents used for validation, criteria used, and parameters reviewed for EPA Level 3 and 4 equivalent data packages.

5.6.3 Data Validation Qualifiers and Codes

This section shall consist of a table with the data validation qualifier flags and any explanation codes (i.e., "-" negative bias due to low surrogate recoveries). The data validation report must provide definitions for the explanation codes.

5.6.4 Summary of Qualified Data

This section shall consist of a brief summary of the data validation findings for each analysis by method and matrix. Each criteria reviewed will be outlined, the data validation results will be presented, and the potential impact on data quality will be discussed. A summary table will be provided which presents all data generated with qualification flags presented with explanation codes provided. In addition each nonconformance will be designated whether it is a contract compliance issue.

5.7 DAILY QUALITY CONTROL REPORTS (DQCR's):

The Contractor shall prepare a DQCR for each day of the project. Information contained in this report shall include, as a minimum:

- location of work;
- weather conditions;
- work performed;
- results of any inspections performed;
- problems identified and associated corrective actions taken,
- any instructions received from government personnel for retesting;
- types of tests performed, the individuals performing the tests and test results;
- general comments;
- calibration procedures; and
- the Contractor's certification.

The Contract Laboratory shall perform DQCR's for each day of laboratory activities associated with this project summarizing daily quality control activities. The laboratory DQCR's may be limited to out-of-control data events and corrective action taken to resolve them. The QAPP shall specify the content of field and laboratory DQCR's. DQCR's shall be submitted to the CO on a weekly basis. The QAPP shall acknowledge the requirement for these reports and describe the content of them as reflected in these specifications.

5.8 QUALITY CONTROL SUMMARY REPORT (QCSR)

A Quality Control Summary Report shall be prepared for each delivery order for this contract. Issues covered in this report shall include the quality control practices employed in execution of the contract and a discussion of all data points, which may have been compromised, and their impact on the Data Quality Objectives or remedial decisions. Normally the QCSR would be prepared at the end of a project. For delivery orders incorporating field work involving sampling and analysis extending beyond thirty calendar days one QCSR shall be prepared for each thirty day period. The QCSR shall be received by the CO within 65 calendar days of the initiation of fieldwork involving sampling and analysis and again at thirty-day intervals thereafter. The QAPP shall acknowledge this requirement and describe the content of this report as detailed in these specifications. If the QCSR is unacceptable relative to the requirements of these specifications the Contractor shall revise the document to the satisfaction of the Government within 15 days of receipt of comments from the USACE CO. The QCSR shall be prepared by compiling information relative to the project according to the following

outline:

5.8.1 Project Scope
This section will address the sampling and field event covered by the QCSR.

5.8.2 Project Description
This section shall describe the site background and investigation strategy.

5.8.3 Sampling Procedures
This section shall focus on deviations from planned activities and any field work variances to be listed in an Appendix.

5.8.4 Quality Control Activities
The following information will be provided: numbers and types of QC samples collected, discussion of QC problems encountered, and discussion field and laboratory quality control activities. This section should focus on the rationale and documentation for any deviations from planned activities. The Contractor shall include summaries of field and laboratory oversight activities, provide a discussion of the reliability of the data, discuss QC problems encountered, and a summary of the evaluation of data quality for each analysis and matrix as indicated by the laboratory QC data and any other relevant findings.

5.8.5 Analytical Procedures
The analytical and preparation procedures used will be briefly described.

5.8.6 Chemical Data Quality Assessment

A. Summary Data Quality Assessment (assessment of data based on project DQOs)
Field Duplicate Result Table (with difference factors and acceptability result)
Detected Analyte Table (hits Only with qualifiers)
Results Summary Table (all analytical results with qualifiers)
Rejected Results Table (with expanation codes and contract compliance indication)

Results for field duplicates shall be discussed in the qualitative description of completeness. The field duplicate data will be compared with the criteria stipulated in the CDQMP. This criteria will be consistent with the Shell Guidance and the USACE, CRREL Special Report No. 96, Comparison Criteria for Environmental Chemical Analyses of Split Samples Sent to Different Laboratories – Corps of Engineers Archived Data, Grant, C.G., Jenkins, T.F., and Mudambi, A.R., USACE Cold Regions & Environmental Research Laboratory, Hanover NH, May, 1996.

Only validated data will be presented in the QCSR and final reports. This section will include a presentation and evaluation of the data to include an overall assessment of the quality of the data for each method and matrix. Any nonconformances, which were identified during data validation, are discussed and impact on the data quality and data usability is discussed. The discussion should include qualitative and quantitative assessments of completeness as described in this document. QC failure should be presented by graphical representation which include histograms and pie charts regarding the data quality, nonconformances identified during data validation, and completeness results.

The Contractor shall describe statistical procedures used in the assessment of data. The Contractor shall discuss any results reflecting significant deviations.

B. Completeness Summary (analytical, contract compliance, technical, and field sampling completeness)
The contract specification requires the calculation of acceptable sample results to all sample results. This doesn't

account for estimated results that may still be useable for project decision making. The four calculations of completeness are required.

$$\text{Contract Completeness} = \frac{\text{\# contract compliant results}}{\text{\# results reported}} \times 100$$

$$\text{Analytical Completeness} = \frac{\text{\# unqualified results}}{\text{\# results reported}} \times 100$$

$$\text{Technical Completeness} = \frac{\text{\# useable results}^{\dagger}}{\text{\# results reported}} \times 100$$

$$\text{Field Sampling Completeness} = \frac{\text{\# samples collected}}{\text{\# samples planned}} \times 100$$

† Estimated results considered as useable for project decision making

The minimum goals for completeness are as follows: 1) Contract = 100%, 2) Analytical = 90% or greater, 3) Technical = 95% or greater, and 4) Field = 100%. The goal for holding times is 100%. Estimated results are treated as usable results for technical completeness. These are considered minimum goals; however, if based on DQOs different goals are established they will be presented in the site-specific SAP. Completeness calculations include QC samples (QA splits, field duplicates, etc.)

A completeness summary will be provided in tabular and graphical format presenting the relevant analyses, the total number of samples analyzed for each method, the number of samples qualified for any reason, the number of samples associated with contract compliance failure, the determination of "analytical completeness" (determined relative to the number of samples qualified for any reason), and "contract compliance completeness" (determined relative to the number of samples qualified for contract compliance failure). Routinely, the value reported for “contract compliance completeness” should be at or near 100% while the value reported for “analytical completeness” may be less than that as a function of matrix effects. Each metal and organic compound is considered a separate analytical parameter rather than considering all of the analytes or compounds in a single analytical category for the purpose of calculating completeness. A single number for completeness in each category for each analysis will be presented to describe the overall data quality. A complete sample will be considered a sample for which all QC parameters are within acceptable limits. Contractual QC elements include: holding time, calibration, laboratory blanks, LCS, MS/MSD, surrogates, etc.). Analytical QC elements include the contractual QC elements and the defined elements that were reviewed and qualified, as defined in the QAPP. There will be overlap between the contractual and analytical QC elements.

5.8.7 Conclusions and Recommendations

Qualified Results Summary Chart and Completeness Summary Chart. A summary of field or analytical procedures that should be changed or modified to better characterize chemical contamination in future work efforts at sites covered by the contract.

5.8.8 References

This section will provide those references (project and guidance) used to review the data.

Appendices-Data Validation Report, Data Qualifier Definitions, and Field Work Variance

All internal QC data (splits, duplicates, etc.) generated during the course of the project must be included in the QCSR.

The QC data presentation shall include tabular summaries correlating sample identifiers with all blank results, matrix spike results (MS/MSD or MS as appropriate), surrogate results, duplicate results (MS/MSD or S/SD as appropriate), LCS results, and batch identifiers. Calibration data shall be included in this summary whenever the results exceed the limits of acceptability. The quantitative description of completeness will be performed by considering aspects related to data quality and to contract compliance.

Where sample results are negatively impacted by adverse quality control criteria the QCSR shall contain a list of the affected sample results for each analyte (indexed by method and matrix) including the appropriate data qualifier flag (J, UJ, R, etc.) Data flags and conventions for flagging of data shall be consistent with those described in the CDQMP. In order to compile this summary all quality control data, including calibration results, must be reviewed following the conventions specified in tables to be included in the CDQMP. Content requirements for these tables of data validation conventions and qualifiers are described in Sections 5.6 and 5.2.5, respectively.

5.9 NON-ROUTINE OCCURRENCES REPORTS

The Contractor shall send written reports of all significant non-routine occurrence events to the CO within 48 hours of occurrence of non-routine events for field and laboratory work.

5.9.1 Project Scope

This section will address the sampling and field event covered by the Non-Routine Occurrences Report. This section shall also briefly describe the site background and investigation strategy.

5.9.2 Problems Identified, Corrective Actions Taken, Instructions from USACE Contracting Officer.

These reports shall identify the problem, corrective action, and verbal/written instructions from the USACE CO to Contractor personnel regarding sampling or reanalysis. Significant events are occurrences impacting on cost of work, schedule of work, quality of work, and quality of environmental analytical data.

5.10 DATA REPORT FOR THE CHEMICAL QUALITY ASSURANCE REPORT

It is the responsibility of the USACE PM to report any significant discrepancies between the primary and QA split results to the Contract Laboratory. In the event of such an occurrence, the Contract Laboratory must initiate an investigation into possible reasons for the discrepancy, and submit a plan to resolve the problem. All such activities shall be considered as non-conformance events, and be supported by the appropriate documentation. Such investigation and correction activities shall be performed at no additional cost to the Government. These requirements shall be acknowledged in the QAPP.

5.10.1 Final Certificates of Analysis

The Consultant shall provide the USACE PM with a copy of the final comprehensive certificate of analysis and a copy of the final SAP for use in preparation of the Chemical Quality Assurance Report (CQAR) by Sacramento District.

5.10.2 Final Sampling and Analysis Plan

The final SAP shall be provided to the QA Lab directly by the Consultant immediately after its approval by the USACE PM.

5.10.3 QA Split and Primary Sample Cross Reference Analytical Summary

The Data Report shall include a summary, which identifies the sample(s) which were split for QA testing, and the corresponding primary sample(s). Table 5-6 presents the QA comparison criteria for USACE projects.

5.10.4 CHEMICAL QUALITY ASSURANCE REPORT

The CQAR may be incorporated into the final Project Report at the discretion of the USACE technical team.

TABLE 5-1
DATA FLAGGING CONVENTION FOR METALS ANALYSES

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects		Nondetects	
		Non Biased	Biased		
HOLDING TIMES	1) Holding time exceeded by 2 times or less	J	J-	UJ	Sample
	2) Holding time exceeded by greater than 2 times	J	J-	R	
INITIAL CALIBRATION	1) r < 0.995	J	J	UJ	All samples associated with initial calibration (Run Batch)
INITIAL CALIBRATION VERIFICATION (ICV)	1) % Recovery > 110% but ≤ 125% (Hg, % Recovery > 120% but ≤ 135%)	J	J+	No qual.	All samples associated with initial calibration verification (Run Batch)
	2) % Recovery > 125% (Hg, % Recovery > 135%)	R	R	No qual.	
	3) % Recovery < 90% but ≥75% (Hg, % Recovery < 80% but ≥ 65%)	J	J-	UJ	
	4) % Recovery < 75% (Hg, % Recovery < 65%)	J	J-	R	

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects		Nondetects	
		Non Biased	Biased		
CALIBRATION VERIFICATION	1) % Recovery > 110% but ≤ 125% (Hg, % Recovery > 120% but ≤ 135%)	J	J+	No qual.	All samples associated with continuing calibration (Analysis Batch)
	2) % Recovery > 125% (Hg, % Recovery > 135%)	R	R	No qual.	
	3) % Recovery < 90% but ≥ 75% (Hg, % Recovery < 80% but ≥ 65%)	J	J-	UJ	
	4) % Recovery < 75% (Hg, % Recovery < 65%)	J	J-	R	
METHOD BLANK CONTAMINATION	Sample results less than or equal to 5 times the blank contamination	U	U	No qual.	All samples in the same Preparation Batch
MATRIX SPIKE RECOVERY	1) % Recovery < CL but ≥ 30%	J	J-	UJ	All samples in the same Method Batch
	2) % Recovery <30%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
	4) RPD > CL	J	J	UJ	
LABORATORY CONTROL SAMPLE RECOVERY	1) % Recovery < CL but ≥ 50%	J	J-	UJ	All samples in the same Preparation Batch
	2) % Recovery <50%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
	4) RPD > CL	J	J	UJ	

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects		Nondetects	
		Non Biased	Biased		
REPORTING LIMITS	1) Reporting limits not matching the project specified limits	No qual.	No qual. J	No qual.	Sample (noted in outlier report)
	2) Reported result less than the project reporting detection limit.	J		No qual.	Sample
FIELD DUPLICATES	RPD > CL	No qual.	No qual.	No qual.	Non-compliant results listed in the ADR outlier report
FIELD BLANKS EQUIPMENT BLANKS	Sample results within 5 times blank contamination	U	U	No qual.	All samples in the same sampling event

TABLE 5-2
DATA FLAGGING CONVENTION FOR ION CHROMATOGRAPHY AND WET CHEMISTRY ANALYSES

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
HOLDING TIMES	1) Holding time exceeded by 2 times or less	J	J-	UJ	Sample
	2) Holding time exceeded by greater than 2 times	J	J-	R	
COOLER TEMPERATURE	1) > 6 degrees Centrigrade	No qual.	No qual.	No qual	Noted on outlier report for samples shipped in affected cooler, if the associated method has temperature requirements.
	2) < 2 degrees Centigrade	No qual.	No qual.	No qual	
INITIAL CALIBRATION	1) %RSD > 20%	J	J	UJ	All samples associated with initial calibration (Run Batch)
	2) r < 0.995	J	J	UJ	
INITIAL CALIBRATION VERIFICATION (ICV)	1) % Recovery = 90-110%	J	J+	No qual.	All samples associated with initial calibration verification (Run Batch)
	2) % Recovery = 90 - 50%	J	J-	UJ	
	3) % Recovery < 50%	J	J-	R	
CALIBRATION VERIFICATION	1) % Recovery = 90-110%	J	J+	No qual	All samples associated with continuing calibration (Analysis Batch)
	2) % Recovery = 90-50%	J	J-	UJ	
	3) % Recovery < 50%	J	J-	R	

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
METHOD BLANK CONTAMINATION	Sample results less than or equal to 5 times the blank contamination	U	U	No qual.	All samples in the same Preparation Batch
MATRIX SPIKE RECOVERY	1) % Recovery < CL but ≥ 30%	J	J-	UJ	All samples in the same Method Batch
	2) % Recovery <30%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
	4) RPD > CL	J	J	UJ	
LABORATORY CONTROL SAMPLE RECOVERY	1) % Recovery < CL but ≥ 50%	J	J-	UJ	All samples in the same Preparation Batch
	2) % Recovery <50%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
	4) RPD > CL	J	J	UJ	
REPORTING LIMITS	1) Reporting limits not matching the project specified limits	No qual.	No qual.	No qual.	Sample (noted in outlier report)
	2) Reported result less than the project reporting detection limit.	J	J		Sample
FIELD DUPLICATES	1) RPD > CL	No qual.	No qual.	No qual.	Non-compliant results listed in the ADR outlier report
FIELD BLANKS EQUIPMENT BLANKS	Sample results within 5 times blank contamination	U	U	No qual.	All samples in the same sampling event

TABLE 5-3
DATA FLAGGING CONVENTION FOR GC ANALYSES

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects		Nondetects	
		Non Biased	Biased		
HOLDING TIMES (Extraction/Analysis)	1) Holding time exceeded by 2 times or less	J	J-	UJ	Sample
	2) Holding time exceeded by greater than 2 times	J	J-	R	
COOLER TEMPERATURE	1) > 6 and ≤10 degrees Centigrade	J	J-	UJ	All samples shipped in the affected cooler. (Shipping Batch)
	2) >10 degrees Centigrade	J	J-	R	
	3) < 2 degrees Centigrade	No qual.	No qual.	No qual.	
INITIAL CALIBRATION	1) %RSD > 20%	J	J	UJ	All samples associated with initial calibration (Run Batch)
	2) r < 0.995	J	J	UJ	
INITIAL CALIBRATION VERIFICATION (ICV)	1) % Difference > +25%	J	J+	No qual.	All samples associated with initial calibration verification (Run Batch)
	2) % Difference < -25% and ≥ -50%	J	J-	UJ	
	3) % Difference < -50%	J	J-	R	
CONTINUING CALIBRATION (CCV)	1) % Difference > +15%	J	J+	No qual.	All samples associated with continuing calibration (Analysis Batch)
	2) % Difference < -15% and ≥ -50%	J	J-	UJ	
	3)% Difference < -50%	J	J-	R	

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects		Nondetects	
		Non Biased	Biased		
METHOD BLANK CONTAMINATION	1) Common lab contaminant results less than or equal to 10 times the blank contamination	U	U	No qual.	All samples in the same Preparation Batch
	2) Other compound results less than or equal to 5 times the blank contamination	U	U	No qual.	
SURROGATE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	Sample
	2) % Recovery <10%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
MATRIX SPIKE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	Parent Sample
	2) % Recovery <10%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
	4) RPD > CL	J	J	UJ	
LABORATORY CONTROL SAMPLE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	All samples in the same Preparation Batch
	2) % Recovery <10%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
	4) RPD > CL	J	J	UJ	
REPORTING LIMITS	1) Reporting limits not matching the project specified limits.	No qual.	No qual.	No qual.	Sample (noted in outlier report)
	2) Results reported below the project reporting detection limit.	J	J	No qual.	Sample

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects		Nondetects	
		Non Biased	Biased		
FIELD DUPLICATES	1) RPD > CL	No qual.	No qual.	no qual.	Non-compliant results listed in the ADR outlier report
FIELD BLANKS EQUIPMENT BLANKS	1) Common lab contaminant results within 10 times blank contamination	U	U	No qual.	All samples in the same sampling event
	2) Other lab contaminant results within 5 times blank contamination	U	U	No qual.	
TRIP BLANKS	1) Common lab contaminant results within 10 times blank contamination	U	U	No qual.	All samples in the same Shipping Batch
	2) Other lab contaminant results within 5 times blank contamination	U	U	No qual.	

TABLE 5-4
DATA FLAGGING CONVENTION FOR GC/MS ANALYSES

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
HOLDING TIMES (Extraction/Analysis)	1) Holding time exceeded by 2 times or less	J	J-	UJ	Sample
	2) Holding time exceeded by greater than 2 times	J	J-	R	
COOLER TEMPERATURE	1) > 6 and ≤10 degrees Centigrade	J	J-	UJ	All samples shipped in the affected cooler (Shipping Batch)
	2) >10 degrees Centigrade	J	J-	R	
	3) < 2 degrees Centigrade	No qual.	No qual.	No qual.	
INSTRUMENT TUNING	1) Ion abundance criteria not met	JN	JN	R	All samples associated to an initial calibration (Run Batch), if tune is associated to an initial calibration. All samples associated to a continuing calibration (Analysis Batch), if tune is associated to a continuing calibration.
INITIAL CALIBRATION	1) Average RRF < 0.05	J	J	R	All samples associated to the initial calibration (Run Batch)
	2) %RSD > 30%	J	J	UJ	
	3) r < 0.995	J	J	UJ	

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
INITIAL CALIBRATION VERIFICATION (ICV)	1) Average RRF < 0.05	J	J	R	All samples associated to the ICV (Run Batch)
	2) % Difference > +25%	J	J+	no qual.	
	3) % Difference < -25% and ≥ -50%	J	J-	UJ	
	4) % Difference < -50%	J	J-	R	
CONTINUING CALIBRATION VERIFICATION (CCV)	1) Average RRF < 0.05	J	J	R	All samples associated to the CCV (Analysis Batch)
	2) % Difference > +25%	J	J+	no qual.	
	3) % Difference < -25% and ≥ -50%	J	J-	UJ	
	4) % Difference < -50%	J	J-	R	
METHOD BLANK CONTAMINATION	1) Common lab contaminant and tentatively identified compound (TIC) results less than or equal to 10 times blank contamination	U	U	No qual.	All samples in the same Preparation Batch as the method blank
	2) Other compound results less than or equal to 5 times blank contamination	U	U	No qual.	
INTERNAL STANDARDS	1) Area counts must not vary by more than a factor of 2 (+/-50%)	JN	JN	UJ	Sample (use of professional judgement is recommended based on evaluation of mass spectra).
	2) The retention time must no vary more than +/- 30 seconds from the retention time of the associated 12 hour calibration standard	JN	JN	UJ	

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
SURROGATE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	Sample
	2) % Recovery <10%	J	J-	R	Note: For semivolatile analysis, two or more surrogates in a fraction must be out of criteria for qualification unless recovery < 10%.
	3) % Recovery > CL	J	J+	no qual.	
MATRIX SPIKE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	Parent Sample
	2) % Recovery <10%	J	J-	R	
	3) % Recovery > CL	J	J+	no qual.	
	4) RPD > CL	J	J	UJ	
LABORATORY CONTROL SAMPLE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	All samples in the same Preparation Batch as the LCS
	2) % Recovery <10%	J	J-	R	
	3) % Recovery > CL	J	J+	no qual.	
	4) RPD > CL	J	J	UJ	
REPORTING LIMITS	1) Reporting limits not matching the project specified limits	No qual.	No qual.	No qual.	Sample (noted on outlier report)
	2) Results reported below the project reporting detection limit.	J	J	No qual.	
FIELD DUPLICATES	1) RPD > CL	No qual.	No qual.	no qual.	Noted in outlier report

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
FIELD BLANKS EQUIPMENT BLANKS	1) Common lab contaminants and tentatively identified compound (TIC) results within 10 times blank contamination	U	U	No qual.	All samples in the same sampling event
	2) Other lab contaminant results within 5 times blank contamination	U	U	No qual.	
TRIP BLANKS	1) Common lab contaminant and tentatively identified compound (TIC) results within 10 times blank contamination	U	U	No qual.	All samples in the same Shipping Batch as the trip blank
	2) Other lab contaminant results within 5 times blank contamination	U	U	No qual.	

TABLE 5-5
DATA FLAGGING CONVENTION FOR HPLC ANALYSES

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
HOLDING TIMES (Extraction/Analysis)	1) Holding time exceeded by 2 times or less	J	J-	UJ	Sample
	2) Holding time exceeded by greater than 2 times	J	J-	R	
COOLER TEMPERATURE	1) > 6 and ≤10 degrees Centigrade	J	J-	UJ	All samples shipped in the affected cooler. (Shipping Batch)
	2) >10 degrees Centigrade	J	J-	R	
	3) < 2 degrees Centigrade	No qual.	No qual.	No qual.	
INITIAL CALIBRATION	1) %RSD > 20%	J	J	UJ	All samples associated with initial calibration (Run Batch)
	2) r < 0.995	J	J	UJ	
INITIAL CALIBRATION VERIFICATION (ICV)	1) % Difference > +15%	J	J+	No qual.	All samples associated with initial calibration verification (Run Batch)
	2) % Difference < -15% and ≥ -50%	J	J-	UJ	
	3) % Difference < -50%	J	J-	R	
CONTINUING CALIBRATION (CV)	1) % Difference > +15%	J	J+	No qual.	All samples associated with continuing calibration (Analysis Batch)
	2) % Difference < -15% and ≥ -50%	J	J-	UJ	
	3) % Difference < -50%	J	J-	R	
METHOD BLANK CONTAMINATION	1) Sample results less than or equal to 5 times the blank contamination.	U	U	No qual.	All samples in the same Preparation Batch

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
SURROGATE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	Sample
	2) % Recovery <10%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
MATRIX SPIKE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	Parent Sample
	2) % Recovery <10%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
	4) RPD > CL	J	J	UJ	
LABORATORY CONTROL SAMPLE RECOVERY	1) % Recovery < CL but ≥ 10%	J	J-	UJ	All samples in the same Preparation Batch
	2) % Recovery <10%	J	J-	R	
	3) % Recovery > CL	J	J+	No qual.	
	4) RPD > CL	J	J	UJ	
REPORTING LIMITS	3) Reporting limits not matching the project specified limits.	No qual.	No qual.	No qual.	Sample (noted in outlier report)
	4) Results reported below the project reporting detection limit.	J	J	No qual.	Sample
FIELD DUPLICATES	1) RPD > CL	No qual.	No qual.	No qual.	Non-compliant results listed in the ADR outlier report
FIELD BLANKS EQUIPMENT BLANKS	1) Common lab contaminant results within 10 times blank contamination	U	U	No qual.	All samples in the same sampling event
	2) Other lab contaminant results within 5 times blank contamination	U	U	No qual.	

QUALITY CONTROL ITEM	EVALUATION	DATA QUALIFIER FLAG			SAMPLE(S) QUALIFIED
		Detects			
		Non Biased	Biased	Nondetects	
TRIP BLANKS	1) Common lab contaminant results within 10 times blank contamination	U	U	No qual.	All samples in the same Shipping Batch
	2) Other lab contaminant results within 5 times blank contamination	U	U	No qual.	

TABLE 5-6
QUALITY ASSURANCE SPLIT RESULTS COMPARISON CRITERIA

Matrix	Parameter	Disagreement	Major Disagreement
All	All	>5X difference when one result is <DL	>10X difference when one result is <DL
All	All	>3X difference when one results is <RL	>5X difference when one results is <RL
Water	All except TPH	>2X difference	>3X the difference
Soil	All except metals, VOCs, BTEX, and TPH	>4X difference	>5X difference
Soil	Metals	>2X difference	>3X difference
Water and Soil	TPH	Arbitrary (suggest >3X difference)	Arbitrary (suggest >5X difference)
Soil	VOCs and BTEX	Arbitrary (suggest >5X difference)	Arbitrary (suggest >10X difference)

Reference: USACE, 1996
BTEX: benzene, toluene, ethylbenzene, and total xylenes
DL: detection limit
RL: reporting limit
TPH: total petroleum hydrocarbons
VOCs: volatile organic compounds

6.0 GENERAL CONTRACT SPECIFICATIONS

6.1 CONTRACT LABORATORY VALIDATION

Prior to collection or analysis of any environmental samples, the Contract Laboratory shall be validated by the USACE HTRW Center of Expertise (HTRW-CX) in accordance with the requirements of USACE EM 200-1-1 as well as the State where delivery order tasks will be executed (California, Utah, Nevada, and Arizona). Laboratories are validated for each environmental matrix and each specific analytical method to be employed. If the prime Contractor selects a laboratory which has a current (within eighteen months) validation for all analytes and matrices specific to its project, additional evaluation will not be necessary. The CO shall be contacted to verify the status of the contract laboratory. If the prime Contractor selects a laboratory which does not have a current validation, the laboratory shall be validated prior to approval of the project specific SAP. Commercial laboratory validation procedures can be obtained from the CO (Note: certification by HTRW-CX may take as long as three months). Samples will not be subcontracted to another laboratory without knowledge and approval of the CO and the second laboratory must be validated for the parameters concerned. The Contractor (or Contract Laboratory, as applicable) shall be responsible for the following:

6.1.1 Selecting a Lab

Laboratories performing analyses for this program will be selected based on their ability to maintain USACE validation, and State certification, their past performance as well as technical and management practices relevant to the attainment of project-specific DQOs. Laboratory certification and validation documentation shall be included in the project-specific SAP. The project laboratories shall agree to the method performance, deliverables, and documentation specifications in this specification and the site-specific SAP. It will be the responsibility of the Primary Contractor to assure compliance with is agreement to provide documentation of nonconformances and actions taken to correct deficiencies.

6.1.2 Laboratory Fraud

Laboratory fraud is defined as the deliberate falsification of analytical and quality assurance results, where failed method and contractual requirements are made to appear acceptable during reporting. Since the early 1990's several major fraud cases have come to light. The U.S. Environmental Protection Agency (US EPA) Office of Inspector General (OIG) reported in its draft audit report of nine Superfund sites in three US EPA regions dated October 28, 1996, that 11 million plus dollars were spent on rejected analyses, re-sampling, and associated costs that could have been avoided through the use of effective quality assurance oversight systems. Repercussions include the compounded losses in time, resources and monies spent to pursue damages and reassess decisions made with fraudulent data is an enormous vulnerability that regulators, decision makers, and laboratory users cannot afford.

In response to these findings, DOD and EPA combined their efforts to develop strategies to eliminate laboratory fraud. The Army has placed a high priority of fraud detection and deterrence for the investigative efforts at Camp Ono due to the sensitivity and potential impact of investigative efforts undertaken in PRP cases. USACE has incorporated measures for fraud deterrence and detection based on the document “Detection and Deterrence of Laboratory Fraud” published by the California Military Environmental Coordination Committee (CMECC). The measures incorporated into this project include the following:

- Development of Data Quality Objectives (Section 5.2.2.4)
- Identification of Quality Assurance and Quality Control Requirements (as a result of the specifications in Section 5.2.2.4)
- Laboratory Selection and Use of Phased Audits (Sections 6.1 and 5.2.4.2)
- PE Samples (Section 5.2.3.5.3)
- QA Split Sample Analysis (Section 5.3.2.3)
- Laboratory Performance Histories (Section 6.1.1)
- Data Validation (Section 5.2.5.2)

- Electronic Data and Tape Audits (Section 5.2.4.4)
- Quality Assurance Officer (Section 5.2.4.5)
- Electronic Data Deliverables (Section 5.5)

6.2 FACILITIES AND PERSONNEL

Provide all laboratory facilities and qualified personnel for sample analyses, and provide access to work, as required.

6.3 STATEMENT OF WORK AND ETHICAL CONDUCT

The Statement of Work for laboratories will include the following:

A) All laboratories will have a company ethics policy read and signed by employees. The laboratory shall have arrangements to ensure that its personnel are free from any commercial, financial, and other pressures, which might adversely affect the quality of their work (ISO Guide 25, and NELAC Quality System Standard).

B) Training will be provided to laboratory staff on the ethics of generating analytical data and for meeting the technical requirements established in the method. Training files on the analyst will be maintained by the laboratory. These files will contain signatures of the analyst certifying that they have received the training. The ethics training received or to be received by the staff will be documented in an approved QAPP or FSP. Certificates of completion will be signed annually.

C) Specific SOPs will be drafted for each method to be performed by the laboratory. These SOPs will identify the specific corrective measures to be performed will problems occur with the analyses. These measures will be strictly adhered to; no deviations will be allowed without documentation. To ensure consistency in performing a method, which may permit different options, the SOP must document the specific activities the analyst will perform.

D) The laboratory's quality system must include "arrangements for ensuring that the laboratory review all new work to ensure that it has the appropriate facilities and resources before commencing such work" (ISO Guide 25, 5.2.I).

E) The laboratory management must provide adequate resources and assign sufficient authority and independence to line management and to staff to enable them to plan, implement, assess, and improve the laboratory's quality system effectively (ANSI/ASQC E-4, 2.1.1).

6.4 USE OF MORE THAN ONE LABORATORY

Several contract laboratories may be used for the program. For each effort, USACE will contract with an independent quality assurance laboratory in order to confirm the performance of the contractor's laboratory. Although submitting samples to more than one laboratory does not prevent fraud from occurring, this practice can detect problems that otherwise may not be apparent. If different laboratories repeatedly provide divergent results in the absence of mitigating factors, further investigation will be initiated by the Primary Contractor. A well-designed split-sample strategy can be used to ensure decisions are supported by more than one laboratory, and are recommended especially in cases where critical decisions are being made. Different laboratories that provide similar results build confidence for the data users that the data are reliable.

6.5 SAMPLE HANDLING

Furnish labor, equipment and facilities to obtain and handle samples at the project site, to facilitate inspections and analyses, and to provide storage, preservation (including refrigeration) and cooling of the samples, as necessary.

6.6 SAMPLE CUSTODY
Provide for and ensure that transportation, chain of custody, and ultimate disposal of samples takes place in accordance with USACE/EPA procedures.

6.7 DATA MANAGEMENT
Data management takes place at varied levels throughout the range of the project. The CDQMP shall identify the individuals responsible for data management, the activities involved with data management, and the minimum required credentials associated with these tasks. The CDQMP shall provide for documentation and data management of the analytical results. A plan for data management will be established for each project handling large amount of data (as defined in the CDQMP).

The CDQMP shall set the minimum standards for electronic data management. At a minimum, the field and laboratory data to be captured electronically will be specified. For example, the data management plan shall include the formulas used, the computer programs used, which data transfers are electronic or manual, and validation steps. All computer programs, spreadsheets, and databases will be validated as to the accuracy of data management before being used on project work. All data acquired electronically will be transferred electronically to reduce errors inherent in manual data manipulation. Data entered, transferred, or calculated by hand will be spot checked for the accuracy of the transfer and the calculations, preferably by someone who did not perform the original entries or calculations. These checks will be documented.

The CDQMP shall specify the design, implementation, and maintenance of program and project databases. The electronic data deliverable file requirements for the laboratories (i.e., fields, structure, definitions, valid values, format, etc.) shall be provided in the CDQMP. The data to be entered into the database includes (at a minimum): sample locations, field drilling data, well construction details, groundwater elevation surveys, product recovery data, chemical analyses data, and field and laboratory quality control sample results. The hardware and software necessary for the program will be specified in the CDQMP. The database platform, structure, and software applications will be evaluated and appropriate programming applications will be designed or modified for the specific uses. This information will be specified in the CDQMP.

6.8 INSPECTIONS, SAMPLING, AND ANALYSIS
Comply with specified standards and ascertain compliance of materials with requirements of the Contract Documents.

6.9 CALIBRATIONS
Provide for calibration of equipment.

6.10 QUALITY ASSURANCE SAMPLE
Provide for laboratory QA samples including splits and duplicates.

6.11 SAMPLE CONTAINERS
Provide for clean sample containers and sample preservation.

6.12 RECORD KEEPING
Maintain internal Record keeping in accordance with good laboratory practices and the provisions of these specifications.

6.13 ACCESS TO DATA
USACE shall have direct access to all data produced by the Contract Laboratory at all times. At any time USACE representatives shall be granted access to data that is currently available at the laboratory for sample analyses for USACE

projects with or without the prior consent of the Contractor. If the Contract Laboratory has an electronic system for delivery or early review of data USACE shall be allowed electronic access to data with or without the consent of the Contractor. The Contractor shall instruct the Contract Laboratory in writing prior to initiation of sampling and analysis that USACE representatives shall have unrestricted access to data and the USACE CO shall be provided with a copy of this communication.

6.14 LATE DELIVERY OF DATA

Late delivery of data will result in a reduction in payment for services related to sample analysis. Data packages are due within 21 days of the time of sampling and will be delivered to the Contractor and the USACE CO concurrently. If analytical data packages are not received in the Sacramento District offices at the specified time 5% of the total contract amount related to sample analyses (for the corresponding sample delivery groups) will be credited to the Government. At the end of the first week and for each week thereafter an additional 10% of the total contract amount related to sample analyses will be credited to the Government up to a maximum of 35% of the total delivery order amount related to sample analyses.

6.15 REJECTION OF DATA

Data will be screened for contract compliance. Failure to execute specific actions related to sampling or analysis required by this contract will result in rejection of data for the corresponding samples. At a minimum the Government will be credited for the cost of sampling and/or analytical work that is rejected for contract compliance failure. Alternatively, the USACE CO can require re-sampling and re-analysis or re-issuance of deliverables at no additional cost to the Government for sampling or analytical work that is rejected for contract compliance failure.