

# **FINAL QUALITY ASSURANCE PROJECT PLAN**

**Vapor Intrusion Investigation, Groundwater Monitoring  
Activities, and Well Abandonment  
at  
Air Force Plant 59  
Johnson City, New York**

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### QUALITY ASSURANCE PROJECT PLAN VAPOR INTRUSION INVESTIGATION, GROUNDWATER MONITORING ACTIVITIES, AND WELL ABANDONMENT AT AFP 59

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## LIST OF ACRONYMS AND ABBREVIATIONS

A2LA	American Association of Laboratory Accreditation
AAB	AFCEE Analytical Batch
AES	Atomic Emission Spectroscopy
AFCEE	Air Force Center for Engineering and the Environment
AFP	Air Force Plant
amu	Average Measured Mass
ASTM	American Society for Testing and Materials
ARAR	Applicable or Relevant and Appropriate Requirements
BFB	4-Bromofluorobenzene
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CL	Control Limit
COC	Chain of Custody
COD	Coefficient of Determination
DEQPPM	Defense Environmental Quality Program Policy Memorandum
DERP	Defense Environmental and Restoration Program
DFTPP	Decafluorotriphenylphosphine
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EICP	Extracted Ion Current Profile
ERPIMS	Environmental Resources Program Information Management System
FID	Flame Ionization Detector
FSP	Field Sampling Plan
GALP	Good Automated Laboratory Practices
GCD	Guidance for Contract Deliverables
GC	Gas Chromatography
GFAA	Graphite Furnace Atomic Absorption
GRO	Gasoline Range Organic
HPLC	High Performance Liquid Chromatography





## **LIST OF ACRONYMS AND ABBREVIATIONS (CONTINUED)**

ICAL	Initial Calibration
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
IDL	Instrument Detection Limit
IRP	Installation Restoration Program
IS	Internal Standard
LCL	Lower Control Limit
LCS	Laboratory Control Sample
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NELAC	National Environmental Laboratory Accreditation Conference
NIST	National Institute Standards and Technology
NTU	Nephelometric Turbidity Units
NYSDEC	New York State Department of Environmental Conservation
ORP	Oxidation-Reduction Potential
PARCCS	Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity
PFTBA	Perfluorotributylamine
PID	Photoionization Detector
ppbv	Parts per Billion by Volume
PQO	Project Quality Objective
PT	Proficiency Testing
QAPP	Quality Assurance Project Plan
QA	Quality Assurance
QC	Quality Control
RCA	Recommendations for Corrective Action
RCRA	Resource Conservation and Recovery Act
RF	Response Factors
RI/FS	Remedial Investigation/Feasibility Study
RL	Reporting Limit
RPD	Relative Percent Difference
RRT	Relative Retention Time
RSD	Relative Standard Deviation





## **LIST OF ACRONYMS AND ABBREVIATIONS (CONTINUED)**

SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SDG	Sample Delivery Group
SIM	Selected Ion Monitoring
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	Spill Prevention Control and Countermeasure
SVOCs	Semivolatile Organic Compounds
S	Standard Deviation
TCE	Trichloroethene
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compounds
UCL	Upper Control Limit
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
WP	Work Plan



## 1.0 INTRODUCTION

This *Final Quality Assurance Project Plan (QAPP)* presents in specific terms the policies, organization, functions, and quality assurance/quality control (QA/QC) requirements designed to achieve the data quality goals described in the approved project-specific *Work Plan (WP)*. It establishes the analytical protocols and documentation requirements to ensure the data are collected, reviewed, and assessed in a consistent manner to meet the overall project goals, and that the data are scientifically valid and defensible. This *QAPP* guidance presents, in specific terms, the policies, organization, functions, and QA/QC requirements designed to achieve the data quality goals to be described in the approved *Sampling and Analysis Plan (SAP)*. This *QAPP* and *Field Sampling Plan (FSP)*, also developed using Air Force Center for Engineering and the Environment (AFCEE) guidance, shall constitute, by definition, the project *SAP*.

The United States Environmental Protection Agency (USEPA) QA policy requires a *QAPP* for every monitoring and measurement project mandated or supported by the USEPA through regulations, contracts, or other formalized means not currently covered by regulation. Guidelines followed in the preparation of this plan are set out in the USEPA *Guidance for QAPPs*, (QA/G-5, December 2002), *Requirements for QAPPs* (EPA QA/R-5, March 2001), and *Guidance for the Data Quality Objectives (DQOs) Process* (EPA QA/G-4, August 2000). Other documents that have been used in the preparation of this *QAPP*, include the *Department of Defense Quality Systems Manual for Environmental Laboratories, Version 2, 2002*; *National Environmental Laboratory Accreditation Conference (NELAC) 2002 Standards (Effective 2004)*; *Uniform Federal Policy for QAPPs*; *Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs, Part 1, UFP-QAPP Manual*, Intergovernmental Data Quality Task Force, Draft Version 1, August 2003; *Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Development of DQOs (American Society for Testing and Materials [ASTM] D579)*; *Guidance for Conducting Remedial Investigations and Feasibility Studies Under Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), Interim Final* (USEPA, 1988); *Compendium of Superfund Field Operations Methods* (USEPA, 1987a); and *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (USEPA SW-846, Third Edition and its subsequent updates).

This project *QAPP*, developed under guidance of the *AFCEE QAPP*, shall be read by all essential staff participating in the work effort. This project *QAPP* shall be in the possession of the field teams and in all laboratories performing analytical services. All contractors and subcontractors shall be required to comply with the procedures documented in this project *QAPP* in order to maintain comparability and representativeness of the data produced.

Controlled distribution of this project *QAPP* shall be implemented by the prime contractor to ensure the current version is being used. A sequential numbering system shall be used to identify controlled copies of this project *QAPP*. Controlled copies shall be provided to applicable Air Force managers, regulatory agencies, remedial project managers, project managers, and QA coordinators. Whenever Air Force revisions are made or addenda added to this project *QAPP*, a document control system shall be put into place to assure (1) all parties holding a controlled copy of this project *QAPP* shall receive the revisions/addenda and (2) outdated material is removed from circulation. The



document control system does not preclude making and using copies of this project *QAPP*; however, the holders of controlled copies are responsible for distributing additional material to update any copies within their organizations.

The distribution list for controlled copies shall be maintained by the prime contractor.



## **2.0 PROJECT DESCRIPTION**

### **2.1 PROJECT BACKGROUND**

Air Force Plant (AFP) 59 is located in south-central New York in the Westover area of the Town of Union, Broome County, immediately west of Johnson City (mailing address); the site is about 3 miles west of the central business district of the City of Binghamton and about 4 miles east of the center of the Village of Endicott. The plant occupies 29.6 acres (including Parking Lot #5, located north of Main Street) and is situated in a highly urbanized area.

AFP 59 is a government-owned, contractor-operated facility. Remington Rand, Inc., the first manufacturer to occupy the plant, manufactured aluminum aircraft propellers at the plant from 1942 to 1945. The plant closed at the end of World War II and remained idle until April 1949, when it was reopened as an aircraft controls manufacturing facility. GE Aerospace was contracted to operate the facility and to direct manufacturing (primarily of parts for electro-mechanical aircraft control systems). Martin Marietta Aircraft Controls acquired GE Aerospace in 1993 and took over the operation of the facility and the manufacturing activities. BAE Systems currently manufactures flight control, laser, weapons control, internal navigation, and guidance systems at AFP 59.

Past and present activities at AFP 59 have generated a variety of waste products including cutting, lubricating, and coolant oils; degreasing agents; plating acids; caustics; chromium; cyanide solutions; and paint residues.

AFP 59 is listed as a Class 2 Site on the New York State Department of Environmental Conservation (NYSDEC) List of Inactive Hazardous Waste Disposal Sites (Site Code 7-04-020). A Class 2 Site is categorized as posing a "significant threat to the public health or environment." AFP 59 is not on the National Priorities List and is not under a Federal Facility Agreement.

### **2.2 PROJECT SCOPE AND OBJECTIVES**

This *QAPP* covers the following activities to be completed at AFP 59 and adjacent areas by AECOM and its subcontractors:

1. Vapor intrusion investigation in manufacturing building and adjacent areas at AFP 59
2. Off-site residential vapor-intrusion investigation
3. Soil Sampling
4. United States Geological Survey (USGS) well abandonment
5. Groundwater monitoring activities both on- and off-site

The objectives of this project are:

1. Determine if contaminants in the subsurface environment underneath and adjacent to the manufacturing building at AFP 59 pose a threat to the health of BAE employees



2. Determine if subsurface contaminants have migrated into residential areas adjacent to AFP 59 and if these contaminants pose a threat to the health of those living in said residences
3. Determine if the 2005 removal of trichloroethene (TCE)-contaminated soil from the East Basement of the manufacturing building had any impact on groundwater contaminant levels on-site.
4. Determine if contaminated groundwater has migrated off-site
5. Characterize the nature and extent of groundwater and soil contamination along the Fire Suppression Reservoir at AFP 59



### **3.0 PROJECT ORGANIZATION AND RESPONSIBILITY**

The project organization and responsibility discussion including (1) a project organizational chart identifying task managers and individuals responsible for performance of the project, (2) a list of names of all key participants, including organization names and telephone numbers for project, field, and laboratory QA officers, (3) a description of the authority given to each key participant with an emphasis on the authority of the key individuals to initiate and approve corrective actions, and (4) the role of regulatory representatives are included in Section 4.0 of the *FSP*.

All contractors and subcontractors are identified and the scope of their performance in the project is clearly defined. Subcontractors proposed to provide backup services are identified. An organizational chart, a list of key personnel, and the previously described descriptive text are included for each subcontractor in Sections 4.0 and 4.1 of the *FSP*.



## **4.0 PROJECT QUALITY OBJECTIVES AND ELEMENTS OF QUALITY CONTROL**

Project quality objectives (PQOs) specify the data type, quality, quantity, and uses needed to make decisions and are the basis for designing data collection activities. PQOs are developed by the contractor with inputs from various sources, including stakeholders, regulators, and environmental professionals, for site-specific applications and become incorporated into the overall project decision-making process. Some factors which may influence the process are existing site location data, the affected media and its current or projected use, local soil and groundwater chemistry, budget, time, and political constraints. Specific project objectives, as summarized in the *FSP*, provide the basis for decision diagrams which specify the quantity and quality of data to be collected and evaluated. An example of a decision diagram is provided to assist the contractor in the overall PQO development thought process and to illustrate the potential complexity and the interdisciplinary nature of the overall data collection program needed for quality, defensible data.

Specific measurement performance criteria for the data quality indicators (precision, accuracy, representativeness, completeness, comparability, and sensitivity [commonly referred to as "PARCCS parameters"]) are developed in the planning phase and become essential elements in the assessment of overall data quality. The goals of these indicators (field and laboratory) are incorporated into the overall PQOs and are included in this project *QAPP*.

### **4.1 DATA TYPES**

The two general types of data are screening data and definitive data. The uses and measurement performance criteria for each must be described in this project *QAPP*.

Screening data are analytical data that are of sufficient quality to support an intermediate or preliminary decision but must eventually be supported by definitive data before the project is complete. Screening data are often generated by rapid methods of analysis with less rigorous sample preparation, calibration, and/or QC requirements. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data may provide analyte identification and quantitation, although the quantitation may be relatively imprecise. Physical test methods (e.g., dissolved oxygen measurements, temperature, pH, moisture content, turbidity, conductance, etc.) have been designated by definition as screening methods (see Section 6).

Screening methods may be confirmed, as required in Section 3.2 of the *FSP*, by definitive analytical methods. Whenever screening data is confirmed by definitive analysis, comparability criteria must be established and documented in this project *QAPP* prior to data collection.

Confirmation samples shall be selected to include both detected and non-detected results from the screening method.

Definitive data are analytical data that are suitable for final decision-making. Often, they are generated using rigorous analytical methods (see Section 7) such as approved USEPA SW-846 reference methods. It is also possible, depending upon the PQOs, that definitive data can be generated in a mobile or off-site laboratory with prior approval of AFCEE. Definitive data are not





restricted in their use unless quality problems require data qualification. All screening and definitive methods to be used must be clearly presented in this project *QAPP*.

## **4.2 DATA QUALITY INDICATORS**

Measurement performance criteria should be determined for each matrix, analytical group, concentration level, and analyte, as appropriate. The criteria should relate to the data quality indicators (DQIs): PARCCS parameters. The DQIs are discussed in the following subsections. Procedures to measure data quality and the use of these indicators must be clearly presented in this project *QAPP*. AFCEE recommended measurement performance criteria for precision, accuracy, and sensitivity for each method and matrix are identified in Sections 6 and 7.

### **4.2.1 Precision**

Precision refers to the reproducibility of measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar or prescribed conditions. Precision reflects random error and may be affected by systematic error. It also reflects variation imposed by a given matrix.

Laboratory precision is measured by the variability associated with duplicate (two) or replicate (more than two) analyses. One type of sample that can be used to assess laboratory precision is the laboratory control sample or laboratory control sample (LCS). Multiple LCS analyses over the duration of the project can be used to evaluate the overall laboratory precision for the project. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch, rather the comparison is between LCSs analyzed in multiple batches.

Total precision is the measurement of the variability associated with the entire sampling and analytical process. It is determined by analysis of duplicate or replicate (split) field samples and measures variability introduced by both the laboratory and field operations. Field duplicate samples and matrix duplicate spiked samples shall be analyzed to assess field and laboratory precision. The precision is evaluated using the relative percent difference (RPD) between the duplicate sample results. The formula for the calculation of precision is provided in Table 4.2.1-1 as RPD. For replicate analyses, the relative standard deviation (RSD) is determined and used as the measure of precision. The formula for the calculation of RSD is provided in Table 4.2.1-1.

The required level of precision should be identified in the PQOs. AFCEE recommended values are listed in the accuracy and precision tables in Section 7.



**Table 4.2.2-1**  
**Statistical Calculations**

Statistic	Symbol	Formula	Definition	Uses
Mean	$\bar{X}$	$\frac{\left( \sum_{i=1}^n x_i \right)}{n}$	Measure of central tendency.	Used to determine average value of measurements.
Standard Deviation	S	$\left( \frac{\sum (x_i - \bar{X})^2}{(n-1)} \right)^{1/2}$	Measure of relative scatter of the data.	Used in calculating variation of measurements.
Relative Standard Deviation	RSD	$(S / \bar{X}) \times 100$	RSD adjusts for magnitude of observations.	Used to assess precision for replicate results.
Percent Difference	%D	$\frac{x_1 - x_2}{x_1} \times 100$	Measure of the difference of 2 observations.	Used to assess accuracy.
Relative Percent Difference	RPD	$\left( \frac{(x_1 - x_2)}{(x_1 + x_2) / 2} \right) \times 100$	Measure of variability that adjusts for the magnitude of observations.	Used to assess total and analytical precision of duplicate measurements.
Percent Recovery	%R	$\left( \frac{X_{meas}}{X_{true}} \right) \times 100$	Recovery of spiked compound in clean matrix.	Used to assess accuracy.
Percent Recovery	%R	$\left( \frac{\text{value of spiked sample} - \text{value of unspiked sample}}{\text{Value of added spike}} \right) \times 100$	Recovery of spiked compound in sample matrix.	Used to assess matrix effects and total precision.
Correlation Coefficient	r	see SW8000B Section 7.5.3		Evaluation of "goodness of fit" of a regression line.
Coefficient of Determination	COD	see SW8000B Section 7.5.3		Evaluation of "goodness of fit" of a polynomial equation.

x = Observation (concentration)

n = Number of observations



#### **4.2.2 Accuracy**

Accuracy is of the degree of agreement between an observed value and a “true” value (correctness) and includes a combination of the random error (precision) and systematic error (bias) components that result from the sampling and analytical procedures. It therefore reflects the total error associated with a measurement. A measurement is considered accurate when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit (CL). For volatile and semivolatile organic compounds, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed. Analysis of proficiency testing (PT) samples may also be used to provide additional information for assessing the accuracy of the analytical data being produced.

Both accuracy and precision are calculated for each AFCEE analytical batch, and the associated sample results are interpreted by considering these specific measurements. The formula for calculation of accuracy is included in Table 4.2.1-1 as percent recovery (%R) from pure and sample matrices. Accuracy requirements are listed for each method in Section 7.

#### **4.2.3 Representativeness**

Representativeness is a qualitative term, which refers to the degree in which data accurately and precisely depicts the characteristics of a population, whether referring to the distribution of contaminant within a sample, a sample within a matrix, or the distribution of a contaminant at a site. Representativeness is determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, and sampling locations. Objectives for representativeness are defined for each sampling and analysis task and are a function of the investigative objectives. Assessment of representativeness shall be achieved through use of the standard field, sampling, and analytical procedures. Decisions regarding sample/well/boring locations and numbers, and the statistical sampling design shall be documented in Section 3.3 of the project *FSP*.

#### **4.2.4 Completeness**

Completeness is a measure of the amount of valid data obtained compared with the amount that was expected to be obtained under correct, normal conditions. It is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples (e.g., by site) as set out in the PQOs. Valid data is data which is usable in the context of the project goals. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set.

For completeness requirements, valid results are all results not qualified with an R-flag after a usability assessment has been performed. Completeness should not be determined only on the basis of laboratory data qualifiers. (See Section 8 for an explanation of flagging criteria.) The goal for completeness, which should be based on specific project goals, is typically 95 percent for aqueous



samples and 90 percent for soil samples. The prime contractor must evaluate completeness with respect to project goals to determine its impact on the decision-making process.

The formula for calculation of completeness is presented below:

$$\% \text{ completeness} = \frac{\text{number of valid (i.e., non-R flagged) results}}{\text{number of possible results}}$$

#### **4.2.5 Comparability**

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another data set. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions encountered are considered in determining comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms shall support the assessment of comparability. Analysis of PT samples and reports from audits shall also be used to provide additional information for assessing the comparability of analytical data produced among subcontracting laboratories. Historical comparability shall be achieved through consistent use of methods and documentation procedures throughout the project. Assessment of comparability is primarily subjective and results should be interpreted by experienced environmental professionals with a clear knowledge of the PQOs and project decisions. Assessment should include a discussion of the level of uncertainty associated with the comparability of the specific data set and the potential consequences of using non-comparable data.

#### **4.2.6 Sensitivity**

Sensitivity is the ability of an analytical method or instrument to discriminate between measurement responses representing different concentrations. This capability is established during the planning phase to meet project-specific objectives. It is important to be able to detect the target analytes at the levels of interest. Sensitivity requirements include the establishment of various limits, which are described in Section 4.3, such as calibration requirements, instrument detection limits (IDLs), method detection limits (MDLs), and project-specific reporting limits (RLs). Both the IDLs and MDLs are normally based on an interference-free matrix (i.e. reagent water or purified solid), which do not take into account matrix effects and may not be achievable for environmental samples.

### **4.3 METHOD DETECTION LIMITS, REPORTING LIMITS, AND INSTRUMENT CALIBRATION REQUIREMENTS**

The MDLs, RLs, and instrument calibration procedures shall be provided in this project *QAPP* according to guidelines set forth below.



### 4.3.1 Method Detection Limits

Laboratories participating in this work effort shall demonstrate the MDLs for each instrument, including confirmatory columns, method of analysis, analyte, and matrix (i.e., water and soil) using the following instructions:

1. Estimate the MDL using one of the following:
  - a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5.
  - b) The concentration equivalent of 3 times the standard deviation of replicate measurement of the analyte in reagent water.
  - c) The region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).
2. Prepare (i.e., extract, digest, etc.) and analyze seven samples of a matrix spike (MS) (ASTM Type II water for aqueous methods, Ottawa sand for soil methods, glass beads of 1 mm diameter or smaller for metals) containing the analyte of interest at a concentration three to five times the estimated MDL.
3. Determine the variance ( $S^2$ ) for each analyte as follows:

$$S^2 = \frac{1}{n-1} \left[ \sum_{i=1}^n (x_i - \bar{x})^2 \right]$$

where  $x_i$  = the  $i$ th measurement of the variable  $x$  and  $\bar{x}$  = the average value of  $x$

$$\bar{X} = \frac{1}{n} \sum_{i=1}^n x_i$$

4. Determine the standard deviation ( $s$ ) for each analyte as follows:

$$s = (S^2)^{1/2}$$

5. Determine the MDL for each analyte as follows:

$$\text{MDL} = 3.14(s)$$

(note: 3.14 is the one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using 7 samples)

6. If the spike level used in step 2 is more than 10 times the calculated MDL, repeat the process using a smaller spiking level.

Where multiple instruments are used, the MDL used for reporting purposes shall represent the least sensitive instrument.



### **4.3.2 Method Detection Limit Verification**

An MDL verification check shall be performed on each instrument immediately following an MDL study and can be performed quarterly in place of the annual (every 12 months) MDL study. However, this may not substitute for the initial MDL determination. The MDL check sample shall be spiked at approximately two times the current reported MDL and taken through all preparatory and analytical steps. The MDL is verified if the laboratory can reliably detect and identify all analytes in the check sample by the method-specific criteria. If the method has no confirmation criteria, the check sample must produce a signal that is at least three times the instrument's noise level. If the MDL is not verified, spike at successively higher concentrations until the verification criteria are met, and use the first successful concentration as the reported MDL.

### **4.3.3 Reporting Limits**

The laboratories participating in this work effort shall compare the results of the MDL demonstrations to the RLs for each method that is listed in Section 7.0. The MDL may not be more than one-half the corresponding RL. The laboratories shall also verify RLs by including a standard at or below the RL as the lowest point on the calibration curve. All results shall be reported at or above the MDL values; however, for those results falling between the MDL and the RL, an "F" flag shall be applied to the results indicating the variability associated with the result (see Section 8.0). Results shall not be reported below the MDL.

### **4.3.4 Instrument Calibration**

Analytical instruments shall be calibrated in accordance with the analytical methods. All analytes reported shall be present in the initial and continuing calibrations, and these calibrations shall meet the acceptance criteria specified in Section 7.0. All results reported shall be within the calibration range. Results outside the calibration range are unsuitable for quantitative work and will only give an estimate of the true concentration. or SW6010 and SW6020, results shall be within the working range determined by linear range studies. Records of standard preparation and instrument calibration shall be maintained. Records shall unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards shall be traceable to standard materials.

Instrument calibration shall be checked using all of the analytes listed in the QC acceptance criteria table in Section 7.0 for the method. This applies equally to multi-response analytes (except as noted in Section 7.0). All calibration criteria shall satisfy SW-846 requirements at a minimum. The initial calibration shall be checked at the frequency specified in the method using materials prepared independently of the calibration standards. Multipoint calibrations shall contain the minimum number of calibration points specified in the method with all points used for the calibration being contiguous. If more than the minimum number of standards is analyzed for the initial calibration, all of the standards analyzed shall be included in the initial calibration. The only exception to this rule is a standard that has been statistically determined as being an outlier can be dropped from the calibration, providing the requirement for the minimum number of standards is met. Acceptance criteria for the calibration check are presented in Section 7.0. Analyte concentrations are determined with either calibration curves or response factors (RFs). For gas chromatography (GC) and GC/mass spectrometry methods, when using RFs to determine analyte concentrations, the





average RF from the initial five-point calibration shall be used. The continuing calibration shall not be used to update the RFs from the initial five-point calibration. The continuing calibration verification cannot be used as the LCS, except volatile organic compound (VOC) analysis. In addition, the concentration used for the calibration verification sample shall be at or below the middle of the calibration curve. Finally, the lowest standard used must be at or below the RL for each analyte in the method.

#### **4.4 ELEMENTS OF QUALITY CONTROL**

QC elements relevant to screening data are presented in Section 6.0. This section presents QC requirements relevant to analysis of environmental samples that shall be followed during the analytical activities for fixed-base, mobile, and field laboratories producing definitive data. The purpose of this QC program is to produce data of known quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials.

Laboratory QC samples (e.g., blanks and laboratory control samples) shall be included in the preparation batch with the field samples. An AFCEE analytical batch is a number of samples (not to exceed 20 environmental samples plus the associated laboratory QC samples) that are similar in composition (matrix) and that are extracted or digested at the same time and with the same lot of reagents. Matrix spikes and matrix spike duplicates (MSDs) count as environmental samples. The term AFCEE analytical batch also extends to cover samples that do not need separate extraction or digestion (e.g., volatile analyses by purge and trap). This AFCEE analytical batch is a number of samples (20 environmental samples plus the associated laboratory QC samples) that are similar in composition (matrix) and analyzed sequentially. AFCEE allows 20 field samples plus MS/MSD pair per batch. The identity of each AFCEE analytical batch shall be unambiguously reported with the analyses so that a reviewer can identify the QC samples and the associated environmental samples. The references to the analytical batch in the following sections and tables in this *QAPP* refer to the AFCEE analytical batch.

The type of QC samples and the frequency of use of these samples are discussed below and in the method-specific subsections of Section 7.0.

##### **4.4.1 Laboratory Control Sample**

The LCS is analyte-free water for aqueous analyses or a choice of Ottawa sand, sodium sulfate, or glass beads 1 mm or smaller in diameter for soil spiked with all analytes listed in the QC acceptance criteria table in Section 7.0 for the method. Each analyte in the LCS shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. (The midpoint is defined as the median point in the curve, not the middle of the range.) The LCS shall be carried through the complete sample preparation and analysis procedure.

The LCS is used to evaluate each AFCEE analytical batch and to determine if the method is in control. Except for VOCs, the LCS cannot be used as the continuing calibration verification.





One LCS shall be included in every AFCEE analytical batch. If more than one LCS is analyzed in an AFCEE analytical batch, results from all LCSs analyzed shall be reported. A QC failure of an analyte in any of the LCSs shall require appropriate corrective action including qualification of the failed analyte in all of the samples as required.

The performance of the LCS is evaluated against the QC acceptance limits given in the tables in Section 7.0. Whenever an analyte in an LCS is outside the acceptance limit, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, all samples in the AFCEE analytical batch shall be reanalyzed for the out-of-control analyte(s). When an analyte in an LCS exceeds the upper or lower control limit (UCL or LCL) and no corrective action is performed or the corrective action was ineffective, the appropriate validation flag, as described in Sections 7.0 and 8.0, shall be applied to all affected results.

#### **4.4.1.1 Marginal Exceedance**

A number of sporadic marginal exceedances of the LCS CLs are allowed. The number of exceedances is based on the total number of analytes spiked into the LCS and may not exceed 5 percent of the total number of analytes in the LCS. The table below presents the allowable number of marginal exceedances for a given number of analytes in the LCS.

<b>Number of Analytes in LCS</b>	<b>Allowable Number of Marginal Exceedances of LCS CLs</b>
>90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

A *marginal exceedance* is defined as beyond the LCS CLs but within the marginal exceedance limits, which are set at 4 standard deviations around the mean. This outside boundary prevents a grossly out-of-control LCS from passing.

Marginal exceedances must be sporadic (i.e., random). If the same analyte exceeds the LCS CLs repeatedly (e.g., 2 out of 3 consecutive LCS), that is an indication that the problem is systematic, not random. The source of error should be located and appropriate corrective actions taken. The allowance for marginal exceedance is a new policy being introduced Department of Defense (DoD)-wide.

#### **4.4.1.2 Laboratory Control Sample Failure**

Each LCS must be evaluated against the LCS CLs and marginal exceedance limits before being accepted. The recoveries for the analytes spiked into the LCS should first be compared with the LCS CLs. If a recovery is less than the LCL or greater than the UCL, that is an exceedance. The



laboratory should note which analytes exceeded the CLs and make a comparison to the list of project-specific analytes of concern. If a project-specific analyte of concern exceeds its LCS control limit, the LCS has failed. Next, the laboratory should add up the total number of exceedances for the LCS. Based on the number of analytes spiked into the LCS, the total number of exceedances is compared with the allowable number in the table. If an LCS has more than the allowable number of marginal exceedances, the LCS has failed. Finally, the recoveries for those analytes that exceeded the LCS CLs should be compared to the marginal exceedance limits. If a single analyte exceeds its marginal exceedance limit, the LCS has failed. (This only applies to methods with greater than 10 analytes.)

Note: The target analytes from Section 7.0 should not be considered project-specific analytes of concern unless the client separately specifies the analytes. A requirement to analyze all compounds on the target analyte list does not define a project-specific analyte.

In summary, failure of the LCS can occur several ways:

- Exceedance of an LCS control limit by any project-specific analyte of concern.
- Marginal exceedance of the LCS CLs by more than the allowable number of analytes.
- Exceedance of the marginal exceedance limits by one or more analytes.

Once an LCS has failed, corrective action is required.

#### **4.4.1.3 Corrective Action**

If a sample fails based on any criteria in Section 4.4.1.2, correction is required. The corrective action requirement applies to all analytes that exceeded the LCS CLs, even if one specific analyte's exceedance was not the trigger of LCS failure. All exceedances of the LCS CLs, marginal or otherwise, are subject to corrective action. If an LCS fails, an attempt must be made to determine the source of error and find a solution. All findings and corrective action should be documented. After the system problems have been resolved and system control has been reestablished, all samples in the AFCEE analytical batch shall be reprepmed and reanalyzed for the out-of-control analyte(s) or the batch rerun with a new LCS. The corrective action applied shall be based on professional judgment in the review of other QC measures (i.e., surrogates). If an analyte falls outside the LCS CLs a second time or if there is not sufficient sample material available to be reanalyzed, then all the results in the AFCEE analytical batch for that analyte must be flagged. The recoveries of those analytes subject to corrective action must be documented in the cast narrative, whether flagging is needed or not.

#### **4.4.2 Matrix Spike/Matrix Spike Duplicate**

A MS and MSD is an aliquot of sample spiked with known concentrations of all analytes listed in the QC acceptance criteria table in Section 7.0 for the method. The spiking occurs prior to sample preparation and analysis. Each analyte in the MS and MSD shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. Only AFCEE samples shall be used for spiking. The MS/MSD shall be designated on the chain of custody.



The MS/MSD is used to document the bias of a method due to sample matrix. Thus, for soil samples, laboratories may use the same container for the parent sample, the MS sample, and the MSD sample (except for volatile organics analyses [VOAs]), if there is enough sample. AECOM will select the samples for MS/MSDs. The sample replicates will be generated in the field, to be used by the laboratory to prepare the appropriate MS/MSDs. They are used to document potential matrix effects associated with a site. The MS/MSD results and flags must be associated or related to samples that are collected from the same site from which the MS/MSD set were collected. AFCEE does not use MSs and MSDs to control the analytical process.

A site-specific MS/MSD should be specified for each media, e.g., any different soil, water, soil gas, or sediment for each site during each sampling event, which should not exceed 5 working days in 1 week. A minimum of one MS and one MSD shall be designated by the field manager for each site and analyzed with every batch of AFCEE samples in a sample delivery group (SDG) of up to 20 field samples (i.e., collect up to 20 field samples followed by 2 additional samples designated as MS and MSD). More than one MS/MSD pair may be submitted as part of the sample group of environmental samples; however, project managers must coordinate with the laboratory providing analytical services for most cost effective sampling.

The performance of the MS and MSD is evaluated against the QC acceptance limits given in the tables in Section 7.0. If either the MS or the MSD is outside the QC acceptance limits, the analytes in all related samples shall be qualified according to the data flagging criteria in Sections 7.0 and 8.0. Please note: The laboratory will not report batch QC samples such as MS/MSD from another project.

#### **4.4.3 Surrogates**

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples.

Surrogates are used to evaluate accuracy, method performance, and extraction efficiency.

Surrogates shall be added to environmental samples, controls, and blanks, in accordance with the method requirements.

Whenever a surrogate recovery is outside the acceptance limit, corrective action must be performed. After the system problems have been resolved and system control has been reestablished, reprepare and reanalyze the sample. If corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Sections 7.0 and 8.0, shall be applied to the sample results.

#### **4.4.4 Internal Standards**

Internal standards (ISs) are measured amounts of certain compounds added after preparation or extraction of a sample. They are used in an IS calibration method to correct sample results affected by column injection losses, purging losses, or viscosity effects.

ISs shall be added to environmental samples, controls, and blanks, in accordance with the method requirements.



When the IS results are outside of the acceptance limits, corrective actions shall be performed. After the system problems have been resolved and system control has been reestablished, all samples analyzed while the system was malfunctioning shall be reanalyzed. If corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Sections 7.0 and 8.0, shall be applied to the sample results.

#### **4.4.5 Retention Time Windows**

Retention time windows are used in GC analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard on multiple days. The procedure and calculation method are given in SW-846 Method 8000C.

When the retention time is outside of the acceptance limits, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, reanalyze all samples analyzed since the last acceptable retention time check. If corrective actions are not performed, the appropriate validation flag, as described in Sections 7.0 and 8.0, shall be applied to the sample results.

#### **4.4.6 Interference Check Sample**

The interference check sample (ICS), used in inductively coupled plasma (ICP) analyses only, contains both interfering and analyte elements of known concentrations.

The ICS is used to verify background and interelement correction factors.

The ICS is run at the beginning and end of each run sequence for SW6010B and SW6020B.

When the interference check sample results are outside of the acceptance limits stated in the method, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, reanalyze the ICS. If the ICS result is acceptable, reanalyze all affected samples. If corrective action is not performed or the corrective action was ineffective, the appropriate validation flag, as described in Sections 7.0 and 8.0, shall be applied to all affected results.

#### **4.4.7 Method Blank**

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank shall be carried through the complete sample preparation and analytical procedure.

The method blank is used to document contamination resulting from the analytical process.

A method blank shall be included in every AFCEE analytical batch.

The presence of analytes in a method blank at concentrations equal to or greater than the RL indicates a need for corrective action. Corrective action shall be performed to eliminate the source of contamination prior to proceeding with analysis. After the source of contamination has been eliminated, all samples containing the analyte(s) found in the method blank above the RL shall be reprepared and reanalyzed. No analytical data shall be corrected for the presence of analytes in



blanks. When an analyte is detected in the method blank and in the associated samples and corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Sections 7.0 and 8.0, shall be applied to the sample results. If the target compounds detected in the method blank is greater than or equal to the MDL, then the lab will flag all associated samples with a “B” qualifier. The lab will perform a corrective action if the target compounds are greater than  $\frac{1}{2}$  of the RL or greater than the RLs.

#### **4.4.8 Ambient Blank**

The ambient blank consists of ASTM Type II reagent grade water poured into a VOC sample vial at the sampling site (in the same vicinity as the associated samples). It is handled like an environmental sample and transported to the laboratory for analysis. Ambient blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes.

Ambient blanks are used to assess the potential introduction of contaminants from ambient sources (e.g., active runways, engine test cells, gasoline motors in operation, etc.) to the samples during sample collection. Ambient blanks will be collected while the direct push is being used in the field.

An ambient blank shall be collected for each VOCs sampling event where the potential for introduction of contaminants from surrounding sources exist. Ambient blank samples shall be collected downwind of possible VOC sources. Flagging of sample results associated with contaminated ambient blanks is discussed in Section 8.

#### **4.4.9 Equipment Blank**

An equipment blank is a sample of ASTM Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis.

Equipment blanks are used to assess the effectiveness of equipment decontamination procedures.

The frequency of collection for equipment blanks is specified in Section 3.2 of the project-specific *WP*. Equipment blanks shall be collected immediately after the equipment has been decontaminated. The blank shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site.

When an analyte is detected in the equipment blank the appropriate validation flag, as described in Section 8, shall be applied to all sample results from samples collected with the affected equipment.

#### **4.4.10 Trip Blank**

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

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. Each cooler of samples sent to the laboratory for





analysis of VOCs shall contain one trip blank. For methanol preserved soil samples being analyzed for gasoline range organic (GRO) or VOC, a methanol blank shall be utilized.

When an analyte is detected in the trip blank the appropriate validation flag, as described in Section 8, shall be applied to all sample results from samples in the cooler with the affected trip blank.

#### **4.4.11 Field Duplicates**

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned a unique identification number in the field. Specific locations are designated for collection of field duplicate samples prior to the beginning of sample collection.

Duplicate sample results are used to assess precision of the sample collection process. Precision of soil samples to be analyzed for VOCs is assessed from collocated samples because the compositing process required to obtain uniform samples could result in loss of the compounds of interest.

The frequency of collection for field duplicates is specified in Section 3.2 of the project-specific *WP*.

#### **4.4.12 Field Replicates**

A field replicate sample, also called a split, is a single sample divided into two equal parts for analysis. The sample containers are assigned a unique identification number in the field. Specific locations are designated for collection of field replicate samples prior to the beginning of sample collection.

Replicate sample results are used to assess precision. The frequency of collection for field replicates is specified in Section 3.2 of the project-specific *WP*.

### **4.5 QUALITY CONTROL PROCEDURES**

#### **4.5.1 Holding Time Compliance**

All sample preparation and analysis shall be completed within the method-required holding times. The holding time for a sample begins at the time of sample collection. Some methods have more than one holding time requirement (e.g., methods SW8081A, SW8270C, etc.). The preparation holding time is calculated from the time of sample collection to the time of completion of the sample preparation process as described in the applicable method, prior to any necessary extract cleanup and/or volume reduction procedures. If no preparation (e.g., extraction) is required, the analysis holding time is calculated from the time of sample collection to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses. In methods requiring sample preparation prior to analysis, the analysis holding time is calculated from the time of preparation completion to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses.



If holding times are exceeded and the analyses are performed, the results shall be flagged according to the procedures as described in Section 8.

#### **4.5.2 Confirmation**

Quantitative confirmation of results at or above the RL for samples analyzed by GC or high performance liquid chromatography (HPLC) shall be required, unless otherwise specified for the method in Section 7, and shall be completed within the method-required holding times. For GC methods, a second column is used for confirmation. For HPLC methods, a second column or a different detector will be used. The result from the primary column/detector is the result that shall be reported. If holding times are exceeded and the analyses are performed, the results shall be flagged according to the procedures as described in Section 8.

#### **4.5.3 Control Charts**

Control charts are used to track the performance of laboratory control sample recoveries over time. All analytes spiked into the LCS should be tracked via control charts. These charts are useful in identifying trends and problems in an analytical method. Updating these charts on an annual basis and reviewing them on a quarterly basis for possible trends that could compromise data quality is recommended. These charts can also be used to benchmark a laboratory's performance against AFCEE requirements to determine possible areas to look for improvement.

#### **4.5.4 Standard Materials**

Standard materials, including second source materials, used in calibration and to prepare samples shall be traceable to National Institute Standards and Technology (NIST), USEPA, American Association of Laboratory Accreditation (A2LA) or other equivalent AFCEE approved source, if available. If an NIST, USEPA or A2LA standard material is not available, the standard material proposed for use shall be included in an addendum to the *SAP* and approved before use. The standard materials shall be current, and the following expiration policy shall be followed: The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory shall label standard and QC materials with expiration dates.

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





#### **4.5.5 Supplies and Consumables**

The laboratory shall inspect supplies and consumables prior to their use in analysis. The materials description in the methods of analysis shall be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents shall be monitored by analysis of LCSs. An inventory and storage system for these materials shall assure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions.



## 5.0 SAMPLING PROCEDURES

### 5.1 FIELD SAMPLING

The field sampling procedures for collecting samples and sampling methods shall be included in Section 4.3 of the project-specific *WP*.

#### 5.1.1 Sample Containers

Sample containers are purchased precleaned and treated according to USEPA specifications for the methods. Sampling containers that are reused are decontaminated between uses by the USEPA-recommended procedures (i.e., USEPA 540/R-93/051). Containers are stored in clean areas to prevent exposure to fuels, solvents, and other contaminants. Amber glass bottles are used routinely where glass containers are specified in the sampling protocol.

#### 5.1.2 Sample Volumes, Container Types, and Preservation Requirements

Sample volumes, container types, and preservation requirements for the analytical methods performed on AFCEE samples are listed in Table 5.1.2-1. The required sample volumes, container types, and preservation requirements for analytical methods proposed for project work not listed in use.

**Table 5.1.2-1  
 Requirements for Containers, Preservation  
 Techniques, Sample Volumes, and Holding Times**

Name	Analytical Methods	Containers	Preservation	Maximum Holding Time
Air VOCs	TO15	SUMMA <sup>®</sup> canister or equivalent	N/A	30 days
Volatile organics (VOCs)	SW8260B	<u>Aqueous</u> : 3 x 40ml glass VOA vial, polytetrafluoroethylene (PTFE) septum caps <u>Solid</u> : Encore or equivalent samplers	<u>Aqueous</u> : pH ≤ 2 with HCl <u>Solid</u> : Cool to 4°C.	<u>Aqueous</u> : 14 days <u>Solid</u> : 48 hours
1,4-dioxane (Semivolatile organics)	SW8270C SIM	<u>Aqueous</u> : 2 x 1-liter amber glass bottles with Teflon-lined cap(s)	<u>Aqueous</u> : If no residual chlorine present, cool to 4°C. If residual chlorine present, add 1 mL sodium thiosulfate per liter of water, cool to 4°C.	<u>Aqueous</u> : 7 days until extraction and 40 days after extraction



**Table 5.1.2-1**  
**Requirements for Containers, Preservation**  
**Techniques, Sample Volumes, and Holding Times (Continued)**

Name	Analytical Methods	Containers	Preservation	Maximum Holding Time
<b>IDW Soil/Sludge</b>				
TCLP Volatile fraction	SW1311	<b>Aqueous:</b> 500 mL glass bottle with PTFE-lined septum <b>Solid:</b> 125 mL glass jar with PTFE septum or Encore sampler	<b>Aqueous &amp; Solid:</b> Cool to 4°C.	<b>Aqueous &amp; Solid:</b> 14 days to TCLP extraction and extracts analyzed within 14 days after extraction
TCLP Extractable fraction	SW1311	<b>Aqueous:</b> 3 x 1-liter amber glass bottle with PTFE-lined lid <b>Solid:</b> 500 mL wide-mouth glass jar with PTFE lined lid	<b>Aqueous &amp; Solid:</b> Cool to 4°C.	<b>Aqueous &amp; Solid:</b> 14 days to TCLP extraction, 7 days to prep extraction and extracts analyzed within 40 days after prep extraction
TCLP Inorganic fraction (except Hg)	SW1311	<b>Aqueous:</b> N/A <b>Solid:</b> N/A	N/A	180 days to TCLP extraction, 180 days after TCLP extraction
TCLP Inorganic fraction (Hg)	SW1311	<b>Aqueous:</b> N/A <b>Solid:</b> N/A	N/A	28 days to TCLP extraction, 28 days after TCLP extraction
Ignitability	SW1010/SW1020	<b>Aqueous:</b> 250 mL glass or HDPE bottle <b>Solid:</b> N/A	<b>Aqueous:</b> Cool to 4°C	N/A
Corrosivity	SW9040/SW9045	<b>Aqueous:</b> 60 mL glass or HDPE bottle <b>Solid:</b> 125 mL wide-mouth glass bottle	<b>Aqueous:</b> Non required <b>Solid:</b> Cool to 4°C.	<b>Aqueous:</b> 24 hours <b>Solid:</b> As soon as possible
Reactivity-cyanide or sulfide	SW-846, Section 7.3	<b>Aqueous:</b> 1-liter glass or HDPE bottle <b>Solid:</b> 250 mL wide-mouth glass jar	<b>Aqueous:</b> Adjust pH to $\geq 12$ with 50% NaOH. If oxidizing agents present, add 5 mL NaAsO <sub>2</sub> per liter, or 0.6g ascorbic acid per liter. Cool to 4°C <b>Solid:</b> Cool to 4°C	14 days

PTFE = Polytetrafluoroethylene



## 5.2 SAMPLE HANDLING AND CUSTODY

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in field and laboratory records.

The contractor shall maintain chain-of-custody records for all field and field QC samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in their possession, (2) it is in their view, after being in their possession, (3) it was in their possession and they locked it up or, (4) it is in a designated secure area.

The following information concerning the sample shall be documented on the AFCEE chain of custody (CoC) form (as illustrated in Section 8):

- Unique sample identification for each container.
- Date and time of sample collection.
- Source of sample (including name, location, and sample type).
- Designation of MS/MSD.
- Preservative used.
- Analyses required.
- Name of collector(s).
- Pertinent field data (pH, temperature, etc.).
- Serial numbers of custody seals and transportation cases (if used).
- Custody transfer signatures, and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories.
- Bill of lading or transporter tracking number (if applicable).

All samples shall be uniquely identified, labeled, and documented in the field at the time of collection.

Samples collected in the field shall be transported to the laboratory or field testing site as expeditiously as possible. When a 4°C requirement for preserving the sample is indicated, the samples shall be packed in ice or chemical refrigerant to keep them cool during collection and transportation. During transit, it is not always possible to rigorously control the temperature of the samples. As a general rule, storage at low temperature is the best way to preserve most samples. A temperature blank (a VOC sampling vial filled with tap water) shall be included in every cooler and used to determine the internal temperature of the cooler upon receipt of the cooler at the laboratory. If the temperature of the samples upon receipt exceeds the temperature requirements, the exceedance shall be documented in laboratory records and discussed with AFCEE. The decision regarding the potentially affected samples shall also be documented.

Once the samples reach the laboratory, they shall be checked against information on the CoC form for anomalies. For the safety of the personnel involved, coolers containing AFCEE samples shall be



opened in a hood in case there has been any breakage of container of potentially contaminated sample material. The condition, temperature, and appropriate preservation of samples shall be checked and documented on the CoC form. Checking an aliquot of the sample using pH paper is an acceptable procedure except for VOCs where an additional sample is required to check preservation. The occurrence of any anomalies in the received samples and their resolution shall be documented in laboratory records. All sample information shall then be entered into a tracking system, and unique analytical sample identifiers shall be assigned. A copy of this information shall be reviewed by the laboratory for accuracy. Sample holding time tracking begins with the collection of samples and continues until the analysis is complete. Holding times for methods required routinely for AFCEE work are specified in Table 5.1.2-1. **Samples not preserved or analyzed in accordance with these requirements shall be resampled and analyzed, at no additional cost to AFCEE.** Subcontracted analyses shall be documented with the AFCEE CoC form. Procedures ensuring internal laboratory CoC shall also be implemented and documented by the laboratory. Specific instructions concerning the analysis specified for each sample shall be communicated to the analysts. Analytical batches shall be created, and laboratory QC samples shall be introduced into each batch.

While in the laboratory, samples shall be stored in limited-access, temperature-controlled areas. Refrigerators, coolers and freezers shall be monitored for temperature seven days a week. Acceptance criterion for the temperatures of the refrigerators and coolers is  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Acceptance criterion for the temperatures of the freezers shall be less than  $0^{\circ}\text{C}$ . All of the cold storage areas shall be monitored by thermometers that have been calibrated with a NIST-traceable thermometer. As indicated by the findings of the calibration, correction factors shall be applied to each thermometer. Records that include acceptance criteria shall be maintained. Samples for volatile organics determination shall be stored separately from other samples, standards, and sample extracts. Samples shall be stored after analysis until disposed of IAW applicable local, state, and federal regulations. Disposal records shall be maintained by the laboratory. Refrigerators storing AFCEE VOA samples shall contain a blank that shall be analyzed at a minimum of every two weeks.

Standard operating procedures (SOPs) describing sample control and custody shall be maintained by the laboratory.



## 6.0 SCREENING ANALYTICAL METHODS

The analytical screening methods contained in this section are shown in Table 6-1. This section includes brief descriptions of the methods and QC required for screening procedures commonly used to conduct work efforts. The methods and QC procedures were taken from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (USEPA SW-846, Third Edition, and its first, second and third update), *Methods for Chemical Analysis of Water and Waste* (USEPA 1979), *ASTM Annual Book of Standards* (1993), and from manufacturers' literature.

**Table 6-1  
Screening Analytical Methods**

Method	Parameter
SW846 (3550)	Moisture (as % solids)
SW1020A / SW1010/ SW1030	Ignitability
SW1110	Corrosivity
SW9040B	pH (water)
SW9045C	pH (soil)
SW9050A	Conductance
E170.1	Temperature
E180.1	Turbidity
E360.1	Dissolved oxygen
Organic vapor-analysis using an instrument equipped with photoionization detector (PID)	Soil gas screening-halogenated, aromatic, and petroleum hydrocarbons. Screening of drill cuttings, borings, monitoring wells, and temporary probes.
ASTM D1498	Oxidation-reduction potential

## 6.1 ANALYTICAL SCREENING METHOD DESCRIPTIONS

Section 6.1 contains subsections for each analytical procedure. Each subsection contains the following information:

- A brief method description
- The RL (if applicable)

### 6.1.1 Methods SW1010/SW1020A/SW1030 – Ignitability

Method SW1010 makes use of the Pensky-Martens tester to determine the flash point of liquid samples, including those that form surface films and/or contain non-filterable suspended solids.

Method SW1020A makes use of the Setaflash Closed Tester to determine the flash point of liquids that have flash points between 0 and 110 °C and viscosities lower than 150 stokes at 25 °C. If a



sample contains non-filterable suspended solids, use SW1010 (Pensky-Martens Ignitability) instead of Method SW1020.

Method SW1030 is used to determine the ignitability of solids and is suitable for the pastes, granular materials, solids that can be cut into strips, and powdery substances.

### **6.1.2 Method SW1110 – Corrosivity**

This test exposes steel to liquid waste to determine the corrosivity of the waste.

### **6.1.3 Methods SW9040B (Water)/SW9045C (Soil) – pH**

pH measurements shall be performed for aqueous samples using Method SW9040. pH measurements of soil or solid samples are performed using Method SW9045C. Measurements are determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode. pH measurements are important tools for predicting the extent of contamination as well as providing information regarding the potential ionization forms of contaminants in groundwater. This can be used to predict their respective fate and transport.

### **6.1.4 Method SW9050A – Conductance**

Standard conductivity meters are used. Temperature is also reported. Conductivity is an important parameter used in fate and transport modeling of contaminants.

### **6.1.5 United States Environmental Protection Agency Method 170.1 – Temperature**

Temperature measurements are made with a mercury-filled or dial type centigrade thermometer or a thermistor.

### **6.1.6 United States Environmental Protection Agency Method 180.1 – Turbidity**

This method is based on a comparison of the light scattered by the sample under defined conditions with the light intensity scattered by a standard reference suspension - the higher the intensity, the greater the turbidity. Turbidity measurements are made in a nephelometer and are reported in terms of nephelometric turbidity units (NTUs). The working range for the method is from 0 - 40 NTU. Higher levels of turbidity can be measured by diluting the sample with turbidity-free de-ionized water.

### **6.1.7 United States Environmental Protection Agency Method 360.1 – Dissolved Oxygen**

An instrumental probe, usually dependent upon an electrochemical reaction, is used for determination of dissolved oxygen in water. Under steady-state conditions, the current or potential can be correlated with dissolved oxygen concentrations. This measurement is used in fate and transport modeling as well as a factor in the determination of natural attenuation potential. It is also useful in predicting the chemical forms of the contaminants and their breakdown products.





### **6.1.8 American Society for Testing and Materials D1498 – Oxidation-Reduction Potential**

This method is designed to measure the oxidation-reduction potential (ORP) in water, which is defined as the electromotive force between a noble metal electrode and a reference electrode when immersed in a solution. This measurement is used in fate and transport modeling as well as a factor in the determination of natural attenuation potential.

### **6.1.9 SW-846 (Described in Method SW3550) – Percent Solids**

Percent solids is determined for solid samples undergoing analysis for inorganic and organic analytes. The sample is weighed, dried, and then reweighed. Percent solids is calculated as:

$$\frac{\text{Dried Weight}}{\text{Initial Weight}} \times 100 = \% \text{ solids}$$

The solid content is used to calculate results for soil samples on a dry weight basis using the calculation presented below:

$$\frac{\text{Result of analysis on a wet weight basis}}{\% \text{ solids} / 100} = \text{Result of analysis on a dry weight basis}$$

All MDLs for solids samples shall be reported on a dry-weight basis. Soil sample results shall be reported on a dry-weight basis.

### **6.1.10 Real-Time Portable Organic Vapor Analyzers**

Two types of portable analyzers shall be used to perform real-time nonspecific analyses of hydrocarbon vapors. The instruments include a PID (e.g., HNu® Systems [HNu®] trace gas analyzer) organic vapor monitor. One or more of these instruments may be used at a specific site, depending on the contaminant species of interest. When used together, the instruments provide complementary information because they are sensitive to different types of hydrocarbon vapors.

The portable analyzers shall be used as a screening tool to help determine the optimum locations for the collection of samples. Field data recorded on the CoC forms give the laboratory analysts an indication of the approximate concentration of contaminants and aid in calculating dilution factors before analysis. Additionally, the real-time instruments are used to aid in selecting the proper level of personal protective equipment and monitoring air emissions during sampling activities. The comparability of results obtained from the PID instrument can be considered only to be within the variability of this type of screening instrument. Comparability is greatest when the instruments are calibrated with the same standards and operated within similar concentration ranges.

The PID uses a photoionization detector to detect and measure total hydrocarbon vapors. The instrument has an operating range of 0 to 2,000 ppm. During operation, a gas sample is drawn into the probe and past an ultraviolet light source by an internal pumping system. Contaminants in the sample are ionized, producing an instrument response if their ionization potential is equal to or less



than the ionizing energy supplied by the lamp. The radiation produces a free electron for each molecule of ionized contaminant, which generates a current directly proportional to the number of ions produced. This current is measured and displayed on the meter. The PID measures the *total* value for all species present with ionization potentials less than or equal to that of the lamp.

## **6.2 CALIBRATION AND QUALITY CONTROL PROCEDURES FOR SCREENING METHODS**

All screening data shall be flagged with an “S” data qualifier to show the reported data are screening data (see Section 8). The other data qualifiers that shall be used with screening data are also shown in Table 6.2-1 and Section 8. Flagging criteria are applied (except for the “S” flag) when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Table 6.2-1 presents the calibration and QC procedures for each method. These requirements as well as the corrective actions and data flagging criteria are included. In this table, the first two columns designate the method number and the class of analytes that may be determined by the method. The third column lists the method-required calibration and QC elements. The fourth column designates the minimum frequency for performing each calibration and QC element. The fifth column designates the acceptance criteria for each calibration and QC element. The sixth column designates the corrective action in the event that a calibration or QC element does not meet the acceptance criteria. The last column designates the data flagging criteria that must be applied in the event that the method-required calibration and QC acceptance criteria are not met.



**Table 6.2-1  
Summary of Calibration and Quality Control Procedures  
for Screening Methods**

<b>Method</b>	<b>Applicable Parameter</b>	<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action<sup>a</sup></b>	<b>Data Flagging Criteria<sup>b</sup></b>
SW-846 <sup>c</sup>	Moisture	Field duplicate	1 per 20 samples	% solid RPD ≤ 15%	Correct problem, repeat measurement. If still out, flag data	J if RPD > 15% and Q if RPD > 30%
SW9040B	pH (water)	2-point calibration with pH buffers	Once per day	± 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration	Flagging criteria are not appropriate
		pH 7 buffer	At each sample location	± 0.1 pH units	Correct problem, recalibrate	Flagging criteria are not appropriate
		Field duplicate	10% of field samples	± 0.1 pH units	Correct problem, repeat measurement	J
SW9045C	pH (soil)	2-point calibration with pH buffers	Once per day	± 0.05 pH unit	Check with new buffers; if still out, repair meter; repeat calibration check	Flagging criteria are not appropriate
		pH 7 buffer	At each sample location	± 0.1 pH unit	Recalibrate	Flagging criteria are not appropriate
		Field duplicate	10% of field samples	± 0.1 pH unit	Correct problem, repeat measurement. If still out, repeat calibration and reanalyze samples	J
SW9050A	Conductance	Calibration with KCl standard	Once per day at beginning of testing	± 5%	If calibration is not achieved, check meter, standards, and probe; recalibrate	Flagging criteria are not appropriate
		Field duplicate	10% of field samples	± 5%	Correct problem, repeat measurement	J
E170.1	Temperature	Field duplicate	10% of field samples	± 1.0 C	Correct problem, repeat measurement	J



**Table 6.2-1**  
**Summary of Calibration and Quality Control Procedures**  
**for Screening Methods (Continued)**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Data Flagging Criteria <sup>b</sup>
E180.1	Turbidity	Calibration following manufacturer's instructions (minimum one blank and three standards)	As needed	In accordance with manufacturer's instructions	If calibration is not achieved, check meter; replace if necessary, recalibrate	Flagging criteria are not appropriate
		Calibration verification (mid-range)	Daily, before sample analysis	± 10% of expected value	Correct problem, repeat measurement, recalibrate	Flagging criteria are not appropriate
		Field duplicate	10% of field samples	RPD ≤ 20%	Correct problem, repeat measurement	J
None	Organic vapor concentrations (PID)	3 point calibration	Monthly	correlation coefficient ≥ 0.995	Recalibrate; check instrument and replace if necessary	Flagging criteria are not appropriate
		Calibration verification and check	Daily at beginning and end of day	Response ± 20% of expected value	Correct problem, recalibrate	Flagging criteria are not appropriate
ASTM D1498	Oxidation-reduction potential	Sensitivity verification	Daily	ORP should decrease when pH is increased	If ORP increases, correct the polarity of electrodes. If ORP still does not decrease, clean electrodes and Repeat procedure	Flagging criteria are not appropriate
		Calibration with one standard	Once per day	Two successive readings ± 10 millivolts	Correct problem, recalibrate	Flagging criteria are not appropriate
		Field duplicate	10% of field samples	± 10 millivolts	Correct problem, repeat measurement	J
SW1110	Corrosivity	Duplicate	10% of field samples	RPD ≤ 20%	Correct problem, repeat measurement	J
E360.1	Dissolved oxygen	Field duplicate	10% of field samples	RPD ≤ 20%	Correct problem, repeat measurement	J

- a. All corrective actions shall be documented, and the records shall be maintained by the prime contractor.
- b. All screening results are typically flagged with an "S" and also any other appropriate validation flags identified in the Data Flagging Criteria column of the table. For example "SJ", "SB", "SR". However, because the limited amount of screening data that will be generated will only be from health and safety monitoring (and potentially the measurement of *in-situ* groundwater parameters), the screening data will not be subjected to the typical data review, qualification, and validation process.



## 7.0 DEFINITIVE DATA ANALYTICAL METHODS AND PROCEDURES

Section 7.1 contains brief descriptions of preparation methods. Section 7.2 contains subsections for each analytical procedure. Each subsection contains the following information:

- A brief method description.
- A table of RLs.
- A table of QC acceptance criteria.
- A table of calibration procedures, QC procedures, and data validation guidelines.

This information was obtained from the *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (USEPA SW-846, Third Edition, and its first, second and third update); *Guidance for Contract Deliverables (GCD)*, Version 1.1, March 1998. Definitions of terms are given in Section 4.0, and data validation procedures are presented in Section 8.0.

### 7.1 PREPARATIVE METHODS

Typical SW-846 and other USEPA extraction and digestion procedures for liquid and solid matrices are presented in Table 7.1-1. These preparatory methods are also listed with the associated analytical procedures in Table 7.1-2. Method specific preparations are covered in the appropriate determinative methods.

**Table 7.1-1  
Sample Preparation Methods**

Method	Parameter
<b><i>Volatile Organics</i></b>	
SW5030B	Purge and Trap for Volatile Organic Compounds (aqueous samples)
SW5035A	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
<b><i>Extractable Organics</i></b>	
SW3510C	Separatory Funnel Liquid-Liquid Extraction (aqueous samples)
SW3520C	Continuous Liquid-Liquid Extraction (aqueous samples)
<b><i>Leaching Procedures</i></b>	
SW1311	Toxicity Characteristic Leaching Procedure (aqueous and solid samples)



**Table 7.1-2  
Analytical Methods**

Analytical Method	Parameter	Preparation Methods	
		Water/Aqueous	Soil/Solid
<b>GC/Mass Spectrometry</b>			
SW8260B	Volatile organics	5030B	5035A
TO-15	Volatile Organics in Air	N/A	N/A
<b>GC/Mass Spectrometry SIM</b>			
SW8270C SIM	1,4-dioxane	3510C, 3520C	N/A

### 7.1.1 Method SW5030B – Purge and Trap for Volatile Organic Compounds

Method SW5030B describes sample preparation and extraction for the analysis of VOCs. This method is applicable to aqueous samples.

An inert gas is then bubbled through the sample solution at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and back flushed with inert gas to desorb the components onto a GC column.

### 7.1.2 Method SW5035A – Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

Method SW5035A describes sample preparation and extraction for the analysis of VOCs in solid matrices. The method involves a heated purge of volatile components followed by analysis on a GC or GC/mass spectrometry. Analyzing the sample unpreserved within the prescribed 48-hour holding time is the preferred option.

### 7.1.3 Method SW3510C – Separatory Funnel Liquid-Liquid Extraction

Method SW3510C is designed to quantitatively extract nonvolatile and semivolatile organic compounds (SVOCs) from liquid samples using standard separatory funnel techniques. The sample and the extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method used to analyze the extract.

### 7.1.4 Method SW3520C – Continuous Liquid-Liquid Extraction

Method SW3520C is a procedure for isolating organic compounds from aqueous samples and is designed for extraction solvents with greater density than the sample.

### 7.1.5 Method SW1311 – Toxicity Characteristic Leaching Procedure

Method SW1311 is used to prepare samples for determination of the concentration of organic (semivolatile and volatile) and inorganic constituents that are leachable from waste or other





material. It is applicable for estimating the mobility of specific contaminants in wastes that are destined for disposal in municipal landfills.

QC is accomplished by preparing a toxicity characteristic leaching procedure (TCLP) blank at a rate of one blank for every 20 extractions conducted in the extraction vessel. Additional extract is prepared so one MS is performed for each waste type (samples of similar waste types shall be batched together). One MS must be analyzed in each AFCEE analytical batch. These QA measures are in accordance with USEPA Method SW1311, Section 8.0.

## 7.2 DETERMINATIVE METHODS

The analytical methods presented in this section are listed in Table 7.2-1. This section is organized by methodologies including GC/mass spectrometry and GC/mass spectrometry SIM.

A brief description and two tables for each method are included in the following subsections. The first table presents the RLs for the default analytes in the method. The RLs are presented for both soil and water matrices. The analytes included in these tables are not all inclusive lists. Specific lists of analytes for each method should be determined by regulatory requirements and site-specific information. The analytes in these tables should be used as defaults when no other target analyte list has been developed. The second table presents acceptance criteria for the accuracy of spiked analyte and surrogate recoveries. This table also presents acceptance criteria for the precision of matrix spike, field duplicate, and laboratory duplicate samples.

An additional table presents the calibration and QC procedures for each methodology (i.e., GC, GC/mass spectrometry, etc.). Corrective actions and data flagging criteria are also included in this table. The first two columns in this table designate the QC check and minimum frequency that the check is to be performed. The third column designates the acceptance criteria for each calibration and QC element, and the fourth column designates the corrective action in the event that a calibration or QC element does not meet the acceptance criteria. The last column designates the data flagging criteria that shall be applied by the laboratory in the event that the method-required calibration and QC acceptance criteria or acceptance criteria are not met. It should be clearly understood that these are laboratory data qualifiers. If a laboratory has more and they are consistent with these and properly defined, the laboratory may use them. When other flags are required contractually, they shall be used. Data usability should be carefully assessed by an individual experienced in data review who represents the data user or the user's agent.

It should be clearly understood that each analyst must demonstrate his or hers ability to generate acceptable accuracy and precision using at least four replicate analyses of a QC check sample. If the acceptance criteria are not met, then the problem must be located and fixed and demonstration successfully rerun prior to the analyst analyzing project samples. All data analyzed by an unqualified analyst (i.e., failing to meet QC criteria) shall be flagged R.

### 7.2.1 Gas Chromatography/Mass Spectrometry Methods

#### 7.2.1.1 Method SW8260B – Volatile Organics

Volatile (or purgeable) organics in water and soil samples are analyzed using method SW8260B. This method uses a capillary column GC/mass spectrometry technique. Volatile compounds are





introduced into the GC by purge and trap (SW5030B or SW5035) or other approved method (see Table 7.1-1). An inert gas is bubbled through the water samples (or soil-water slurry for soil samples) to transfer the purgeable organic compounds from the liquid to vapor phase. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a capillary GC column where they are separated and then detected with a mass spectrometer. The analytes detected and RLs (using a 25 mL purge) for this method are listed in Table 7.2.1.1-1. Soil samples with higher contaminant levels can be extracted using methanol before purging. However, the RLs arising from the use of this preparatory method will be higher than those listed in Table 7.2.1.1-1 and the accuracy and precision requirements listed in Table 7.2.1.1-2 will not be met as well. Project specific DQOs and analytical protocols will need to be established if this preparatory method is used.

Calibration – The mass spectrometer is tuned daily to give an acceptable spectrum for 4-Bromofluorobenzene (BFB). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

mass 50	15 percent to 40 percent of mass 95
mass 75	30 percent to 60 percent of mass 95
mass 95	base peak, 100 percent relative abundance
mass 96	5 percent to 9 percent of mass 95
mass 173	less than 2 percent of mass 174
mass 174	greater than 50 percent of mass 95
mass 175	5 percent to 9 percent of mass 174
mass 176	greater than 95 percent, but less than 101 percent of mass 174
mass 177	5 percent to 9 percent of mass 176

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample. Table 7.2.1.1-2 provides acceptance criteria for accuracy of spiked analytes and ISs, precision of duplicate/replicate analyses, and recommended IS associations. Also included in Table 7.2.1.1-2 are the marginal exceedances limits taken from the DoD QSM. Table 7.2.1.1-3 identifies the, QC checks, minimum frequencies, acceptance criteria, corrective actions, and flagging criteria.



**Table 7.2.1.1-1**  
**Reporting Limits for Method SW8260B**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs SW8260B	1,1,1,2-Tetrachloroethane	0.5	µg/L	0.003	mg/kg
	1,1,1-TCA	1.0	µg/L	0.005	mg/kg
	1,1,2,2-Tetrachloroethane	0.5	µg/L	0.003	mg/kg
	1,1,2-TCA	1.0	µg/L	0.005	mg/kg
	1,1-DCA	1.0	µg/L	0.005	mg/kg
	1,1-DCE	1.0	µg/L	0.006	mg/kg
	1,1-Dichloropropene	1.0	µg/L	0.005	mg/kg
	1,2,3-Trichlorobenzene	1.0	µg/L	0.005	mg/kg
	1,2,3-Trichloropropane	1.0	µg/L	0.005	mg/kg
	1,2,4-Trichlorobenzene	1.0	µg/L	0.005	mg/kg
	1,2,4-Trimethylbenzene	1.0	µg/L	0.006	mg/kg
	1,2-DCA	0.5	µg/L	0.003	mg/kg
	1,2-DCB	1.0	µg/L	0.005	mg/kg
	1,2-Dibromo-3-chloropropane	2.0	µg/L	0.01	mg/kg
	1,2-Dichloropropane	1.0	µg/L	0.005	mg/kg
	1,2-Dibromoethane (EDB)	1.0	µg/L	0.005	mg/kg
	1,3,5-Trimethylbenzene	1.0	µg/L	0.005	mg/kg
	1,3-DCB	1.0	µg/L	0.006	mg/kg
	1,3-Dichloropropane	0.4	µg/L	0.002	mg/kg
	1,4-DCB	0.5	µg/L	0.002	mg/kg
	1-Chlorohexane	1.0	µg/L	0.005	mg/kg
	2,2-Dichloropropane	1.0	µg/L	0.005	mg/kg
	2-Chlorotoluene	1.0	µg/L	0.005	mg/kg
	4-Chlorotoluene	1.0	µg/L	0.005	mg/kg
	Acetone	10	µg/L	0.05	mg/kg
	Benzene	0.4	µg/L	0.002	mg/kg
	Bromobenzene	1.0	µg/L	0.005	mg/kg
	Bromochloromethane	1.0	µg/L	0.005	mg/kg
	Bromodichloromethane	0.5	µg/L	0.002	mg/kg
	Bromoform	1.0	µg/L	0.006	mg/kg
	Bromomethane	3.0	µg/L	0.01	mg/kg
	Carbon tetrachloride	1.0	µg/L	0.005	mg/kg
	Chlorobenzene	0.5	µg/L	0.002	mg/kg
	Chloroethane	1.0	µg/L	0.005	mg/kg
	Chloroform	0.3	µg/L	0.002	mg/kg
	Chloromethane	1.0	µg/L	0.005	mg/kg
Cis-1,2-DCE	1.0	µg/L	0.005	mg/kg	
Cis-1,3-Dichloropropene	0.5	µg/L	0.003	mg/kg	
Dibromochloromethane	0.5	µg/L	0.003	mg/kg	
Dibromomethane	1.0	µg/L	0.005	mg/kg	
Dichlorodifluoromethane	1.0	µg/L	0.005	mg/kg	
Ethylbenzene	1.0	µg/L	0.005	mg/kg	
Hexachlorobutadiene	0.6	µg/L	0.003	mg/kg	



**Table 7.2.1.1-1**  
**Reporting Limits for Method SW8260B (Continued)**

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs SW8260B (concluded)	Isopropylbenzene	1.0	µg/L	0.005	mg/kg
	Methylene chloride	1.0	µg/L	0.005	mg/kg
	Methyl t-butyl ether (MTBE)	5.0	µg/L	0.02	mg/kg
	MEK (2-Butanone)	10	µg/L	0.02	mg/kg
	MIBK (methyl isobutyl ketone)	10	µg/L	0.02	mg/kg
	n-Butylbenzene	1.0	µg/L	0.005	mg/kg
	n-Propylbenzene	1.0	µg/L	0.005	mg/kg
	m,p-Xylene	2.0	µg/L	0.005	mg/kg
	Naphthalene	1.0	µg/L	0.005	mg/kg
	o-Xylene	1.0	µg/L	0.005	mg/kg
	p-Isopropyltoluene	1.0	µg/L	0.006	mg/kg
	Sec-Butylbenzene	1.0	µg/L	0.005	mg/kg
	Styrene	1.0	µg/L	0.005	mg/kg
	TCE	1.0	µg/L	0.005	mg/kg
	Tert-Butylbenzene	1.0	µg/L	0.005	mg/kg
	Tetrachloroethene	1.0	µg/L	0.005	mg/kg
	Toluene	1.0	µg/L	0.005	mg/kg
	Trans-1,2-DCE	1.0	µg/L	0.005	mg/kg
	Trans-1,3-Dichloropropene	1.0	µg/L	0.005	mg/kg
	Trichlorofluoromethane	1.0	µg/L	0.005	mg/kg
Vinyl chloride	1.0	µg/L	0.005	mg/kg	



**Table 7.2.1.1-2  
Quality Control Acceptance Criteria for Method SW8260B**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS
SW8260B	1,1,1,2-Tetrachloroethane	81-129	≤ 20	74-125	≤ 30	2
	1,1,1-TCA	67-132	≤ 20	68-130	≤ 30	1
	1,1,2,2-Tetrachloroethane	63-128	≤ 20	59-140	≤ 30	3
	1,1,2-TCA	75-125	≤ 20	62-127	≤ 30	1
	1,1-DCA	69-133	≤ 20	73-125	≤ 30	1
	1,1-DCE	68-130	≤ 20	65-136	≤ 30	1
	1,1-Dichloropropene	73-132	≤ 20	70-135	≤ 30	1
	1,2,3-Trichlorobenzene	67-137	≤ 20	62-133	≤ 30	3
	1,2,3-Trichloropropane	73-124	≤ 20	63-130	≤ 30	3
	1,2,4-Trichlorobenzene	66-134	≤ 20	65-131	≤ 30	3
	1,2,4-Trimethylbenzene	74-132	≤ 20	65-135	≤ 30	3
	1,2-DCA	69-132	≤ 20	72-137	≤ 30	1
	1,2-DCB	71-122	≤ 20	74-120	≤ 30	3
	1,2-Dibromo-3-chloropropane	50-132	≤ 20	49-135	≤ 30	3
	1,2-Dichloropropane	75-125	≤ 20	71-120	≤ 30	1
	1,2-EDB	80-121	≤ 20	70-124	≤ 30	2
	1,3,5-Trimethylbenzene	74-131	≤ 20	65-133	≤ 30	3
	1,3-DCB	75-124	≤ 20	72-124	≤ 30	3
	1,3-Dichloropropane	73-126	≤ 20	76-123	≤ 30	2
	1,4-DCB	74-123	≤ 20	72-125	≤ 30	3
	1-Chlorohexane	70-125	≤ 20	60-135	≤ 30	2
	2,2-Dichloropropane	69-137	≤ 20	67-134	≤ 30	1
	2-Chlorotoluene	73-126	≤ 20	69-128	≤ 30	3
	4-Chlorotoluene	74-128	≤ 20	73-126	≤ 30	3
	Acetone	40-135	≤ 20	40-141	≤ 30	1
	Benzene	81-122	≤ 20	73-126	≤ 30	1
	Bromobenzene	76-124	≤ 20	66-121	≤ 30	3
	Bromochloromethane	65-129	≤ 20	71-127	≤ 30	1
	Bromodichloromethane	76-121	≤ 20	72-128	≤ 30	1
	Bromoform	69-128	≤ 20	66-137	≤ 30	2
	Bromomethane	53-141	≤ 20	45-141	≤ 30	1
	Carbon Tetrachloride	66-138	≤ 20	67-133	≤ 30	1
	Chlorobenzene	81-122	≤ 20	75-123	≤ 30	2
	Chloroethane	58-133	≤ 20	41-141	≤ 30	1
	Chloroform	69-128	≤ 20	72-124	≤ 30	1
	Chloromethane	56-131	≤ 20	51-129	≤ 30	1
	Cis-1,2-DCE	72-126	≤ 20	67-125	≤ 30	1
	Cis-1,3-Dichloropropene	69-131	≤ 20	72-126	≤ 30	1
	Dibromochloromethane	66-133	≤ 20	66-130	≤ 30	2
	Dibromomethane	76-125	≤ 20	73-128	≤ 30	1
Dichlorodifluoromethane	53-153	≤ 20	34-136	≤ 30	1	



**Table 7.2.1.1-2  
Quality Control Acceptance Criteria for Method SW8260B (Continued)**

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	
SW8260B (Concluded)	Ethylbenzene	73-127	≤ 20	74-127	≤ 30	2	
	Hexachlorobutadiene	67-131	≤ 20	53-142	≤ 30	3	
	Isopropylbenzene	75-127	≤ 20	77-129	≤ 30	3	
	m,p-Xylene	76-128	≤ 20	79-126	≤ 30	2	
	Methylene chloride	63-137	≤ 20	63-137	≤ 30	1	
	Methyl t-butyl ether (MTBE)	65-123	≤ 20	50-135	≤ 30	1	
	MEK (2-Butanone)	49-136	≤ 20	40-135	≤ 30	1	
	MIBK (methyl isobutyl ketone)	58-134	≤ 20	47-147	≤ 30	3	
	n-Butylbenzene	69-137	≤ 20	65-138	≤ 30	3	
	n-Propylbenzene	72-129	≤ 20	63-135	≤ 30	3	
	Naphthalene	54-138	≤ 20	51-135	≤ 30	3	
	o-Xylene	80-121	≤ 20	77-125	≤ 30	2	
	p-Isopropyltoluene	73-130	≤ 20	75-133	≤ 30	3	
	Sec-Butylbenzene	72-127	≤ 20	63-132	≤ 30	3	
	Styrene	65-134	≤ 20	74-128	≤ 30	2	
	TCE	70-127	≤ 20	77-124	≤ 30	1	
	Tert-butylbenzene	70-129	≤ 20	65-132	≤ 30	3	
	Tetrachloroethene	66-128	≤ 20	67-139	≤ 30	2	
	Toluene	77-122	≤ 20	71-127	≤ 30	1	
	Trans-1,2-DCE	63-137	≤ 20	66-134	≤ 30	1	
	Trans-1,3-Dichloropropene	59-135	≤ 20	65-127	≤ 30	1	
	Trichlorofluoromethane	57-129	≤ 20	49-139	≤ 30	1	
	Vinyl Chloride	50-134	≤ 20	58-126	≤ 30	1	
	<i>Surrogates:</i>						
		Dibromofluoromethane	85-115		65-135		
		Toluene-D8	81-120		84-116		
		4-Bromofluorobenzene	76-119		84-118		
		1,2-DCA-D4	72-119		52-149		
	<i>Internal Standards:</i>						
		Fluorobenzene					1
	Chlorobenzene-D5					2	
	1,4-Dichlorobenzend-D					3	



**Table 7.2.1.1-3**  
**Summary of Calibration and Quality Control Procedures**  
**for Method SW8260**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8260B	VOCs	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq 0.30^c$ and %RSD for RFs for calibration check compounds (CCCs) $\leq 30\%$ and one option below	Correct problem then repeat initial calibration	Problem must be corrected. Samples may not be analyzed until is a valid initial calibration (ICAL).
				<i>option 1 linear</i> - mean RSD for all analytes $\leq 15\%$ with no individual analyte RSD $> 30\%$		Problem must be corrected. Samples may not be analyzed until is a valid ICAL.
				<i>option 2 linear</i> - linear least squares regression $r \geq 0.995$ for each analyte		Problem must be corrected. Samples may not be analyzed until is a valid ICAL.
				<i>option 3 non-linear</i> - COD $\geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)		Problem must be corrected. Samples may not be analyzed until is a valid ICAL.
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.





**Table 7.2.1.1-3  
 Summary of Calibration and Quality Control Procedures  
 for Method SW8260 (Continued)**

<b>Method</b>	<b>Applicable Parameter</b>	<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action<sup>a</sup></b>	<b>Flagging Criteria<sup>b</sup></b>
SW8260B	VOCs	Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within $\pm 0.06$ RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply Q-flag to all results for the specific analyte(s) in the sample which are outside the established window.
		Continuing Calibration verification	Daily, before sample analysis and after every 12 hours of analysis time	SPCCs average RF $\geq 0.30^c$ ; and CCCs $\leq 20\%$ difference (when using RFs) or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply Q-flag to all results for the specific analyte(s) $>20\%$ D for all samples associated with the calibration verification.
				All calibration analytes within $\pm 20\%$ of expected value		Apply Q-flag to all results for the specific analyte(s) $>20\%$ D for all samples associated with the calibration verification.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.1.1-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply Q to all results for all samples analyzed by the analyst



**Table 7.2.1.1-3**  
**Summary of Calibration and Quality Control Procedures**  
**for Method SW8260 (Continued)**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8260B	VOCs	ISs	Each sample	Retention time $\pm 30$ seconds from retention time of the IS in the ICAL mid-point std. Extracted Ion Current Profile (EICP) area within -50% to +100% of area from IS in ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; if system was malfunctioning, mandatory reanalysis of associated samples	Apply Q to all results for analytes associated with a failed IS unless a matrix effect can be verified, then apply M.
		Method blank	One per analytical batch	No analytes detected $\geq \frac{1}{2}$ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.1.1-2  See Section 4.4.1.1 for guidance on determining marginal exceedances.	Correct problem then reanalyze  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch	If corrective action fails, apply Q-flag to the specific analyte(s) which are not marginal exceedances in all samples in the associated preparatory batch.



**Table 7.2.2.1-3**  
**Summary of Calibration and Quality Control Procedures for**  
**Method SW8260 (Continued)**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8260B	VOCs	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.1.1-2	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3) MS/MSD RPD > CL
SW8260B	VOCs	Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (Section 7.2.1)	Retune instrument and verify	Not appropriate
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.1.1-2	Correct problem then reextract and analyze sample	<p>For the samples;</p> <p>if the %R &gt; UCL for a surrogate, apply J to all positive results</p> <p>if the %R &lt; LCL for a surrogate, apply J to all positive results; apply UJ to all non-detect results</p> <p>If any surrogate recovery is &lt;10%, apply Q to all results</p>



**Table 7.2.2.1-3  
 Summary of Calibration and Quality Control Procedures  
 for Method SW8260 (Continued)**

<b>Method</b>	<b>Applicable Parameter</b>	<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action<sup>a</sup></b>	<b>Flagging Criteria<sup>b</sup></b>
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 7.2.1.1-1. All analytes must be detected and identified by method-specified criteria for the verification check to be valid.	Run MDL verification check at higher level and set higher MDL or reconduct MDL study	N/A
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.
- c. Except > 0.10 for bromoform, and > 0.10 for chloromethane and 1,1-DCA.



### **7.2.1.2 Methods TO-15 – Volatile Organics in Ambient Air**

VOCs in air samples are analyzed using Method TO-15. Whole air samples are collected in evacuated stainless steel canisters. In the laboratory, the VOCs are concentrated in a trap, revolatilized onto a capillary GC column where they are separated and then detected with a mass spectrometer. The mass spectrometry may be operated in either the SCAN or selected ion monitoring (SIM) mode. The GS/mass spectrometry/SCAN provides positive identification for a wide range of compounds, while the GC/mass spectrometry/SIM procedure has greater sensitivity for a more limited list of preselected VOCs. The project-specific requirements will determine which mode is appropriate.

Method TO-15 is strictly a GC/mass spectrometry method, and AFCEE requires that TO-14A also be performed with a mass spectrometer detector. In addition, Method TO-15 differs from Method TO-14A in its approach to water management. As a result, it addresses a more extensive analyte list than Method TO-14A. Table 7.2.1.2-1 lists the default analytes and RLs for these methods. The analytes in Table 7.2.1.2-1 represent an abbreviated analyte list common to both methods. If a more extensive analyte list is required, Method TO-15 is recommended. Table 7.2.1.2-1 also provides the accuracy and precision acceptance criteria for these methods. Also included are the marginal exceedances limits.

Table 7.2.1.2-2 identifies the QC checks, minimum frequencies, acceptance criteria, corrective actions, and flagging criteria for these analyses. Method TO-15 uses internal standards and has enhanced provisions for inherent QC. AFCEE requires that Method TO-14A analyses meet these same criteria.

Tuning - The mass spectrometer must be hardware tuned to give an acceptable spectrum for BFB. There are slight differences in the tuning criteria between Methods TO-14A and TO-15. Table 7.2.1.2-2 gives the tuning acceptance criteria for each method in terms of ion abundances for each specified mass. The more stringent requirements of TO-14A may be used for TO-15; however, that is not an AFCEE requirement.



**Table 7.2.1.2-1  
Reporting Limits and Quality Control Acceptance Criteria for Methods TO-15**

Analyte	RL (ppbv)	Accuracy (% R)	Precision RPD (%)	ME Limits
1,1,1-TCA	0.5	70 - 130	≤ 25	60 - 140
1,2-DCA	0.5	70 - 130	≤ 25	60 - 140
1,2-Dibromoethane	0.5	70 - 130	≤ 25	60 - 140
Benzene	0.5	70 - 130	≤ 25	60 - 140
Carbon tetrachloride	0.5	70 - 130	≤ 25	60 - 140
Chloroform	0.5	70 - 130	≤ 25	60 - 140
Styrene	0.5	70 - 130	≤ 25	60 - 140
TCE	0.5	70 - 130	≤ 25	60 - 140
m,p-Xylene	0.5	70 - 130	≤ 25	60 - 140
o-Xylene	0.5	70 - 130	≤ 25	60 - 140
Tetrachloroethylene	0.5	70 - 130	≤ 25	60 - 140
Toluene	0.5	70 - 130	≤ 25	60 - 140
Ethylbenzene	0.5	70 - 130	≤ 25	60 - 140
cis-1,2-DCE	0.5	70 - 130	≤ 25	60 - 140
cis-1,2-Dichloropropene	0.5	70 - 130	≤ 25	60 - 140
Methylene chloride	0.5	70 - 130	≤ 25	60 - 140
Chloromethane	0.5	70 - 130	≤ 25	60 - 140
Chloroethane	0.5	70 - 130	≤ 25	60 - 140
Chlorobenzene	0.5	70 - 130	≤ 25	60 - 140
Vinyl chloride	0.5	70 - 130	≤ 25	60 - 140
1,1,2,2-Tetrachloroethane	0.5	70 - 130	≤ 25	60 - 140
1,1-Dichloroethene	0.5	70 - 130	≤ 25	60 - 140
1,1,2-Trichloroethane	0.5	70 - 130	≤ 25	60 - 140
1,1-DCA	0.5	70 - 130	≤ 25	60 - 140
1,2-Dichloropropane	0.5	70 - 130	≤ 25	60 - 140
trans-1,2-DCE	0.5	70 - 130	≤ 25	60 - 140
Trans-1,2-Dichloropropene	0.5	70 - 130	≤ 25	60 - 140
<b>Surrogates:</b>				
1,2-Dichloroethane- <i>d4</i>	-	60 - 140	-	-
Toluene- <i>d8</i>	-	60 - 140	-	-
4-Bromofluorobenzene	-	60 - 140	-	-
<b>Internal Standards:</b>				
Bromochloromethane	-	-	-	-
Chlorobenzene- <i>d5</i>	-	-	-	-
1,4-Difluorobenzene	-	-	-	-





**Table 7.2.1.2-2**  
**Summary of Calibration and Quality Control Procedures**  
**for Methods TO-15**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Mass spectrometry tuning check (Use BFB)	Prior to initial calibration and calibration verification	Refer to criteria listed in Table 7.2.1.2-1.	Retune instrument and verify.	Not appropriate
Initial multipoint calibration for all analytes (minimum five standards) (ICAL)	Initial calibration prior to sample analysis	One of the options below:  <i>Option 1:</i> linear – RSD for each analyte $\leq 30\%$ .  <i>Option 2:</i> linear – least squares regression $r \geq 0.995$ for each analyte.  <i>Option 3:</i> non-linear – COD $\geq 0.99$ . (six points shall be used for second order, seven points shall be used for third order)	Correct problem then repeat initial calibration.	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
Second-source calibration verification	Once per ICAL	All analytes within $\pm 30\%$ of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
Calibration verification (CCV)	Daily, before sample analysis unless ICAL performed on same day and every 24 hours of analysis time	All analytes within $\pm 30\%$ of expected value	Correct problem, rerun CCV. If that fails, repeat initial calibration.	Apply Q-flag to all results for the specific analyte(s) $> 30\% D$ for all samples associated with the calibration verification.



**Table 7.2.1.2-2  
Summary of Calibration and Quality Control Procedures  
for Methods TO-15 (Continued)**

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action<sup>a</sup></b>	<b>Flagging Criteria<sup>b</sup></b>
Internal Standards (ISs)	Each sample	Retention time $\pm$ 0.33 minutes from retention time of the IS in the most recent valid calibration. (ICAL mid-point standard or CCV) EICP area within $\pm$ 40% of area of the IS in most recent valid calibration	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while the system was malfunctioning is mandatory.	Apply Q-flag to all results for analytes associated with a failed IS unless a matrix effect can be verified, then apply M-flag.
Method blank (humid zero air)	Immediately after ICAL or daily CCV	No analytes detected $\geq$ RL	Assess data. Correct problem. If necessary, -reprep and analyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all associated positive results for the specific analyte(s) as appropriate. See guidance Section 8.2.
LCS for all analytes	One LCS per analytical batch	Acceptance criteria: Table 7.2.1.2-1	Correct problem then reanalyze. If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE analytical batch.	If corrective action fails, apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.
MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	Acceptance criteria: Table 7.2.1.2-1	Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs	For specific analyte(s) in all samples collected from the same site matrix as the parent, apply M-flag
Sample duplicate	One sample duplicate per analytical batch	Acceptance criteria: Table 7.2.1.2-1	Correct problem and reanalyze sample and duplicate.	If corrective action fails, apply J-flag to the specific analyte(s) in the sample.



**Table 7.2.1.2-2  
Summary of Calibration and Quality Control Procedures  
for Methods TO-15 (Continued)**

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action<sup>a</sup></b>	<b>Flagging Criteria<sup>b</sup></b>
MDL study	At initial setup and subsequently once per 12-month period or quarterly MDL verification checks.	Detection limits established shall be $\leq$ ½ the RLs in Table 7.2.1.2-1. See 40 CFR, Part 136 Appendix B. Verification checks must produce a response at least 3X instrument noise level and must produce a response greater than the blanks associated with the MDL study.	Run MDL verification check at higher level and set higher MDL or reconduct MDL study.	N/A
Results reported between MDL and RL	None	None	None	Apply F-flag to all results between MDL and RL.

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



## 7.2.2 Gas Chromatography/Mass Spectrometry Selected Ion Monitoring Method

### 7.2.2.1 Method SW8270-SIM for 1,4-Dioxane

A GC/mass spectrometry method with SIM is used for detection of 1,4-dioxane in water. Samples are extracted and then concentrated by evaporation. Compounds of interested are separated by capillary column GC and quantitated using the method of internal standards.

Tuning – Prior to analysis, the mass spectrometer must be tuned to give an acceptable spectrum. Possible tuning compounds include perfluorotributylamine (PFTBA) and decafluorotriphenylphosphine (DFTPP). Tuning should meet manufacturer’s specifications or other documented source.

RLs are listed in Table 7.2.2.1-1. Table 7.2.2.1-2 provides acceptance criteria for Method 8270-SIM along with suggested surrogates and internal standards. Table 7.2.2.1-3 identifies the QC checks, minimum frequencies, acceptance criteria, corrective actions, and flagging criteria.

**Table 7.2.2.1-1  
Reporting Limits for Method 8270-SIM  
for 1,4-Dioxane**

Parameter	Water		Soils	
	RL	Unit	RL	Unit
1,4-dioxane	0.2	µg/L	--	--

**Table 7.2.2.1-2  
Quality Control Acceptance Criteria Method 8270-SIM  
for 1,4-Dioxane**

Analyte	Accuracy (% R)	Precision RPD (%)	ME Limits
1,4-dioxane	70 - 130	≤ 40	60 - 140



**Table 7.2.2.2-3  
Summary of Calibration and Quality Control Procedures  
for Method SW8270-SIM for 1,4-Dioxane**

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action<sup>a</sup></b>	<b>Flagging Criteria<sup>b</sup></b>
Mass spectrometry tuning check DFTPP (SW 8270C)	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description.	Retune instrument and verify.	Not appropriate
GC Performance Check (8270C only)	Daily prior to analysis of sample or calibration standards	No visible peak tailing for benzidine or pentachlorophenol (As a default, tailing factors should be less than 3.0 and 5.0, respectively.)	Correct problem, then repeat performance check.	Not appropriate
Initial multipoint calibration for all analytes (minimum five standards) (ICAL)	Initial calibration prior to sample analysis	<p><b>SPCCs:</b> Average RF <math>\geq 0.030^c</math> (SW8260B), <math>\geq 0.050</math> (SW8270C)</p> <p><b>CCCs:</b> % RSD for RFs <math>\leq 30\%</math></p> <p><b>and</b> one of the options below: <i>Option 1:</i> linear – RSD for each analyte <math>&lt; 15\%</math></p> <p><i>Option 2 linear</i> – linear least squares regression <math>r \geq 0.995</math> for each analyte</p> <p><i>Option 3 non-linear</i> – COD <math>\geq 0.99</math> (6 points shall be used for second order, 7 points shall be used for third order)</p>	Correct problem, then repeat initial calibration.	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.



**Table 7.2.2.2-3  
Summary of Calibration and Quality Control Procedures  
for Method SW8270-SIM for 1,4-Dioxane (Continued)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Second-source calibration verification	Once per ICAL	All analytes within $\pm 25\%$ of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard of the initial calibration curve.	N/A	N/A
Retention time window verified for each analyte	Each sample	Relative retention time (RRT) of the analyte within $\pm 0.06$ RRT units of ICAL	Correct problem, then reanalyze all samples analyzed since the last retention time check.	Apply Q-flag to all results for the specific analyte(s) in the sample which are outside the established window.
Continuing Calibration verification (CCV)	Daily, before sample analysis unless ICAL performed on same day and after every 12 hours of analysis time	<p><b>SPCCs:</b> average RF <math>\geq 0.050</math> (SW8270C);</p> <p><b>CCCs:</b> <math>\leq 20\% D</math></p> <p>All analytes within <math>\pm 20\% D</math> of expected value from ICAL <b>Note:</b> D = difference when using RFs or drift when using least squares, regression or non-linear calibration.</p>	Correct problem, then rerun CCV. If that fails, repeat initial calibration.	Apply Q-flag to all results for the specific analyte(s) $> 20\% D$ for all samples associated with the calibration verification.





**Table 7.2.2.2-3  
Summary of Calibration and Quality Control Procedures  
for Method SW8270-SIM for 1,4-Dioxane (Continued)**

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action<sup>a</sup></b>	<b>Flagging Criteria<sup>b</sup></b>
Internal Standards (ISs)	Each sample	Retention time $\pm$ 30 seconds from retention time of the IS in the ICAL mid-point std. EICP area within - 50% to +100% of area from IS in ICAL mid-point standard	Inspect mass spectrometer and GC for malfunctions and corrections made as appropriate. Reanalysis of samples analyzed while the system was malfunctioning is mandatory.	Apply Q-flag to all results for analytes associated with a failed IS unless a matrix effect can be verified, then apply M-flag.
Method blank	One per analytical batch	No analytes detected > 1/2 RL  For common lab contaminants no analytes detected > RL	Assess data. Correct problem. If necessary, -reprep and analyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all associated positive results for the specific analyte(s) as appropriate.
LCS for all analytes	One LCS per analytical batch	Acceptance criteria: Table 7.2.2.2-2	Correct problem, then reanalyze.  If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE batch.	If corrective action fails, apply Q-flag to the specific analyte(s) which are not marginal exceedances in all samples in the associated preparatory batch.
MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	Acceptance criteria: Table 7.2.2.2-2	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M-flag if: (1) %R for MS or MSD > UCL, (2) %R for MS or MSD < LCL, or (3) MS/MSD RPD > CL



**Table 7.2.2.2-3  
Summary of Calibration and Quality Control Procedures  
for Method SW8270-SIM for 1,4-Dioxane (Continued)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Surrogate spike	Every sample, spiked sample, standard, and method blank	Acceptance criteria: Table 7.2.2.2-2	Correct problem, then reprep and reanalyze the affected samples.  If matrix effect is verified, discuss in case narrative.	For the samples:  If the %R > UCL for any surrogate, apply J-flag to all positive results for associated analytes.  If the %R < LCL for any surrogate, apply J-flag to all positive results for associated analytes and UJ -flag to all associated non-detects.  If any surrogate recovery is <10%, apply Q-flag to all results for all associated analytes.
MDL study	At initial setup and subsequently once per 12-month period or quarterly MDL verification checks.	Detection limits established shall be ≤ ½ the RLs in Tables 7.2.2.2-1. See 40 CFR, Part 136 Appendix B. All analytes must be detected and identified by method-specified criteria for the for the verification check to be valid.	Run MDL verification check at higher level and set higher MDL or reconduct MDL study.	N/A
Results reported between MDL and RL	None	None	None	Apply F-flag to all results between MDL and RL.

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.



## **8.0 DATA MANAGEMENT AND EVALUATION**

The data reduction, verification, validation, assessment and reporting procedures described in this section will ensure (1) the data are reviewed and documented (2) transcription and data reduction errors are minimized, (3) complete documentation is maintained, and (4) the reported results are accurate, or qualified if necessary. Laboratory data reduction and verification procedures are required to ensure the data deliverable(s) meet the overall project objectives. Data reduction, whether performed by instrumentation or manually, shall follow methodologies specified in the laboratory SOPs or approved analytical methods. Project-specific variations of general procedures, statistical approach, or formulas must be identified and be detailed in this project *QAPP*. Any variances from established procedures must be requested and approved in advance. Automated procedures shall be verified as required by USEPA's *Guidance on Good Automated Laboratory Practices* (GALP, EPA 2185); all software shall be tested with a sample set of data to verify its correct operation via accurate capture, processing, manipulation, transfer, recording, and reporting of data. Data are reported in hardcopy data package(s) and as electronic data deliverable(s) (EDDs).

### **8.1 DATA REVIEW REQUIREMENTS FOR SCREENING DATA**

The prime contractor shall complete a 100 percent review of all screening data. The screening data methods are identified in Table 6-1 of Section 6. Applicable screening data shall be qualified with an S-flag and shall be further qualified if critical calibration and QC requirements are not within acceptable limits. The calibration, QC requirements, corrective action requirements, and flagging criteria required are shown in Table 6.2-1 in Section 6. The flagging criteria should be applied when acceptance criteria are not met and corrective action was either not successful or not performed. S- flags shall be maintained in the final data qualification. Also, any data that has been affected by multiple qualifiers shall retain these qualifiers in the final reviewed data package.

Screening data deliverables shall be prepared for all field analyses as described in Section 8.8. The screening data shall be reported on the AFCEE screening data report forms (AFCEE Forms S-1 through S-3), as illustrated in Section 8.8. The prime contractor's project manager shall be responsible for the review of the entire screening data report package, including the associated field records. The results of this review shall (1) determine if the project objectives have been met, and (2) calculate the completeness of the screening data for the project. These results shall be included in the screening data deliverable.

### **8.2 DATA REVIEW REQUIREMENTS FOR DEFINITIVE DATA**

Scientifically sound data of known and documented quality which meet PQOs are essential for use in the decision-making process. Data review is the process whereby data are examined and evaluated to varying levels of detail and specificity by a variety of personnel who have different responsibilities within the data management process. It includes verification, validation, and usability assessment. There must be persuasive records which document data review activities to afford effective assessment of the data for its quality and usability. The data can then move forward with associated qualifiers indicating the overall usability of the data.



Data verification is the first step in data review. As used here, data verification is confirmation that the specified requirements have been performed, i.e., it is a completeness check.

Data validation extends this and is confirmation that the requirements for a specific intended use are fulfilled. Data validation is the systematic process of evaluating the compliance of the data with the pre-defined requirements of the project, including method, procedural, or contractual requirements and the comparison of the data with criteria based on the quality objectives documented in the project *QAPP*. The purpose of data validation is to assess the performance associated with the analysis in order to determine the quality of the data. Data validation includes a determination, to the extent possible, of the reasons for any failure to meet performance requirements, and an evaluation of the impact of such failures on the usability of the data.

The data usability assessment is an evaluation based on the results of data validation and verification in the context of the overall project decisions or objectives. The assessment determines whether the project execution and resulting data meet project quality objectives. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

The requirements for data reporting and data review must be clearly defined in this project *QAPP* and be appropriate for the project-specific decision goals. In general, the standard data package required by AFCEE does not allow for complete independent reconstruction of the analytical data. Depending upon the project objectives and intended use of the data, a more rigorous data validation regimen may be required. This more extensive review requires a more comprehensive data deliverable package. This data package must contain sufficient information to completely reconstruct the chemical analyses and includes all batch QC results, instrument QC results (e.g., initial calibration verification, continuing calibration verification, and instrument performance checks), MDL studies, and raw data (e.g., run logs, sample preparation logs, standard preparation logs, and printed instrumental output such as chromatograms).

### 8.2.1 Laboratory Requirements

The chemistry data package must contain adequate information and be presented in a clear and concise manner. Minimum requirements include cover sheet which identifies the project; table of contents; case narrative which summarizes samples, analyses, and discusses any issues which may affect data usability; analytical results; laboratory reporting limits; sample management records; and internal laboratory QA/QC information. The AFCEE Forms (Section 8.8) may be used for this. Equivalent formats are acceptable, provided they include all essential information. The laboratory data package should be organized such that the analytical results are reported on a per AFCEE analytical batch (AAB) basis, unless otherwise specified. This will facilitate subsequent review, validation, and assessment. Based on the information in the data package, a reviewer should be able to determine the PARCCS and completeness of the data. The amount of information required to demonstrate attainment of PQOs depends upon the acceptable level of uncertainty for the intended data use and should be addressed in this project *QAPP*. Additional information may be required, depending on the detail of data review performed.

A schedule for data delivery should be established so that data packages (i.e. SDGs) are provided in a timely manner to the prime contractor for data review/validation, assessment and use. This



includes identifying the anticipated number or frequency of these data packages in light of project objectives, i.e., the amount of data produced or project duration.

### 8.2.1.1 Laboratory Data Reporting Requirements

An important part of the laboratory documentation is the case narrative. The case narrative contains essential information which affords an informed evaluation of data usability. The case narrative shall include but not be limited to:

- Table summarizing samples received, correlating field sample numbers, laboratory sample numbers, and laboratory tests completed.
- Discussion of sample appearance and integrity issues which may affect data usability (temperature, preservation, pH, sample containers, air bubbles, multiphases, etc.).
- Samples received but not analyzed and why.
- Discussion of holding time excursions for sample prep and analyses.
- Analysis of all out-of-control or discrepancies of calibrations, continuing calibrations or QC.
- sample results (surrogates, LCS, MS/MSD, post-digestion spikes, etc.), raw data/chromatograms and corrective actions taken.
- Identification of samples and analytes for which manual integration was necessary.
- Discussion of all qualified data and definition of qualifying flags.
- Discussion and recommendations of potential data usability of qualified data including detailed discussion of conditions associated with Q-flagged data.

Reporting details:

- DLs and sample results should be reported to one decimal place more than the corresponding RL, unless the appropriate number of significant figures for the measurement dictates otherwise.
- Soil samples shall have results reported on a dry weight basis. A wet weight aliquot of sample equivalent to the method specified dry weight aliquot of sample should be taken for analysis. Alternatively, the lab may choose to use a consistent wet weight aliquot that is expected to be large enough to compensate for the moisture in the sample (e.g., 50% more) and use this as a consistent weight.

**Note:** RLs are project specific requirements and are NOT adjusted for sample moisture. Detection limits may have to be adjusted for moisture; however, the laboratory should ensure that the minimum relationship between adjusted MDLs and corresponding RLs are maintained.

- If possible, samples should be analyzed undiluted and non-detects reported to the AFCEE specified RLs. RLs for minority constituents in highly contaminated samples may have to be adjusted for dilutions.



### 8.2.1.2 Manual Integrations

Manual integrations are an integral part of the chromatographic analysis process; they should be used judiciously to correct any incorrect integration by the automated instrumentation and not as a routine procedure for the purpose of meeting calibration or method QC acceptance criteria. Improper use of manual integrations (for example, peak shaving or peak enhancement) are considered improper, unethical, or illegal actions if performed solely to meet QC requirements. Manual integrations shall be done only as a corrective action measures. Examples of instances where manual integration would be warranted include, but are not limited to, co-eluting compounds resulting in poor peak resolution, a misidentified peak, an incorrect retention time, or a problematic baseline. When manual integrations are used, the following procedures are to be implemented for documenting the event and for consistency in performing the manual integration:

- There be a laboratory or section SOP for manual integrations. This SOP shall specify when automated integrations by the instrument are likely to be unreliable, what constitutes an unacceptable automated integration, and how the problems should be resolved by the analyst.
- This includes procedures for the analyst to follow in documenting any required manual integrations.
- When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations. The raw data records shall include the results of both the automated and manual integrations (i.e., “before” and “after” chromatograms of manually integrated peaks), notation of the cause and justification for performing the manual integrations, and date, and signature/initials of person performing the manual operations.
- All manual integrations must be reviewed and approved by the Section supervisor and/or the QA officer.

**Note:** Both the primary and secondary reviews (analyst’s and supervisory) may be performed electronically, provided all documentation and data integrity are maintained.

- All manual integrations must be identification in the case narrative.

This will ensure consistency when manual integrations are performed and facilitate review and acceptance of manually integrated data.

### 8.2.1.3 Tentatively Identified Compounds

Tentatively identified compounds (TICs) are compounds not associated with the calibration standards which are identified in methods with mass spectrometry detection. All peaks with a response greater than 10% of the nearest internal standard are potential TICs and should be examined. Qualitative identification of TICs is by computer searches of standard reference libraries and may be reported as a specific chemical or as a member of a chemical family. Concentrations are estimated assuming a response factor of 1 between the TIC and the nearest internal standard. The laboratory must have established procedures for reporting TICs.





#### **8.2.1.4 Laboratory Data Review Requirements**

All analytical data generated by the laboratory shall be verified prior to submittal to the AFCEE prime contractor. This internal data review process, which is multi-tiered, shall include all aspects of data generation, reduction, and QC assessment. Procedures for laboratory verification and validation of data should be summarized in this project *QAPP*. In each laboratory analytical section, the analyst performing the tests shall review 100 percent of the definitive data. After the analyst's review has been completed, 100 percent of the data shall be reviewed independently by a senior analyst or by the supervisor of the respective analytical section using the same criteria.

The following elements for review/verification at each level must include but not be restricted to:

- Sample receipt procedures and conditions.
- Sample preparation.
- Appropriate SOPs and analytical methodologies.
- Accuracy and completeness of analytical results.
- Correct interpretation of all raw data, including all manual integrations.
- Appropriate application of QC samples and compliance with established CLs.
- Verification of data transfers.
- Documentation completeness (e.g., all anomalies in the preparation and analysis have been identified, appropriate corrective actions taken, and have been documented in the case narrative(s), associated data have been appropriately qualified, anomaly forms are complete).

Accuracy and completeness of data deliverables (hard copy and electronic).

#### **8.2.1.5 Laboratory Data Evaluation**

The calibration, QC, corrective actions, and flagging requirements for definitive data are shown in the tables in Section 7.2. Data qualifiers shall be applied by the laboratory according to the requirements in the tables in Sections 6 and 7 as part of their validation activities. The allowable data qualifiers for definitive data are *Q*, *M*, *J*, *F*, *B*, *U*, *UJ* and *T*. The definitions of the data qualifiers are provided in Table 8.2.1.5-1. Flagging criteria apply when acceptance criteria are not met and corrective actions were not successful or not performed. The data qualifiers are reviewed by the supervisor of the respective analytical sections after the first and second level reviews of the laboratory data have been performed.

The one exception to these data flagging criteria is for TICs. The TIC numerical results will always be qualified with one and only one flag: the T-flag.

The laboratory QA section shall perform a 100 percent review of 10 percent of the completed data packages, and the laboratory project representative shall complete a final review on all the completed data packages.





The prime contractor subsequently evaluates the flags applied by the laboratory as part of their data validation and usability assessment activities. The flags may be accepted, modified, or rejected. For all data qualifiers which are changed, the prime contractor must provide clear justification for those modifications based on project-specific quality objectives. All Q-flagged data must be evaluated by the prime contractor and either accepted without qualification, accepted with qualification, or rejected.

**Table 8.2.1.5-1  
Data Qualifiers**

Qualifier	Description
J	The analyte was positively identified, the quantitation is an estimation.
U	The analyte was analyzed for, but not detected. The associated numerical value is at or below the MDL.
UJ	The analyte was not detected; however, the result is estimated due to discrepancies in meeting certain analyte-specific quality control criteria.
F	The analyte was positively identified but the associated numerical value is below the RL.
Q	One or more quality control criteria (for example, LCS recovery, surrogate spike recovery) failed. Data must be carefully assessed by the prime contractor (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data.
B	The analyte was found in an associated blank above one-half the RL, as well as in the sample.
M	A matrix effect was present.
S	To be applied to all field screening data.
T	Tentatively identified compound: The analyte is a tentatively identified compound (mass spectrometry methods only).

### 8.2.1.6 Method Blank Evaluation Guidance

The following criteria shall be used to evaluate the acceptability of the blank data, unless project quality objectives specify otherwise. For method blanks, the source of contamination shall be investigated and measures taken to correct, minimize, or eliminate the problem if the concentration exceeds one-half the RL. (Use the RL for common laboratory contaminants.) If one-half the RL is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria: 1) the method blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparation batch, or 2) there is evidence the blank contamination otherwise affects the sample results. Except when the sample analysis resulted in a non-detect, all samples associated with method blank contamination and meeting these criteria shall be reprocessed in a subsequent preparation batch. If no sample volume remains for reprocessing, the results shall be reported with a B-flag, along with any other appropriate data qualifier. If an analyte is found only in the method blank, but not in any batch samples, no flagging is necessary. Method blanks should also be examined to verify that any TICs present in the samples are not found in the blank. Method blank contamination must be addressed in the case narrative.



## 8.2.2 Prime Contractor Requirements

The ultimate goal of data review is to ensure that the decisions, which are made as a result of the environmental data collection effort, are supported by data of the type and quality suitable for their intended use. The prime contractor has overall responsibility for data quality and may be assisted in its review by external organizations. Parties performing data review should be clearly identified in this project *QAPP*.

### 8.2.2.1 Responsibility and Qualifications

The data validation/usability assessment processes involves exercise of professional judgment. Regardless of who performs these, the individual(s) should possess the disciplinary expertise, experience, and theoretical knowledge to perform the task. It is also imperative that these individuals possess a complete understanding of the intended use of the data and the relationship of the QC results to the usability of the data. For this reason, it is essential that they be involved during project planning in the systematic planning process, choice of preparation and analytical methods, and decisions made regarding data verification and data validation. When this is not feasible, such as when a third party is contracted for data validation, all project planning documents and procedures, as well as sample collection information must be made available to the individuals assigned to the task.

Although discussed sequentially below, certain steps in the data review process may be performed simultaneously.

### 8.2.2.2 Data Verification Guidelines

The data verification performed by the laboratory should be reviewed for completeness and accuracy. Data verification may be done electronically or manually, or by a combination of both, and shall include (but is not limited to):

- Sampling documentation (CoC form, etc.),
- Preservation summary and technical holding times,
- Presence of all analyses and analytes requested,
- Use of the required sample preparation and analysis procedures,
- Method detection and reporting limits evaluated against the project requirements,
- The correctness of the concentration units, and
- Case narrative.

### 8.2.2.3 Data Validation Guidelines

The data validation process builds on data verification. The laboratory case narrative, QC sample results, and calibrations shall be reviewed and data qualifiers removed or added in light of project knowledge for 100 percent of the data. Method-specific instrument calibration and QC parameters shall be reviewed for compliance with calibration and QC requirements specified in Section 7.0.



An in-depth review of the raw data to verify accuracy shall be performed on 10 percent of the data and include the following, but is not limited to:

- Instrument calibration and QC parameters (method-specific) (these shall be reviewed for compliance with the criteria specified in the applicable Summary of Calibration and QC Procedures tables, and flagged as necessary).
- Review of raw data such as instrument print outs, preparation logs, and run logs.
- Review of system performance.
- Random check of calculations, including, but not limited to, sample and QC results, initial calibration response factors and RSDs, calibration verification standard response factors, and percent differences or percent drifts from the expected values.
- Random verification of sample results to the raw data.
- Check for interference problems or system performance problems.
- Estimated results (F-qualifiers).
- Resolution by the laboratory of any identified problems, as necessary.

#### **8.2.2.3.1 Raw Data Review**

This may include, but is not limited to:

- Instrument Calibration and QC Parameters (Method-Specific). These shall be reviewed for compliance with the criteria specified in the applicable Summary of Calibration and QC Procedures tables, and flagged as necessary.
- Review of raw data and inspections of chromatograms.
- Review of System Performance.
- Review for proper integration (if applicable).
- Review of spectral matches, and/or retention times to verify analyte identification (where applicable).
- TIC data.
- Random check of calculations, including, but not limited to sample and QC results, initial calibration response factors and RSDs, calibration verification standard response factors, and percent differences or percent drifts from the expected values.
- Check for interference problems or system performance problems, such as chromatographic baseline anomalies and drifts, evidence of column degradation, etc.
- Estimated Results (F-qualifiers).
- Resolution by the laboratory of any identified problems, as necessary.

#### **8.2.2.3.2 Data Analysis and Interpretation**

This phase of the data validation process (assessment) relies heavily on the validator's professional judgment. It may include, but is not limited to:



- Evaluation of all Q-flagged data and final determination of its usability. All Q-flagged data must be accepted without qualification, accepted with qualification, or rejected. The Q-flag is not to be used in the final assessment (see Section 8.2.1.5).
- Evaluation of all B-flagged data and final determination of its usability (see Section 8.2.2.3.3).
- Evaluation of duplicate, replicate, and split sample analyses. Indications of poor precision should be investigated for cause and the impact on the overall usability of the data must be discussed (see Section 8.2.2.3.4).
- Evaluation of all M-flagged data. Only the matrix spike sample is qualified by the laboratory. The prime contractor shall apply any additional qualifying flag for a matrix effect to all samples collected from the same site as the parent sample or all samples showing the same lithologic characteristics as the MS/MSD (see Section 8.2.2.3.5).
- Evaluation of the impact of multiple data issues on the final analytical results (for example, variability of results obtained from different dilutions, or different methods; chromatographic issues; etc.).
- Evaluation of the deficiencies identified during data verification and assessment of their impact on the sample results.
- Incorporation of site-specific factors and assessment of their impact on the data.
- Assessment of data usability and assignment of final data qualifiers, as necessary.
- Discussion of completeness, representativeness, and comparability.

A data validation report will be prepared summarizing the findings and discussing their impact on the overall data usability. This may be incorporated into the final usability assessment.

#### **8.2.2.3.3 Blank Evaluation Guidelines**

The prime contractor is expected to evaluate laboratory B-qualified data such as method blanks, as well as other blanks (field blanks such as trip blanks, or equipment blanks, etc.) based on the concentration of the analyte in the samples in relation to the concentration in the blank, during the data validation process. The B-flag may be removed and not utilized if the analyte concentrations in the samples are much higher ( $\geq 5X$ ) than in the blank. ( $\geq 10X$  in case of common laboratory contaminants). Any blank contamination which may impact data usability must be discussed by the prime contractor in conjunction with project-specific goals.

#### **8.2.2.3.4 Duplicate/Replicate Evaluation Guidance**

As discussed in Section 4, QC measures for precision include field duplicates, laboratory duplicates, matrix spike duplicates, analytical replicates, and surrogates. These measures are evaluated by the laboratory and qualified according to the guidelines in Sections 7 and 8 with the exception of the field duplicates. Specifically, field duplicates (replicates) or split samples should be sent to the laboratory(ies) as blind samples and should be given unique sample identification numbers. These sample results can then be associated by the prime contractor and can be used to assess field



sampling precision, laboratory precision, and, potentially, the representativeness of the matrix sampled. The prime contractor must use experience and site specific knowledge to assess the value of the field duplicate samples as a measure of precision or representativeness. Flagging of results associated with field duplicates should be assigned such that the level of uncertainty required, as provided by the project-specific objectives, is taken into account. Poor overall precision may be the result of one or more of the following: field instrument variation, analytical measurement variation, poor sampling technique, sample transport problems, or spatial variation (heterogeneous sample matrices). To identify the cause of imprecision, the field sampling design rationale and sampling techniques should be evaluated by the prime contractor, and both field and analytical duplicate/replicate sample results should be reviewed. If poor precision is indicated in both the field and analytical duplicates/replicates, then the laboratory may be the source of error. If poor precision is limited to the field duplicate/replicate results, then the sampling technique, field instrument variation, sample transport, and/or spatial variability may be the source of error. If data validation reports indicate that analytical imprecision exists for a particular data set or SDG, then the impact of that imprecision on usability must be discussed in the report.

#### **8.2.2.3.5 Matrix Interface Evaluation Guidance**

In the case of matrix interference, the laboratory will follow the guidelines specified in appropriate Tables in Section 7. However, the prime contractor must apply M flags to additional samples from the same site and same matrix, as applicable.

#### **8.2.2.4 Flagging Conventions**

The allowable final data qualifiers for definitive data and the hierarchy of data qualifiers, listed in order of the most severe through the least severe, are *R*, *M*, *J*, *F*, *B*, *U*, and *UJ*. Their definitions are summarized in Table 8.2.1.5-1. The T-flag is used only for TICs.

Tables 8.2.2.4-1 and 8.2.2.4-2 present the general guidelines for applying these data qualifiers. The tables in Section 7 should be consulted for specific details.

### **8.3 QUALITY ASSURANCE REPORTS**

The laboratory QA staff shall issue QA reports to the laboratory management, laboratory supervisors, and task leaders. These reports shall describe the results of QC measurements, performance audits, and systems audits, and confirmation sample comparisons performed for each sampling and analysis task. Quality problems associated with performance of methods, completeness of data, comparability of data including field and confirmatory data, and data storage shall be documented with the corrective actions that have been taken to correct the deficiencies identified.

### **8.4 ENVIRONMENTAL RESOURCES PROGRAM INFORMATION MANAGEMENT SYSTEM ELECTRONIC DATA REPORTS**

The prime contractor shall provide an electronic deliverable report in the Electronic Resources Program Information Management System (ERPIMS) format as specified by the Statement of Work (SOW) for the project.



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**Table 8.2.2.4-1  
General Flagging Conventions**

<b>QC Requirement</b>	<b>Criteria</b>	<b>Flag</b>	<b>Flag Applied To</b>
Holding Time	Time exceeded for extraction or analysis	R	All analytes in the sample
LCS	% R > UCL % R < LCL	J for the positive results J for the positive results, R for the nondetects	The specific analyte(s) in all samples in the associated AAB
Method Blank	Analyte(s) detected $\geq$ 1/2 RL	B	The specific analyte(s) in all samples in the associated AAB with results above the RL
Equipment Blank	Analyte(s) detected $\geq$ 1/2 RL	B	The specific analyte(s) in all samples with the same sampling date as the equipment blank
Field duplicates	Field duplicates > RLs AND RPD outside CL	J for the positive results UJ for the nondetects	The specific analyte(s) in all samples collected on the same sampling date
MS/MSD	MS or MSD % R > UCL OR MS or MSD % R < LCL OR MS/MSD RPD > CL	M for all results	The specific analyte(s) in all samples collected from the same site as the parent sample
Sample Preservation/ Collection	Preservation/collection requirements not met	R for all results	All analytes in the sample
Sample Storage	< 2°C or > 6°C or as required	J for the positive results R for the nondetects	All analytes in the sample

UCL = upper control limit    LCL = lower control limit    CL = control limit

	<b>Criteria</b>	<b>Flag*</b>
Quantitation	$\leq$ MDL	U
	> MDL < RL	F
	$\geq$ RL	as needed
	$\geq$ high std / linear range	J

\* Example 1: if the MDL is 0.04, the RL is 0.9 and the result is 0.03, the concentration reported on the result form would be 0.04 (the MDL) and the qualifier flag would be U.

Example 2: if the MDL is 0.04, the RL is 0.9 and the result is 0.07, the concentration reported on the result form would be 0.07 and the qualifier flag would be F.

Example 3: if the MDL is 0.04, the RL is 0.9 and the result is 1.2, the concentration reported on the result form would be 1.2 and the qualifier would be any flag needed because of a data quality problem (e.g., R, J, B, etc.).





**Table 8.2.2.4-2**  
**Flagging Conventions Specific to Organic Methods**

QC Requirement	Criteria	Flag	Flag Applied To
Ambient Blank (VOC samples only)	Analyte(s) detected $\geq$ RL	B	The specific analyte(s) in all samples with the same matrix and sampling date
Trip Blank (VOC samples only)	Analyte(s) detected $\geq$ RL	B	The specific analyte(s) in all samples shipped in the same cooler as the blank
Initial Five Point Calibration (GC & HPLC methods)	Linearity criterion not met	N/A	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
Initial Five Point Calibration (GC/mass spectrometry methods)	SPCC or CCC criteria not met	N/A	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
	Linearity criterion not met	N/A	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
Second Source Calibration Verification	CL exceeded	N/A	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
Initial Daily Calibration Verification (GC & HPLC methods)	CL exceeded	N/A	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
Calibration Verification (GC/mass spectrometry methods)	SPCC or CCC criteria not met	Q	All analytes in all samples associated with the calibration verification
Retention time	Retention time of analyte outside of established retention time window	Q	The specific analyte(s) in the sample
Surrogates	surrogate % R > UCL OR surrogate % R < LCL OR surrogate recovery < 10%	J for the positive results  J for the positive results UJ for the nondetects  Q for all results	All analytes in the sample associated with the surrogate
Mass Spectrometer Tune	Ion abundance criteria not met	R for all results	All analytes in all samples associated with the tune

UCL = upper control limit    LCL = lower control limit    CL = control limit



**Table 8.2.2.4-2  
Flagging Conventions Specific to Organic Methods (Continued)**

<b>QC Requirement</b>	<b>Criteria</b>	<b>Flag</b>	<b>Flag Applied To</b>
Second Column/Second Detector Confirmation (GC & HPLC methods)	Not performed	R	All analytes $\geq$ RL
	Agreement between results not within $\pm 40\%$	J	All affected analytes
Internal Standard	Retention time not within $\pm 30$ seconds; EICP area not within -50% to +100% of last calibration verification	Q	Apply Q to all results for specific analytes associated with the IS
Tentatively Identified Compounds (TICs)		T	All TICs



ERPIMS is a data management system designed to accommodate all types of data collected for IRP projects. Specific codes and data forms have been developed to allow consistent and efficient input of information to the system. The database information shall be provided by the prime contractor via ASCII files in specified ERPIMS format on 3.5" floppy diskettes. The information transferred shall include all required technical data such as site information; well characteristics; and hydrogeologic, geologic, physical, and chemical analysis results. Electronic data reporting formats and requirements are given in the most current version of the *ERPIMS Data Loading Handbook*.

## **8.5 ARCHIVING**

Hardcopy and electronic data shall be archived in project files and on electronic archive tapes for the duration of the project or a minimum of five years, whichever is longer.

## **8.6 PROJECT DATA FLOW AND TRANSFER**

The data flow from the laboratory and field to the project staff and data users shall be sufficiently documented to ensure the data are properly tracked, reviewed, and validated for use.

## **8.7 RECORD KEEPING**

The laboratory shall maintain electronic and hardcopy records sufficient to recreate each analytical event conducted pursuant to the Scope of Work. The minimum records the laboratory shall keep contain the following: (1) CoC forms, (2) initial and continuing calibration records including standards preparation traceable to the original material and lot number, (3) instrument tuning records (as applicable), (4) method blank results, (5) IS results, (6) surrogate spiking records and results (as applicable), (7) spike and spike duplicate records and results, (8) laboratory records, (9) raw data, including instrument printouts, bench work sheets, and/or chromatograms with compound identification and quantitation reports, (10) corrective action reports, (11) other method and project required QC samples and results, and (12) laboratory-specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples.

## **8.8 HARD COPY DATA REPORT FORMS FOR REPORTING SCREENING DEFINITIVE DATA**

AFCEE forms described below shall be included in this project *QAPP* and used unless a variance is requested and approved in advance and that the forms included in this project *QAPP*, to be used by the contractor can be verified that they contain at a minimum the information requested on the AFCEE forms.

A screening data report package shall consist of the following AFCEE forms: CoC, S-1, S-2, and S-3.

A definitive data inorganic report package shall consist of the following AFCEE forms: CoC, I-1, I-2, I-3, I-3A, I-3B, I-4, I-5, I-6, I-7, I-8, I-9, I-10, I-11, and I-12, for each AAB with inorganic analyses performed.



A definitive data organic report package shall consist of the following AFCEE forms: CoC, O-1, O-2, O-3, O-3A, O-4, O-5, O-5A, O-6, O-7, O-8, O-9, O-10, O-11, and O-12 for each AAB with organic analyses performed.

A definitive data wet chemistry report package shall consist of the following AFCEE forms: CoC, W-1, W-2, W-3, W-4, W-5, W-6, W-7, W-8, and W-9 for each AAB with wet chemistry analyses performed.

Exceptions to these report forms are as follows: for mercury analysis, form I-3A shall be substituted for form I-3 in the inorganic report package; for cyanide analysis, form I-3B shall be substituted for form I-3 in the inorganic report package; for GC/mass spectrometry analyses, forms O-3A and O-5A shall be used and form O-11 shall be added to the organic report package. A complete list and description of forms is provided in Table 8.8-1. Other forms shall be included in this project *QAPP*, as needed.

### **INSTRUCTIONS FOR COMPLETING AFCEE REPORT FORMS**

The following instructions shall be used in completing the AFCEE report forms for screening and definitive data. The bold lettering identifies the fields on the AFCEE report form.

Use as many sheets as necessary. Sheets may be duplicated with only those sections necessary to be completed filled out (i.e., you do not have to duplicate previously reported information from one sheet to the next). Sequentially number the sheets at the bottom of the page if more than one sheet is necessary.

**Reporting Dilutions:** Justification for diluting samples shall be provided in the comments section on the appropriate form (i.e., I-2, O-2, or W-2). If the result for any analyte is outside the calibration range (i.e., greater than the highest calibration standard), the sample shall be diluted appropriately and reanalyzed. Results from the undiluted and diluted sample shall be reported on the appropriate form (i.e., I-2, O-2, or W-2). The results of the analysis of the diluted sample shall be reported with the dilution

#### ALL INORGANIC, ORGANIC AND WET CHEM FORMS

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Lab Name:** enter the laboratory name (e.g., Garland Labs, Inc.)
- **Contract #:** enter the Air Force contract number and delivery order number under which the analytical work is being performed (e.g., F21625-94-D-8005/0001)
- **Comments:** enter any comments

#### FORM I-1

- **Base/Command:** enter the base name and the Air Force command (e.g., Banks AFB/SPACECOM)
- **Prime Contractor:** enter the name of the prime contractor (e.g., RDS, Inc)



**Table 8.8-1**  
**List of AFCEE Analytical Forms**

AFCEE Form Number	Description
I-1	Inorganic Analyses Data Package
I-2	Inorganic Analyses Data Sheet 2 – Results
I-3	Inorganic Analyses Data Sheet 3 – Initial Multipoint Calibration
I-3A	Inorganic Analyses Sheet 3a – Mercury Initial Multipoint Calibration
I-3B	Inorganic Analyses Data Sheet 3b – Cyanide Initial Multipoint Calibration
I-4	Inorganic Analyses Sheet 4 – Calibration Verification
I-5	Inorganic Analyses Data Sheet 5 – ICP-Mass Spectrometry Tune
I-6	Inorganic Analyses Data Sheet 6 –Serial Dilution
I-7	Inorganic Analyses Data Sheet 7 – Post-Digestion Spike Sample Recovery
I-8	Inorganic Analyses Data Sheet 8 – Blanks
I-9	Inorganic Analyses Data Sheet 9 – Laboratory Control Sample
I-10	Inorganic Analyses Data Sheet 10 – Matrix Spike/Matrix Spike Duplicate Sample Recovery
I-11	Inorganic Analyses Data Sheet 11 – Holding Times
I-12	Inorganic Analyses Data Sheet 12 – Instrument Analysis Sequence Log
O-1	Organic Analyses Data Package
O-2	Organic Analyses Sheet 2 – Results
O-3	Organic Analyses Data Sheet 3a – Initial Multipoint Calibration
O-3A	Organic Analyses Data Sheet 3A – Initial Multipoint Calibration-GC/Mass Spectrometry Analysis
O-4	Organic Analyses Data Sheet 4 – Second Source Calibration Verification
O-5	Organic Analyses Data Sheet 5 – Calibration Verification
O-5A	Organic Analyses Data Sheet 5A – Calibration Verification-GC/Mass Spectrometry Analysis
O-6	Organic Analyses Data Sheet 6 – Second Column/Detector Confirmation
O-7	Organic Analyses Data Sheet 7 – Blank
O-8	Organic Analyses Data Sheet 8 – Laboratory Control Sample
O-9	Organic Analyses Data Sheet 9 – Matrix Spike/Matrix Spike Duplicate Sample Recovery
O-10	Organic Analyses Data Sheet 10 – Holding Times
O-11	Organic Analyses Data Sheet 11 – Instrument Analysis Sequence Log
O-12	Organic Analyses Data Sheet 12 – GC/Mass Spectrometry Performance Check (BFP OR DFTPP)
W-1	Wet Chem Analyses Data Package
W-2	Wet Chem Analyses Data Sheet 2
W-3	Wet Chem Analyses Data Sheet 3 – Initial Multipoint Calibration
W-4	Wet Chem Analyses Data Sheet 4 – Calibration Verification
W-5	Wet Chem Analyses Data Sheet 5 – Blanks
W-6	Wet Chem Analyses Data Sheet 6 – Laboratory Control Sample



**Table 8.8-1**  
**List of AFCEE Analytical Forms (Continued)**

AFCEE Form Number	Description
W-7	Wet Chem Analyses Data Sheet 7 – Matrix Spike/Matrix Spike Duplicate Sample Recovery
W-8	Wet Chem Analyses Data Sheet 8 – Holding Times
W-9	Wet Chem Analyses Data Sheet 9 – Instrument Analyses Sequence Log
S-1	Screening Data Package
S-2	Screening Data Sheet 2 – Results
S-3	Screening Data Sheet 3 – Field Duplicates
MDL Form (Method Specific)	MDL Study Report Form
CoC	Chain of Custody Record



- **Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate, and field blanks)
- **Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that corresponds to the Field Sample ID

### FORM I-2

This form is completed for all environmental samples including the MS and MSD.

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate, and field blanks)
- **Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that corresponds to the Field Sample ID
- **Matrix:** enter the sample matrix (e.g., water and soil)
- **% Solids:** enter the % solids
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the sample results
- **Date Received/Prepared/Analyzed:** enter the appropriate dates in the format DD-*MMM*-YY (e.g., 3 Jun 04)
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg dry weight)
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7
- **MDL:** enter the laboratory derived method detection limit
- **RL:** enter the project AFCEE reporting limit as stated in the *QAPP* or approved variance for each analyte
- **Concentration:** enter the numeric result
- **Dilution:** enter the dilution (if applicable) (e.g., 1:5)
- **Q:** enter the qualifier flag (see *QAPP* Sections 7 and 8)

### FORM I-3

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)





- **Date of Initial Calibration:** enter the appropriate date in the format DD-*MMM*-YY (e.g., 3 Jun 04)
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Std 1, Std2, Std3, ...:** enter the concentrations of the standards
- **r:** enter the correlation coefficient
- **Q:** enter a "\*" for all corresponding correlation coefficients that were not acceptable as per *QAPP* Section 7

#### FORM I-3A

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Date of Initial Calibration:** enter the appropriate date in the format DD-*MMM*-YY (e.g., 3 Jun 04)
- **Initial Calibration ID:** enter the unique identifying number given to this initial calibration event
- **Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)
- **Std 1, Std 2, Std 3, Std 4, Std 5, ...:** enter the concentrations of the standards
- **r:** enter the correlation coefficient
- **Q:** enter a "\*" for all corresponding correlation coefficients that were not acceptable as per *QAPP* Section 7

#### FORM I-3B

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Date of Initial Calibration:** enter the appropriate date in the format DD-*MMM*-YY (e.g., 3 Jun 04)



- **Initial Calibration ID:** enter the unique identifying number given to this initial calibration event
- **Concentration Units:** enter the appropriate units (i.e., mg/L or mg/kg)
- **Std 1, Std 2, Std 3, Std 4, Std 5, Std 6, ...:** enter the concentrations of the standards
- **r:** enter the correlation coefficient
- **Q:** enter a "\*" for all corresponding correlation coefficients that were not acceptable as per *QAPP* Section 7
- **Expected:** enter the expected result (i.e., the concentration of the calibration material)
- **Found:** enter the measured result
- **%D:** enter the percent difference between the expected and found

#### FORM I-4

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if these calibration events pertain to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the calibration verification results
- **2nd Source ID:** enter the unique identifier for the 2nd source standard such that the standard could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., 2S040603])
- **CCV #1 ID:** enter the unique identification number for the first CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., CCV040603-1])
- **CCV #2 ID:** enter the unique identification number for the second CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., CCV040603-2])
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7
- **Expected:** enter the expected result (i.e., the concentration of the calibration material)
- **Found, Found 1, and Found 2:** enter the measured result. Found 1 corresponds to the first CCV run, Found 2 corresponds to the second CCV run, etc.
- **%D:** enter the percent difference between the expected and found
- **Q:** enter a "\*" for any %D that was not acceptable as per *QAPP* Section 7



#### FORM I-5

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Injection Date/Time:** enter the date (in the format DD-MMM-YY) and time (in 24-hour format) of the performance check
- **Element:** enter element as appropriate
- **Mass:** enter the mass of the ion used for tuning (see *QAPP* Section 7)
- **Average Measured Mass (amu):** enter the average measured mass
- **Average Peak width at 10% Peak Height (amu):** enter average peak width at 10% peak height (amu)
- **% RSD:** enter the percent relative standard deviation in the average measured mass

#### FORM I-6

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Analytical Method:** enter the analytical method
- **Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Matrix:** enter the sample matrix (e.g., water, soil)
- **Date of Analysis:** enter the date of analysis
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7
- **Initial Sample Result:** enter the initial sample result and any data qualifier (See *QAPP* Sections 7 and 8)
- **Serial Dilution Result:** enter the measured result of the diluted sample and any data qualifier (See *QAPP* Sections 7 and 8)
- **% Difference:** enter the percent difference
- **Q:** enter a “\*” for any %D that was not acceptable as per *QAPP* Section 7



#### FORM I-7

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Matrix:** enter the sample matrix (e.g., water, soil)
- **Date of Analysis:** enter the date of analysis
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7
- **CLs:** enter the CLs required to be met (see *QAPP* Section 7)
- **Spiked Sample Result:** enter the numeric result of the spiked sample and any data qualifier (See *QAPP* Sections 7 and 8)
- **Sample Result:** enter the numeric result of the parent sample and any data qualifier (See *QAPP* Sections 7 and 8). If an analyte was not detected above the MDL, leave this column blank
- **Spike Added:** enter the amount of spike added to the parent sample
- **%R:** enter the percent recovery
- **Q:** enter a "\*" for any %R that was not acceptable as per *QAPP* Section 7

#### FORM I-8

- **AAB#:** enter the unique AFCEE analytical batch number for the method blank (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Initial Calibration Blank ID:** enter the identification number for the calibration blank (the same ID number will be found in the run sequence log (e.g., CB040603))
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the initial calibration blank results
- **Method Blank ID:** enter the unique identifying number given to the method blank (the same ID number will be found in the run sequence log (e.g., MB040603))
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the method blank results
- **CCB #1 ID:** (used for 6010B/6020 analysis) enter the identification number for the first CCB (the same ID number will be found in the run sequence log [e.g., CCB040603-1])



- **CCB #2 ID:** (used for 6010B/6020 analysis) enter the identification number for the second CCB (the same ID number will be found in the run sequence log [e.g., CCB040603-2])
- **CCB #3 ID:** (used for 6010B/6020 analysis) enter the identification number for the third CCB (the same ID number will be found in the run sequence log [e.g., CCB040603-3])
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7
- **Initial Calibration Blank:** enter a numeric result for the calibration blank
- **Continuing Calibration Blank 1:** enter a numeric result for the first continuing calibration blank run
- **Continuing Calibration Blank 2:** enter a numeric result for the second continuing calibration blank run
- **Continuing Calibration Blank 3:** enter a numeric result for the third continuing calibration blank run
- **Method Blank:** enter a numeric result for the method blank
- **RL:** enter the project AFCEE reporting limit as stated in the *QAPP* or approved variance for each analyte
- **Q:** enter a “\*” for any calibration or method blank analytes that were not acceptable as per *QAPP* Section 7

#### FORM I-9

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **LCS ID:** enter the unique identification number for the laboratory control sample such that the LCS could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., LCS040603])
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the LCS results
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7
- **Expected:** enter the expected result (i.e., the concentration at which the analyte was spiked in LCS material)
- **Found:** enter the measured result of the LSC analytes
- **%R:** enter the percent recovery
- **CLs:** enter the CLs required to be met (see *QAPP* Section 7)



- **Q:** enter a “\*” for any %R that was not acceptable as per *QAPP* Section 7

#### FORM I-10

- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **% Solids:** enter the % solids of the parent field sample
- **Parent Field Sample ID:** enter the field sample ID of the parent sample (the sample spiked for the MS and MSD)
- **MS ID:** enter the unique identification number for the matrix spike such that the MS could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log [e.g., MS040603])
- **MSD ID:** enter the unique identification number for the matrix spike duplicate such that the MSD could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log [e.g., MSD040603])
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7
- **Parent Sample Result:** enter the numeric result of the parent sample. If an analyte was not detected above the MDL, leave this column blank
- **Spike Added:** enter the amount of spike added to the parent sample
- **Spiked Sample Result:** enter the numeric result of the MS
- **%R:** enter the percent recovery
- **Duplicate Spiked Sample Result:** enter the numeric result of the MSD
- **%RPD:** enter the relative percent difference between the spike (MS) and spike duplicate (MSD)
- **CLs %R:** enter the CLs required to be met (see *QAPP* Section 7)
- **CLs %RPD:** enter the CLs required to be met (see *QAPP* Section 7)
- **Q:** enter the qualifier flag as needed (see *QAPP* Sections 7 and 8)

#### FORM I-11

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)
- **Date Collected:** enter the date the sample was taken in the field in the format DD-*MMM*-YY (e.g., 6 Jun 04)
- **Date Received:** enter the date the sample was received at the laboratory in the format DD-*MMM*-YY (e.g., 6 Jun 04)





- **Date Analyzed:** enter the date the sample was analyzed by the laboratory in the format DD-MMM-YY (e.g., 6 Jun 04)
- **Max. Holding Time:** enter the maximum allowable holding time in days (see *QAPP* Section 5)
- **Time Held:** enter the time in days elapsed between the date collected and the date analyzed
- **Q:** enter a “\*” for any holding times that were greater than the maximum allowable holding time as per *QAPP* Section 5

#### FORM I-12

- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Field Sample ID/Std ID/Blank ID/QC Sample ID:** enter the unique identifying number of each sample (environmental sample, standard, blank, LCS, MS, MSD, etc.) in the sequence they were analyzed
- **Date Analysis Started:** enter the date the sample analysis was started in the format DD-MMM-YY (e.g., 6 Jun 04)
- **Time Analysis Started:** enter the time the sample analysis was started in 24-hour format (e.g., 0900 and 2130)
- **Date Analysis Completed:** enter the date the sample analysis was completed in the format DD-MMM-YY (e.g., 6 Jun 04)
- **Time Analysis Completed:** enter the time the sample analysis was completed in 24-hour format (e.g., 0900 and 2130)

#### FORM O-1

- **Base/Command:** enter the base name and the Air Force command (e.g., Banks AFB/SPACECOM)
- **Prime Contractor:** enter the name of the prime contractor (e.g., RDS, Inc)
- **Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)
- **Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that corresponds to the Field Sample ID

#### FORM O-2

This form is completed for all environmental samples including the MD and MSD.

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)





- **Lab Sample ID:** enter the unique identifying number given to the sample by the laboratory that corresponds to the Field Sample ID
- **Matrix:** enter the sample matrix (e.g., water, soil)
- **% Solids:** enter the % solids
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the sample results
- **Date Received/Prepared/Analyzed:** enter the appropriate dates in the format DD-  
MMM-YY (e.g., 3 Jun 96)
- **Concentration Units:** enter the appropriate units (i.e.,  $\mu\text{g/L}$  or  $\text{mg/kg}$  dry weight)
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP*  
Section 7
- **MDL:** enter the laboratory derived method detection limit
- **RL:** enter the project AFCEE reporting limit as stated in the *QAPP* or approved variance  
for each analyte
- **Concentration:** enter the numeric result
- **Dilution:** enter the dilution (if applicable) (e.g., 1:5)
- **Confirm:** enter the numeric result from the confirmation column/detector
- **Qualifier:** enter the qualifier flag as needed (see *QAPP* Section 7)
- **Surrogate:** enter the name of the surrogate(s) used
- **Recovery:** enter the percent recovery of the surrogate
- **CLs:** enter the CLs for the recovery of the surrogate (see *QAPP* section 7)
- **Internal Std:** (used for 8260B and 8270C analysis) enter the name of the internal  
standard(s) used

#### FORM O-3 and 3A

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration  
pertains to all of the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a  
definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other  
identifying number/name)
- **Date of Initial Calibration:** enter the appropriate date in the format DD-  
MMM-YY  
(e.g.,  
3 Jun 96)
- **Initial Calibration ID:** enter the unique identifying number given to the initial  
calibration event
- **Concentration Units:** enter the appropriate units (i.e.,  $\mu\text{g/L}$  or  $\text{mg/kg}$ )
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP*  
Section 7. (On form 3A, some analyte names already appear on the form as provided,  
leave those analytes in that order.)



- **RF1, RF2, RF3, RF4, RF5, RF6, RF7:** enter the response factor corresponding to the standard with the same number (RF6 and RF7 are used for nonlinear calibrations)
- **Std 1, Std 2, Std 3, Std 4, Std 5, Std 6, Std 7:** enter the concentration of the standard (Std 6 and Std 7 are used for nonlinear calibrations)
- **%RSD:** enter the percent relative standard deviation of the response factors
- **Mean %RSD:** enter the mean of the RSDs of all analytes for those analytes not using a least squares regression or non-linear calibration
- **r:** (optional) if least squares regression is used for the calibration of an analyte, enter the correlation coefficient
- **COD:** (optional) if a non-linear calibration is used for the calibration of an analyte, enter the coefficient of determination
- **Q:** enter a “\*” for any calibration that was not acceptable as per *QAPP* Section 7 and for any RFs not meeting minimum requirements for SPCCs and/or CCCs

#### FORM O-4

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration event pertains to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the second source calibration verification results
- **2nd Source ID:** enter the unique identifier for the 2nd source standard such that the standard could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., 2S960603)
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7
- **Expected:** enter the expected result (i.e., the concentration of the calibration material)
- **Found:** enter the measured result
- **%D:** enter the percent difference between the expected (i.e., the concentration of the second source calibration material) and measured result
- **Q:** enter a “\*” for any % D that was not acceptable as per *QAPP* Section 7

#### FORM O-5 and O-5A

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if these calibration events pertain to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)



- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the calibration verification results
- **ICV ID:** enter the unique identification number for the ICV such that the ICV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., ICV960603-1)
- **CCV #1 ID:** enter the unique identification number for the CCV run after the first 12 hours of operation such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-1)
- **CCV #2 ID:** enter the unique identification number for the CCV run after the second 12 hours of operation such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-2)
- **Analyte:** enter all analyte names in the same order as listed in the tables in *QAPP* Section 7 (On form O-5A, some analyte names already appear on the form as provided, leave those analytes in that order.)
- **RF:** (form O-5A) enter the response factor for the SPCCs only
- **% D:** enter the percent difference
- **% D or % drift:** (form O-5) enter the percent difference if using RFs or % drift if using CFs
- **Q:** enter a "\*" for any % drift that was not acceptable as per requirements in *QAPP* Section 7
- **AAB#:** enter the unique AFCEE analytical batch number for the method blank (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Concentration Units:** enter the appropriate units (i.e.,  $\mu\text{g/L}$  or  $\text{mg/kg}$ )
- **Method Blank ID:** enter the unique identification number for the method blank (the same ID number will be found in the run sequence log, e.g., MB960603)

#### FORM O-6

- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the method blank results
- **Analyte:** enter the name of the analyte (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Method Blank:** enter a numeric result for the method blank
- **RL:** enter the project AFCEE reporting limit as stated in the *QAPP* or approved variance for each analyte
- **Q:** enter a "\*" for any method blank analyte result that was not acceptable as per *QAPP* Section 7
- **Surrogate:** enter the name of the surrogate(s) used
- **Recovery:** enter the percent recovery of the surrogate



### FORM O-7

- **CLs:** enter the CLs for the recovery of the surrogate (see *QAPP* section 7)
- **Internal Std:** (used for 8260B and 8270C analysis) enter the name of the internal standard(s) used
- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **LCS ID:** enter the unique identification number for the laboratory control sample such that the LCS could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., LCS960603)
- **Concentration Units:** enter the appropriate units (i.e.,  $\mu\text{g/L}$  or  $\text{mg/kg}$ )
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the LCS results
- **Analyte:** enter the name of the analyte (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Expected:** enter the expected result (i.e., the concentration at which the analyte was spiked in the LCS)
- **Found:** enter the measured result of the LSC analytes
- **%R:** enter the percent recovery
- **CLs:** enter the CLs required to be met (see *QAPP* Section 7)
- **Q:** enter a "\*" for any % R that was not acceptable as per *QAPP* Section 7
- **Surrogate:** enter the name of the surrogate(s) used
- **Recovery:** enter the percent recovery of the surrogate
- **Internal Std:** (used for 8260B and 8270C analysis) enter the name of the internal standard(s) used

### FORM O-8

- **Concentration Units:** enter the appropriate units (i.e.,  $\mu\text{g/L}$  or  $\text{mg/kg}$ )
- **Parent Field Sample ID:** enter the field sample ID of the parent sample (the sample spiked for the MS and MSD)
- **% Solids:** enter the % solids
- **MS ID:** enter the unique identification number for the matrix spike such that the MS could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log, e.g., MS960603)
- **MSD ID:** enter the identification number for the matrix spike duplicate such that the MSD could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log, e.g., MSD960603)
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the MS/MSD results



- **Analyte:** enter the name of the analyte (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Parent Sample Result:** enter the result of the parent sample. If an analyte was not detected above the MDL, leave this column blank.
- **Spike Added:** enter the amount of spike added to the parent sample
- **Spiked Sample Result:** enter the numeric result of the MS
- **%R:** enter the percent recovery
- **Duplicate Spiked Sample Result:** enter the numeric result of the MSD
- **%RPD:** enter the relative percent difference between the spike (MS) and spike duplicate (MSD)
- **CLs %R:** enter the CLs required to be met (see *QAPP* Section 7)
- **CLs %RPD:** enter the CLs required to be met (see *QAPP* Section 7)
- **Q:** enter the qualifier flag as needed (see *QAPP* Sections 7)

#### FORM O-9

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)
- **Date Collected:** enter the date the sample was taken in the field in the format DD-MMM-YY (e.g., 3 Jun 96)
- **Date Received:** enter the date the sample was received at the laboratory in the format DD-MMM-YY (e.g., 3 Jun 96)
- **Date Extracted:** enter the date the sample was extracted by the laboratory in the format DD-MMM-YY (e.g., 3 Jun 96)
- **Max. Holding Time E:** enter the maximum allowable holding time in days until the sample is extracted (if applicable - see *QAPP* Section 5)
- **Time Held Ext.:** enter the time in days elapsed between the date collected and the date extracted (if applicable)
- **Date Analyzed:** enter the date the sample was analyzed by the laboratory in the format DD-MMM-YY (e.g., 3 Jun 96)
- **Max. Holding Time A:** enter the maximum allowable holding time in days until the sample is analyzed (see *QAPP* Section 5)
- **Time Held Anal.:** enter the time in days elapsed between the date collected and the date analyzed
- **Q:** enter a "\*" for any holding time (Max. Holding Time E, or Max. Holding Time A, or Time Held Anal.) that was greater than the maximum holding time that was not acceptable as per *QAPP* Section 5



#### FORM O-10

- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Field Sample ID/Std ID/Blank ID/QC Sample ID:** enter the unique identifying number of each sample (environmental sample, standard, blank, LCS, MS, MSD, etc.) in the sequence they were analyzed
- **Date Analysis Started:** enter the date the sample analysis was started in the format DD-MMM-YY (e.g., 3 Jun 96)
- **Time Analysis Started:** enter the time the sample analysis was started in 24-hour format (e.g., 0900, 2130)
- **Date Analysis Completed:** enter the date the sample analysis was completed in the format DD-MMM-YY (e.g., 3 Jun 96)
- **Time Analysis Completed:** enter the time the sample analysis was completed in 24-hour format (e.g., 0900, 2130)

#### FORM O-11

- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Compound:** enter BFB or DFTPP as appropriate
- **Injection Date/Time:** enter the date (in the format DD-MMM-YY) and time (in 24-hour format) of the performance check
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the MS/MSD results
- **Mass:** enter the mass of the ion used for tuning (see *QAPP* Section 7)
- **Ion Abundance Criteria:** enter the criteria for the specific mass (see *QAPP* Section 7)
- **% Relative Abundance:** enter the percent relative abundance as the result of the tune
- **Q:** enter a "\*" for any % relative abundance results that was not acceptable as per *QAPP* Section 7

#### FORM O-12

- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Compound:** enter BFB or DFTPP as appropriate
- **Injection Date/Time:** enter the date (in the format DD-MMM-YY) and time (in 24-hour format) of the performance check
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the MS/MSD results
- **Mass:** enter the mass of the ion used for tuning (see *QAPP* Section 7)





- **Ion Abundance Criteria:** enter the criteria for the specific mass (see *QAPP* Section 7)
- **% Relative Abundance:** enter the percent relative abundance as the result of the tune
- **Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the *QAPP*)
- **MDL:** enter the laboratory derived method detection limit
- **RL:** enter the project AFCEE reporting limit as stated in the *QAPP* or approved variance for each analyte
- **Concentration:** enter the numeric result
- **Dilution:** enter the dilution (if applicable) (e.g., 1:5)
- **Q:** enter the qualifier flag (see *QAPP* Sections 7 and 8)

#### FORM W-3

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if this calibration pertains to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Date of Initial Calibration:** enter the appropriate date in the format DD-MMM-YY (e.g., 3 Jun 04)
- **Initial Calibration ID:** enter the unique identifying number given to this initial calibration event
- **Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Std 1, Std2, Std3:** enter the concentration of the standard
- **r:** enter the correlation coefficient
- **Q:** enter a "\*" for any correlation coefficients that were not acceptable as per *QAPP* Section 7

#### FORM W-4

- **AAB#:** (optional) enter the unique AFCEE analytical batch number if these calibration events pertain to all the samples from one batch (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the calibration verification results





- **2nd Source ID:** enter the unique identifier for the 2nd source standard such that the standard could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., 2S040603])
- **ICV ID:** enter the unique identification number for the ICV such that the ICV could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., ICV040603])
- **CCV #1 ID:** enter the unique identification number for the first CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., CCV040603-1])
- **CCV #2 ID:** enter the unique identification number for the second CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., CCV040603-2])
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Expected:** enter the expected result (i.e., the concentration of the calibration material)
- **Found, Found 1, Found 2:** enter the measured result. Found 1 corresponds to the first CCV run, Found 2 corresponds to the second CCV run, etc.
- **%D:** enter the percent difference between the expected and found
- **Q:** enter a "\*" for any %D that was not acceptable as per *QAPP* Section 7

#### FORM W-5

- **AAB#:** enter the unique AFCEE analytical batch number for the method blank (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Calibration Blank ID:** enter the identification number for the calibration blank (the same ID number will be found in the run sequence log [e.g., CB040603])
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the calibration blank results
- **Method Blank ID:** enter the identification number for the method blank (the same ID number will be found in the run sequence log [e.g., MB040603])
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the method blank results
- **Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Calibration Blank:** enter a numeric result for the calibration blank
- **Method Blank:** enter a numeric result for the method blank



- **RL:** enter the project AFCEE reporting limit as stated in the *QAPP* or approved variance for each analyte
- **Q:** enter a "\*" for any calibration or method blank analyte that was not acceptable as per *QAPP* Section 7

#### FORM W-6

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **LCS ID:** enter the unique identification number for the laboratory control sample such that the LCS could be traced back to its source material (the same ID number will be found in the run sequence log [e.g., LCS040603])
- **Initial Calibration ID:** enter the unique identifying number given to the initial calibration event used in the determination of the LCS results
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Expected:** enter the expected result (i.e., the concentration at which the analyte was spiked in LCS material)
- **Found:** enter the measured result of the LCS analyte\
- **%R:** enter the percent recovery
- **CLs:** enter the CLs required to be met (see *QAPP* Section 7)
- **Q:** enter a "\*" for any %R that was not acceptable as per *QAPP* Section 7

#### FORM W-7

- **% Solids:** enter the % solids
- **Parent Field Sample ID:** enter the field sample ID of the parent sample (the sample spiked for the MS and MSD)
- **MS ID:** enter the unique identification number for the matrix spike such that the MS could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log [e.g., MS040603])
- **MSD ID:** enter the unique identification number for the matrix spike duplicate such that the MSD could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log [e.g., MSD040603])
- **Concentration Units:** enter the appropriate units (e.g., mg/L, mg/kg)
- **Analyte:** enter the name of the analytes (use the same name as used in the tables in Section 7 of the *QAPP*)



- **Parent Sample Result:** enter the numeric result of the parent sample. If an analyte was not detected above the MDL, leave this column blank
- **Spike Added:** enter the amount of spike added to the parent sample
- **Spiked Sample Result:** enter the numeric result of the MS
- **%R:** enter the percent recovery
- **Duplicate Spiked Sample Result:** enter the numeric result of the MSD
- **%RPD:** enter the relative percent difference between the spike (MS) and spike duplicate (MSD)
- **CLs %R:** enter the CLs required to be met (see *QAPP* Section 7)
- **CLs %RPD:** enter the CLs required to be met (see *QAPP* Section 7)
- **Q:** enter the qualifier flag as needed (see *QAPP* Sections 7 and 8)

#### FORM W-8

- **AAB#:** enter the unique AFCEE analytical batch number (see Section 4.4 of the *AFCEE QAPP* for a definition of a batch)
- **Field Sample ID:** enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate, and field blanks)
- **Date Collected:** enter the date the sample was taken in the field in the format DD-  
MMM-YY (e.g., 6 Jun 04)
- **Date Received:** enter the date the sample was received at the laboratory in the format DD-  
MMM-YY (e.g., 6 Jun 04)
- **Date Analyzed:** enter the date the sample was analyzed by the laboratory in the format DD-  
MMM-YY (e.g., 6 Jun 04)
- **Max. Holding Time:** enter the maximum allowable holding time in days (see *QAPP* Section 5)
- **Time Held:** enter the time in days elapsed between the date collected and the date analyzed
- **Q:** enter a "\*" for any holding time that was greater than the maximum allowable holding time as per *QAPP* Section 5

#### FORM W-9

- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Field Sample ID/Std ID/Blank ID/QC Sample ID:** enter the unique identifying number of each sample (environmental sample, standard, blank, LCS, MS, MSD, etc.) in the sequence they were analyzed



- **Date Analysis Started:** enter the date the sample analysis was started in the format DD-  
MMM-YY (e.g., 6 Jun 04)
- **Time Analysis Started:** enter the time the sample analysis was started in 24-hour format  
(e.g., 0900 and 2130)
- **Date Analysis Completed:** enter the date the sample analysis was completed in the  
format DDMMM-YY (e.g., 6 Jun 04)
- **Time Analysis Completed:** enter the time the sample analysis was completed in 24-hour  
format (e.g., 0900 and 2130)

#### FORM S-1

- **Base/Command:** enter the base name and the Air Force command (e.g., Banks  
AFB/SPACECOM)
- **Prime Contractor:** enter the name of the prime contractor (e.g., RDS, Inc)
- **Field Sample ID:** enter the unique identifying number given to the field sample (includes  
MS, MSD, field duplicate, and field blanks)
- **Signature:** signature of person completing data package
- **Name:** name of person completing data package
- **Date:** enter the date the in the format DD-MMM-YY (e.g., 6 Jun 04)
- **Title:** title of person completing data package

#### FORM S-2

- **Field Sample ID:** enter the unique identifying number given to the field sample (includes  
MS, MSD, field duplicate, and field blanks)
- **Matrix:** enter the sample matrix (e.g., water and soil)
- **Date Analyzed:** enter the appropriate dates in the format DD-MMM-YY (e.g., 3 Jun 04)
- **Units:** enter the appropriate units (e.g., µg/L, mg/kg, degrees C ...)
- **Analyte/Test:** enter the name of the analyte or test performed (e.g., pH)
- **MDL:** enter the method detection limit if applicable
- **RL:** enter the project AFCEE reporting limit as stated in the *QAPP* or approved variance  
for each analyte
- **Result:** enter the result
- **Q:** enter the qualifier needed (see *QAPP* Sections 7 and 8)

#### FORM S-3

- **Units:** enter the appropriate units (e.g., µg/L, mg/kg, degrees C...)



- **Analyte/Test:** enter the name of the analyte or test performed (e.g., pH)
- **Sample Result:** enter the result of the sample
- **Duplicate Sample Result:** enter the result of the duplicate sample
- **%D or %RPD:** enter the percent or difference relative percent difference between the sample and duplicate as appropriate
- **Acceptance Criteria:** enter the acceptance criteria required to be met (see *QAPP* Section 6)
- **Q:** enter a “\*” for any % D or % RPD that was not acceptable as per *QAPP* Section 6

#### MDL FORM

- **Matrix:** enter the sample matrix (e.g., water and soil)
- **Analysis Date:** enter the date (or inclusive dates if performed over a period of days) the MDL was performed in the format DD-MMM-YY (e.g., 6 Jun 04)
- **Instrument ID:** enter the instrument identifier (e.g., the serial number or other identifying number/name)
- **Analyte:** enter the name of the analyte (use the same name as used in the tables in Section 7 of the *QAPP*)
- **Amt. Spiked:** enter the amount of spike added to the matrix
- **Replicate 1, 2, 3, 4, 5, 6, 7...:** enter the result of the replicate
- **Std. Dev.:** enter the standard deviation of the replicates
- **MDL:** using the appropriate Student t value for the number of replicates, enter the calculated MDL
- **Relinquished by: (SIG):** enter the signature of the person relinquishing custody of the samples
- **Representing:** enter the company name or affiliation employing the person relinquishing/receiving custody
- **Received by: (SIG):** enter the signature of the person receiving custody of the samples
- **Date:** enter the date in the format M/D/YY (e.g., 6/3/04) when the samples were relinquished/received
- **Time:** enter the time in 24-hour format (e.g., 0900) when the samples were relinquished/received



**AFCEE  
 INORGANIC ANALYSES DATA SHEET 1  
 DATA PACKAGE**

**Analytical Method:** \_\_\_\_\_

**AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_

**Contract #:** \_\_\_\_\_

**Base/Command:** \_\_\_\_\_

**Prime Contractor:** \_\_\_\_\_

**Field Sample ID**

**Lab Sample ID**

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**Comments:**

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 \_\_\_\_\_

I certify this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

**Signature:** \_\_\_\_\_

**Name:** \_\_\_\_\_

**Date:** \_\_\_\_\_

**Title:** \_\_\_\_\_

AFCEE FORM I-1



**AFCEE  
 INORGANIC ANALYSES DATA SHEET 2  
 RESULTS**

**Analytical Method:** \_\_\_\_\_ **Preparatory Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**Field Sample ID:** \_\_\_\_\_ **Lab Sample ID:** \_\_\_\_\_ **Matrix:** \_\_\_\_\_

**% Solids:** \_\_\_\_\_ **Initial Calibration ID:** \_\_\_\_\_

**Date Received:** \_\_\_\_\_ **Date Prepared:** \_\_\_\_\_ **Date Analyzed:** \_\_\_\_\_

**Concentration Units (mg/L or mg/kg dry weight):** \_\_\_\_\_

Analyte	MDL	RL	Concentration	Dilution	Qualifier

**Comments:**  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

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**AFCEE  
 INORGANIC ANALYSES DATA SHEET 3  
 INITIAL MULTIPOINT CALIBRATION**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**Date of Initial Calibration:** \_\_\_\_\_ **Initial Calibration ID:** \_\_\_\_\_

**Instrument ID:** \_\_\_\_\_ **Concentration Units:** \_\_\_\_\_

Analyte	Std 1	Std 2	Std 3	Std 4	Std 5	Std 6	r	Q

r = correlation coefficient

**Comments:**  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE**  
**INORGANIC ANALYSES DATA SHEET 3A**  
**MERCURY INITIAL MULTIPOINT CALIBRATION**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_  
**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_  
**Instrument ID:** \_\_\_\_\_ **Date of Initial Calibration:** \_\_\_\_\_  
**Initial Calibration ID:** \_\_\_\_\_ **Concentration Units (mg/L or mg/kg):** \_\_\_\_\_

Analyte	Std 1	Std 2	Std 3	Std 4	Std 5	r	Q
Mercury							

r = correlation coefficient

**Comments:**

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**AFCEE**  
**INORGANIC ANALYSES DATA SHEET 3B**  
**CYANIDE INITIAL MULTIPOINT CALIBRATION**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_  
**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_  
**Instrument ID:** \_\_\_\_\_ **Date of Initial Calibration:** \_\_\_\_\_  
**Initial Calibration ID:** \_\_\_\_\_ **Concentration Units (mg/L or mg/kg):** \_\_\_\_\_

Analyte	Std 1	Std 2	Std 3	Std 4	Std 5	Std 6	r	Q
Cyanide								

r = correlation coefficient

	Expected	Found	%D	Q
High Distilled Standard				
Low Distilled Standard				

**Comments:**

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_



**AFCEE**  
**INORGANIC ANALYSES DATA SHEET 4**  
**CALIBRATION VERIFICATION**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_  
**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_  
**Instrument ID:** \_\_\_\_\_ **Initial Calibration ID:** \_\_\_\_\_  
**2nd Source ID:** \_\_\_\_\_ **ICV ID:** \_\_\_\_\_  
**CCV #1 ID:** \_\_\_\_\_ **CCV #2 ID:** \_\_\_\_\_  
**Concentration Units:** \_\_\_\_\_

Analyte	2nd Source Calibration Verification			Initial Calibration Verification			Continuing Calibration Verification					Q	
	Expected	Found	%D	Expected	Found	%D	Expected	Found 1	%D	Found 2	%D		

**Comments:** \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE  
INORGANIC ANALYSES DATA SHEET 5  
ICP-MASS SPECTROMETRY TUNE**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**ICP-Mass Spectrometry Instrument ID:** \_\_\_\_\_ **Date:** \_\_\_\_\_

**Time:** \_\_\_\_\_

Element -Mass	Avg. Measured Mass (amu)	Avg. Peak Width at 10% Peak Height (amu)	%RSD

**Comments:**  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_



**AFCEE  
 INORGANIC ANALYSES DATA SHEET 6  
 ICP-AES and ICP-MASS SPECTROMETRY SERIAL DILUTIONS**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_  
**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_  
**Lab Sample ID:** \_\_\_\_\_ **Date of Analysis:** \_\_\_\_\_  
**Concentration Units:** \_\_\_\_\_ **Matrix (soil/water):** \_\_\_\_\_

Analyte	Initial Sample Result		Serial Dilution Result (S)		% Difference	Q
		Qual		Qual		

**Comments:**  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE  
 INORGANIC ANALYSES DATA SHEET 7  
 POST-DIGESTION SPIKE SAMPLE RECOVERY**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**Lab Sample ID:** \_\_\_\_\_ **Date of Analysis:** \_\_\_\_\_

**Concentration Units:** \_\_\_\_\_ **Matrix (soil/water):** \_\_\_\_\_

Analyte	Control Limit %R	Spiked Sample Result		Sample Result		Spike Added	%R	Q
		Qual	Qual	Qual	Qual			

**Comments:**

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

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**AFCEE  
 INORGANIC ANALYSES DATA SHEET 8  
 BLANKS**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**Concentration Units:** \_\_\_\_\_

**Initial Calibration Blank ID:** \_\_\_\_\_ **Initial Calibration ID:** \_\_\_\_\_

**CCB #1 ID:** \_\_\_\_\_ **CCB #2 ID:** \_\_\_\_\_ **CCB #3 ID:** \_\_\_\_\_

**Method Blank ID:** \_\_\_\_\_

Analyte	Initial Calibration Blank	Continuing Calibration Blank			Method Blank	RL	Q
		1	2	3			

**Comments:**

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_



AFCEE  
INORGANIC ANALYSES DATA SHEET 9  
LABORATORY CONTROL SAMPLE

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

LCS ID: \_\_\_\_\_ Date of Analysis: \_\_\_\_\_

Concentration Units: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_

Analyte	Expected	Found	%R	CLs	Q

Comments:  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_



**AFCEE  
 INORGANIC ANALYSES DATA SHEET 10  
 MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLE RECOVERY**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**Concentration Units:** \_\_\_\_\_ **% Solids:** \_\_\_\_\_

**Parent Field Sample ID:** \_\_\_\_\_ **MS ID:** \_\_\_\_\_ **MSD ID:** \_\_\_\_\_

**Date of Analysis:** \_\_\_\_\_

Analyte	Parent Sample Result	Spike Added	Spiked Sample Result	%R	Duplicate Spiked Sample Result	%R	%RPD	CLs %R	CLs %RPD	Q

**Comments:** \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



AFCEE
INORGANIC ANALYSES DATA SHEET 11
HOLDING TIMES

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Table with 7 columns: Field Sample ID, Date Collected, Date Received, Date Analyzed, Max. Holding Time (days), Time Held (days), Q. The table contains 15 empty rows for data entry.

Comments:

Three horizontal lines for entering comments.



**AFCEE  
INORGANIC ANALYSES DATA SHEET 12  
INSTRUMENT ANALYSIS SEQUENCE LOG**

**Analytical Method:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**Instrument ID #:** \_\_\_\_\_

Field Sample ID/Std ID/ Blank ID/QC Sample ID	Date Analysis Started	Time Analysis Started	Date Analysis Completed	Time Analysis Completed

Comments:  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_



**AFCEE  
ORGANIC ANALYSES DATA PACKAGE**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
Base/Command: \_\_\_\_\_ Prime Contractor: \_\_\_\_\_

Field Sample ID	Lab Sample ID
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

I certify this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: \_\_\_\_\_ Name: \_\_\_\_\_  
Date: \_\_\_\_\_ Title: \_\_\_\_\_



**AFCEE  
ORGANIC ANALYSES DATA SHEET 2  
RESULTS**

Analytical Method: \_\_\_\_\_ Preparatory Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Field Sample ID: \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_ Matrix: \_\_\_\_\_

% Solids: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_

Date Received: \_\_\_\_\_ Date Prepared: \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

Concentration Units (ug/L or mg/kg dry weight): \_\_\_\_\_

Analyte	MDL	RL	Concentration	Dilution	Confirm	Qualifier

Surrogate	Recovery	CLs	Qualifier

Internal Std	Qualifier

Comments:  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

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**AFCEE  
ORGANIC ANALYSES DATA SHEET 3A  
INITIAL MULTIPOINT CALIBRATION-GC/MASS SPECTROMETRY ANALYSIS**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
 Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Date of Initial Calibration: \_\_\_\_\_  
 Initial Calibration ID: \_\_\_\_\_ Concentration Units (ug/L or mg/kg): \_\_\_\_\_

Analyte	Std 1	RF 1	Std 2	RF 2	Std 3	RF 3	Std 4	RF 4	Std 5	RF 5	Std 6	RF 6	Std 7	RF 7

\* SPCCs # CCCs

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE  
ORGANIC ANALYSES DATA SHEET 3A  
INITIAL MULTIPOINT CALIBRATION-GC/MASS SPECTROMETRY ANALYSIS**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
Instrument ID: \_\_\_\_\_ Date of Initial Calibration: \_\_\_\_\_  
Initial Calibration ID: \_\_\_\_\_ Concentration Units (ug/L or mg/kg): \_\_\_\_\_

Analyte	% RSD	mean %RSD	r	COD	Q

\* SPCCs # CCCs

Comments:  
\_\_\_\_\_  
\_\_\_\_\_



**AFCEE**  
**ORGANIC ANALYSES DATA SHEET 3**  
**INITIAL MULTIPOINT CALIBRATION-GC/MASS SPECTROMETRY ANALYSIS**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
 Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Date of Initial Calibration: \_\_\_\_\_  
 Initial Calibration ID: \_\_\_\_\_ Concentration Units (ug/L or mg/kg): \_\_\_\_\_

Analyte	Std 1	RF 1	Std 2	RF 2	Std 3	RF 3	Std 4	RF 4	Std 5	RF 5	Std 6	RF 6	Std 7	RF 7

Comments:  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE**  
**ORGANIC ANALYSES DATA SHEET 3**  
**INITIAL MULTIPPOINT CALIBRATION-GC/MASS SPECTROMETRY ANALYSIS**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
 Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Date of Initial Calibration: \_\_\_\_\_  
 Initial Calibration ID: \_\_\_\_\_ Concentration Units (ug/L or mg/kg): \_\_\_\_\_

Analyte	% RSD	mean %RSD	r	COD	Q

Comments:

---



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**AFCEE  
 ORGANIC ANALYSES DATA SHEET 4  
 SECOND SOURCE CALIBRATION VERIFICATION**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
 Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_  
 2nd Source ID: \_\_\_\_\_ Concentration Units (ug/L or mg/kg): \_\_\_\_\_

Analyte	Expected	Found	%D	Q

Comments:  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE  
ORGANIC ANALYSES DATA SHEET 5A  
CALIBRATION VERIFICATION-GC/MASS SPECTROMETRY ANALYSIS**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
 Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_  
 ICV ID: \_\_\_\_\_ CCV #1 ID: \_\_\_\_\_ CCV #2 ID: \_\_\_\_\_

Analyte	ICV		CCV #1		CCV #2		Q
	RF	% D	RF	% D	RF	% D	
Chloromethane *							
1,1-DCA *							
Bromoform *							
Chlorobenzene *							
1,1,2,2-TCA *							
1,1-DCE #							
Chloroform #							
1,2-DCP #							
Toluene #							
Ethylbenzene #							
Vinyl chloride #							

\* SPCCs # CCCs

Comments:  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE  
 ORGANIC ANALYSES DATA SHEET 5  
 CALIBRATION VERIFICATION**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
 Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_  
 ICV ID: \_\_\_\_\_ CCV #1 ID: \_\_\_\_\_ CCV #2 ID: \_\_\_\_\_

Analyte	ICV %D or % drift	CCV#1 %D or % drift	CCV#2 %D or % drift	Q

Comments:  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_





**AFCEE**  
**ORGANIC ANALYSES DATA SHEET 6**  
**BLANK**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Concentration Units (ug/L or mg/kg): \_\_\_\_\_ Method Blank ID: \_\_\_\_\_

Initial Calibration ID: \_\_\_\_\_

Analyte	Method Blank	RL	Q

Surrogate	Recovery	CLs	Qualifier

Internal Std	Qualifier

Comments:

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**AFCEE  
 ORGANIC ANALYSES DATA SHEET 7  
 LABORATORY CONTROL SAMPLE**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

LCS ID: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_

Concentration Units (ug/L or mg/kg): \_\_\_\_\_

Analyte	Expected	Found	%R	CLs	Q

Surrogate	Recovery	CLs	Qualifier

Internal Std	Qualifier

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

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**AFCEE**  
**ORGANIC ANALYSES DATA SHEET 8**  
**MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLE RECOVERY**

Analytical Method: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Concentration Units (ug/L or mg/kg): \_\_\_\_\_ % Solids: \_\_\_\_\_

Parent Field Sample ID: \_\_\_\_\_ MS ID: \_\_\_\_\_ MSD ID: \_\_\_\_\_

Analyte	Parent Sample Result	Spike Added	Spiked Sample Result	% R	Duplicate Spiked Sample Result	% R	% RPD	CLs %R	CLs %RPD	Q

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_



**AFCEE  
 ORGANIC ANALYSES DATA SHEET 9  
 HOLDING TIMES**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Field Sample ID	Date Collected	Date Received	Date Extracted	Max. Holding Time E	Time Held Ext.	Date Analyzed	Max. Holding Time A	Time Held Anal.	Q

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE**  
**ORGANIC ANALYSES DATA SHEET 10**  
**INSTRUMENT ANALYSIS SEQUENCE LOG**

Analytical Method: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Instrument ID #: \_\_\_\_\_

Field Sample ID/Std ID/ Blank ID/QC Sample ID	Date Analysis Started	Time Analysis Started	Date Analysis Completed	Time Analysis Completed

Comments:

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**AFCEE  
ORGANIC ANALYSES DATA SHEET 11  
INSTRUMENT PERFORMANCE CHECK  
(BFB or DFTPP)**

Analytical Method: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

Instrument ID: \_\_\_\_\_ Compound: \_\_\_\_\_ Injection Date/Time: \_\_\_\_\_

Initial Calibration ID: \_\_\_\_\_

Mass	Ion Abundance Criteria	% Relative Abundance	Q



**AFCEE  
WET CHEM ANALYSES DATA SHEET 3  
INITIAL MULTIPOINT CALIBRATION**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_  
**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_  
**Instrument ID:** \_\_\_\_\_ **Date of Initial Calibration:** \_\_\_\_\_  
**Initial Calibration ID:** \_\_\_\_\_ **Concentration Units (mg/L or mg/kg):** \_\_\_\_\_

Analyte	Std 1	Std 2	Std 3	Std 4	Std 5	r	Q

r = correlation coefficient

**Comments:** \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_





AFCEE  
WET CHEM ANALYSES DATA SHEET 4  
CALIBRATION VERIFICATION

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_  
Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_  
Instrument ID: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_  
2nd Source ID: \_\_\_\_\_ CCV #1 ID: \_\_\_\_\_ CCV #2 ID: \_\_\_\_\_

Analyte	2nd Source Calibration Verification			Continuing Calibration Verification					Q
	Expected	Found	%D	Expected	Found 1	%D	Found 2	%D	

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_



**AFCEE  
 WET CHEM ANALYSES DATA SHEET 5  
 BLANKS**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**Concentration Units (mg/L or mg/kg):** \_\_\_\_\_

**Calibration Blank ID:** \_\_\_\_\_ **Initial Calibration ID:** \_\_\_\_\_

**Method Blank ID:** \_\_\_\_\_ **Initial Calibration ID:** \_\_\_\_\_

Analyte	Calibration Blank	Method Blank	RL	Q

**Comments:** \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE**  
**WET CHEM ANALYSES DATA SHEET 6**  
**LABORATORY CONTROL SAMPLE**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**LCS ID:** \_\_\_\_\_ **Initial Calibration ID:** \_\_\_\_\_

**Concentration Units (mg/L or mg/kg):** \_\_\_\_\_

Analyte	Expected	Found	%R	CLs	Q

**Comments:** \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE**  
**WET CHEM ANALYSES DATA SHEET 7**  
**MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLE RECOVERY**

Analytical Method: \_\_\_\_\_ AAB #: \_\_\_\_\_

Lab Name: \_\_\_\_\_ Contract #: \_\_\_\_\_

% Solids: \_\_\_\_\_ Initial Calibration ID: \_\_\_\_\_

Parent Field Sample ID: \_\_\_\_\_ MS ID: \_\_\_\_\_ MSD ID: \_\_\_\_\_

Concentration Units (mg/L or mg/kg): \_\_\_\_\_

Analyte	Parent Sample Result	Spike Added	Spiked Sample Result	%R	Duplicate Spiked Sample Result	%R	%RPD	CLs %R	CLs %RPD	Q

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_



**AFCEE  
WET CHEM ANALYSES DATA SHEET 8  
HOLDING TIMES**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

Field Sample ID	Date Collected	Date Received	Date Analyzed	Max. Holding Time (days)	Time Held (days)	Q

**Comments:** \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_



**AFCEE  
 WET CHEM ANALYSES DATA SHEET 9  
 INSTRUMENT ANALYSIS SEQUENCE LOG**

**Analytical Method:** \_\_\_\_\_ **AAB #:** \_\_\_\_\_

**Lab Name:** \_\_\_\_\_ **Contract #:** \_\_\_\_\_

**Instrument ID #:** \_\_\_\_\_

Field Sample ID/Std ID/ Blank ID/QC Sample ID	Date Analysis Started	Time Analysis Started	Date Analysis Completed	Time Analysis Completed

**Comments:** \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



**AFCEE  
 SCREENING DATA PACKAGE**

Analytical Method: \_\_\_\_\_

Contract #: \_\_\_\_\_

Base/Command: \_\_\_\_\_

Prime Contractor: \_\_\_\_\_

Field Sample ID

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments:

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Signature: \_\_\_\_\_

Name: \_\_\_\_\_

Date: \_\_\_\_\_

Title: \_\_\_\_\_



**AFCEE  
SCREENING DATA SHEET 2  
RESULTS**

Analytical Method: \_\_\_\_\_

Contract #: \_\_\_\_\_ Field Sample ID: \_\_\_\_\_

Matrix: \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

Concentration Units (ug/L, mg/kg dry weight or °C): \_\_\_\_\_

Analyte/Test	MDL	RL	Result	Qualifier

Comments:  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_





**AFCEE  
 SCREENING DATA SHEET 3  
 FIELD DUPLICATES**

Analytical Method: \_\_\_\_\_ Contract #: \_\_\_\_\_

Units: \_\_\_\_\_

Analyte/Test	Sample Result	Duplicate Sample Result	%D or %RPD	Acceptance Criteria	Q

Comments:  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



### MDL STUDY REPORT FORM

Lab Name: \_\_\_\_\_ Analytical Method: \_\_\_\_\_ Matrix: \_\_\_\_\_

Analysis Date: \_\_\_\_\_ Instrument ID: \_\_\_\_\_

Concentration Units (mg/L or mg/kg): \_\_\_\_\_

Analyte	Amt. Spiked	Replicate							Std. Dev.	MDL
		1	2	3	4	5	6	7		

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## **9.0 SYSTEMS AND PERFORMANCE AUDITS, PERFORMANCE EVALUATION PROGRAMS, MAGNETIC TAPE AUDITS, AND TRAINING**

Technical systems and performance audits shall be performed as independent assessments of sample collection and analysis procedures. Audit results will be used to evaluate the ability of an analytical contractor to (1) produce data that fulfill the objectives established for the program, (2) comply with the QC criteria, and (3) identify any areas requiring corrective action. The systems audit is a qualitative review of the overall sampling or measurement system, while the performance audit is a quantitative assessment of a measurement system. Audit guidance can be found in the *HQ AFCEE Technical Services Quality Assurance Program*, current version. Full data review/validation is also a quantitative check of the analytical process, where all documentation and calculations are evaluated and verified. Data review/validation procedures are discussed in Section 8.

### **9.1 PROJECT AUDITS**

#### **9.1.1 State/Federal Project Audits**

Audits by various state and federal agencies are commonly conducted for the laboratories that will analyze project samples. Audit reports from these agencies should be reviewed by the prime contractor to determine whether data produced by the subcontractor analytical laboratory can fulfill the objectives of the program. All laboratories shall participate in the USEPA Water Supply and Water Pollution Studies programs or equivalent programs for state certifications. Satisfactory performance in these nonproject-specific PT sample programs also demonstrates proficiency in methods used to analyze AFCEE samples. The laboratory shall document the corrective actions to unacceptable PT sample results to demonstrate resolution of the problems. Audit findings shall be transmitted from the laboratory to the prime contractor and to AFCEE. The prime contractor shall review the audit findings and provide a written report to AFCEE. This report shall include the recommended corrective actions or procedures to remedy any deficiencies identified during the state/federal audit(s). The audit results and discussion shall be incorporated into the QA report for each sampling effort.

#### **9.1.2 Technical Systems Audits**

A technical systems audit is an on-site, qualitative review of the sampling or analytical system to ensure that the activities are being performed in compliance with the project *SAP* specifications. Sampling, field procedures, and analytical laboratories shall be audited by the prime contractor at the beginning of the project. In addition, a laboratory systems audit may be performed by AFCEE if previous audit reports indicate corrective actions have not been completed, a recent audit has not been conducted, or quality concerns have arisen based upon the use of that laboratory for other projects. The laboratory systems audit results will be used to assess the prime contractor's oversight and to review laboratory operation and ensure the technical procedures and documentation are in place and operating to provide data that fulfill the project objectives and to ensure corrective actions have been implemented.



Critical items for a laboratory systems audit include: (1) sample custody procedures, (2) calibration procedures and documentation, (3) completeness of data forms, notebooks, and other reporting requirements, (4) data review and validation procedures, (5) data storage, filing, and record keeping procedures, (6) QC procedures, tolerances, and documentation, (7) operating conditions of facilities and equipment, (8) documentation of training and maintenance activities, (9) systems and operations overview, and (10) security of laboratory automated systems.

Critical items for field sampling systems audit include: (1) appropriate sampling plans (*QAPP, FSP*) (2) calibration procedures and documentation for field equipment, (3) documentation in field logbooks and sampling data sheets, (4) organization and minimization of potential contamination sources while in the field, (5) proper sample collection, storage, and transportation procedures, and (6) compliance with established CoC and transfer procedures.

After each on-site audit, a debriefing session will be held for all participants to discuss the preliminary audit results. The auditor will then complete the audit evaluation and submit an audit report including observations of the deficiencies and the necessary recommendations for corrective actions (RCAs) to the prime contractor. Compliance with the specifications presented in the *SAP* will be noted and noncompliance or deviations shall be addressed in writing by the prime contractor to AFCEE with corrective actions and a time frame for implementation of the corrective actions. Follow-up audits will be performed prior to completion of the project to ensure corrective actions have been taken.

AFCEE personnel must be notified at least three weeks prior to conducting the field audit. Also, if AFCEE personnel plan to observe field activities during the audit, the prime contractor must provide the AFCEE attendee(s) with any needed personal protective equipment. This should be coordinated directly with AFCEE attendee(s).

### **9.1.3 Project-Specific Performance Evaluation Audits**

Performance audits quantitatively assess the data produced by a measurement system. A performance audit involves submitting project-specific PT samples for analysis for each analytical method used in the project. The prime contractor shall submit project-specific PT samples once per quarter per project. The project-specific PT samples are selected to reflect the expected range of concentrations for the sampling program. The performance audit answers questions about whether the measurement system is operating within CLs and whether the data produced meet the analytical QA specifications.

The project-specific PT samples are made to look as similar to field samples as possible and are submitted as part of a field sample shipment so that the laboratory is unable to distinguish between them and project samples. This approach ensures unbiased sample analysis and reporting by the laboratory.

The critical elements for review of PT sample results include: (1) correct identification and quantitation of the PT sample analytes, (2) accurate and complete reporting of the results, and (3) measurement system operation within established CLs for precision and accuracy.



The concentrations reported for the PT samples shall be compared to the known or expected concentrations spiked in the samples. The percent recovery shall be calculated and the results assessed according to the accuracy criteria for the LCS presented in Section 7 and/or the values from the PT sample provider. If the accuracy criteria are not met, the cause of the discrepancy shall be investigated and a second PT sample shall be submitted. The prime contractor shall notify the project staff, AFCEE, and agencies of the situation at the earliest possible time, and the prime contractor shall keep AFCEE informed of corrective actions and subsequent PT sample results.

#### **9.1.4 Magnetic Tape Audits**

Magnetic tape audits involve the examination of the electronic media used by the analytical laboratory and by the prime contractor to collect, analyze, report, and store data. These audits are used to assess the authenticity of the data generated and the implementation of good automated laboratory practices. AFCEE may perform magnetic tape audits of the laboratories or of the prime contractors when warranted by project PT sample results, on-site audit results, or by other state/federal investigations.

## **9.2 TRAINING**

Training shall be provided to all project personnel to ensure compliance with the health and safety plan and technical competence in performing the work effort. Documentation of this training shall be maintained in the records of the contracted organizations.



## **10.0 PREVENTATIVE MAINTENANCE**

A preventive maintenance program shall be in place to promote the timely and effective completion of a measurement effort. The preventive maintenance program is designed to minimize the downtime of crucial sampling and/or analytical equipment due to unexpected component failure. In implementing this program, efforts are focused in three primary areas: (1) establishment of maintenance responsibilities, (2) establishment of maintenance schedules for major and/or critical instrumentation and apparatus, and (3) establishment of an adequate inventory of critical spare parts and equipment.

### **10.1 MAINTENANCE RESPONSIBILITIES**

Maintenance responsibilities for equipment and instruments are assumed by the respective facility managers. The managers then establish maintenance procedures and schedules for each major equipment item. This responsibility may be delegated to laboratory personnel, although the managers retain responsibility for ensuring adherence to the prescribed protocols.

### **10.2 MAINTENANCE SCHEDULES**

The effectiveness of any maintenance program depends to a large extent on adherence to specific maintenance schedules for each major equipment item. Other maintenance activities are conducted as needed. Manufacturers' recommendations provide the primary basis for the established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g., GC/mass spectrometry instruments, AA spectrometers, and analytical balances).

### **10.3 SPARE PARTS**

Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment downtime. The inventory includes those parts (and supplies) that are subject to frequent failure, have limited useful lifetimes, or cannot be obtained in a timely manner should failure occur.

Field sampling task leaders and the respective laboratory managers are responsible for maintaining an adequate inventory of spare parts. In addition to spare parts and supply inventories, the contractor shall maintain an in-house source of backup equipment and instrumentation.

### **10.4 MAINTENANCE RECORDS**

Maintenance and repair of major field and laboratory equipment shall be recorded in field or laboratory logbooks. These records shall document the serial numbers of the equipment, the person performing the maintenance or repairs, the date of the repair, the procedures used during the repair, and proof of successful repair prior to the use of the equipment.



## **11.0 CORRECTIVE ACTION**

Corrective actions, if necessary, shall be completed once. If acceptance criteria were not met and a corrective action was not successful or corrective action was not performed, apply the appropriate flagging criteria. Requirements and procedures for documenting the need for corrective actions are described in this section.

### **11.1 CORRECTIVE ACTION REPORT**

Problems requiring corrective action in the laboratory shall be documented by the use of a corrective action report. The QA coordinator or any other laboratory member can initiate the corrective action request in the event QC results exceed acceptability limits, or upon identification of some other laboratory problem. Corrective actions can include reanalysis of the sample or samples affected, resampling and analysis, or a change in procedures, depending upon the severity of the problem.

### **11.2 CORRECTIVE ACTION SYSTEM**

A system for issuing, tracking, and documenting completion of formal RCA exists for addressing significant and systematic problems. RCAs are issued only by a member of the QA group or a designee in a specific QA role. Each RCA addresses a specific problem or deficiency, usually identified during QA audits of laboratory or project operations. An RCA requires a written response from the party to whom the RCA was issued. A summary of unresolved RCAs is included in the monthly QA report to management. The report lists all RCAs that have been issued, the manager responsible for the work area, and the current status of each RCA. An RCA requires verification by the QA group that the corrective action has been implemented before the RCA is considered to be resolved. In the event there is no response to an RCA within 30 days, or if the proposed corrective action is disputed, the recommendation and/or conflict is pursued to successively higher management levels until the issue is resolved.



## **12.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT**

At a minimum, the QA coordinator of the laboratory shall prepare a summary report quarterly of the status of the project, of QA/QC problems, corrective actions taken, and unresolved RCAs with recommended solutions for management. The report shall also include results from all PE samples, audit findings, and periodic data quality assessments. This report shall be available for review by AFCEE auditors upon request.





## 13.0 REFERENCES

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