QUALITY ASSURANCE PROJECT PLAN

Prepared for:

Areas of Concern (AOC)
Long Term Monitoring Baseline Study
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PREFACE

This document is the *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP), version 3.0.* This detailed QAPP, (1) has been prepared for use by contractors who perform environmental services to ensure the data are scientifically valid and defensible, and (2) establishes the analytical protocols and documentation requirements to ensure the data are collected, reviewed, and analyzed in a consistent manner. This QAPP and a site specific Field Sampling Plan (FSP) shall constitute, by definition, an AFCEE Sampling and Analysis Plan (SAP). All prime contractors and laboratories performing work in support of AFCEE contracts shall perform their services in accordance with the requirements specified in this QAPP. A variance shall be requested for any exception to or deviation from the requirements in this QAPP. Variance requests are submitted as an addendum to the SAP. Variances from the QAPP shall be identified by chapter, subtitle, paragraph, page, and line with supporting justification for the change. The original text in this QAPP is crossed out and a reference to the appropriate variance request by number in the addendum is added to the QAPP. If any additional analytical methods are required in the SAP that are not in this QAPP, the analytical methods must be included in the addendum to the SAP with all the accompanying quality control requirements, i.e., reporting limits, calibration requirements, quality control measures, corrective action, data validation, and reporting requirements, comparable in format to the analytical tables in Sections 6, 7, and 8. Variances must be approved by the AFCEE Team Chief for the project. Only the variances approved by the AFCEE Team Chief shall be included in the final version of the SAP. A copy of the initial submission and final approved variances can be found in Appendix A of this QAPP.

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LIST OF ACRONYMS AND ABBREVIATIONS

AA	atomic absorption
AFCEE	Air Force Center for Environmental Excellence
AFIID	Air Force installation identification
A2LA	American Association for Laboratory Accreditation
ARAR	applicable or relevant and appropriate requirement
ASCII	American Standard Code Information Interchange
ASTM	American Society for Testing and Materials
BFB	bromofluorobenzene
Br^-	bromide
BTEX	benzene, toluene, ethylbenzene, xylene
°C	degrees Celsius
CCC	calibration check compound
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CF	calibration factor
CFR	Code of Federal Regulation
Cl^-	chloride
CL	control limit
CLP	Contract Laboratory Program
COC	chain of custody
2,4-D	2,4-dichlorophenoxy propanoic acid
2,4-DB	2,4-dichlorophenoxy butyric acid
DCA	dichloroethane
DCB	dichlorobenzene
DCBP	decachlorobiphenyl
DCE	dichloroethene
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethene
DDT	dichlorodiphenyltrichloroethane
DEQPPM	Defense Environmental Quality Program Policy Memorandum
DFTPP	decafluorotriphenylphosphine
DNB	dinitrobenzene

DNT	dinitrotoluene
DOD	Department of Defense
DQO	data quality objective
DRO	diesel range organics
EDB	ethylene dibromide
EICP	extracted ion current profile
EPA	Environmental Protection Agency
ERPIMS	Environmental Resources Program Information Management System
F^-	fluoride
FID	flame ionization detector
FLAA	flame atomic absorption
FS	feasibility study
FSP	field sampling plan
g	gram
G	glass
GC	gas chromatography
GC/MS	gas chromatography/mass spectroscopy
GFAA	graphite furnace atomic absorption
GRO	gasoline range organics
Handbook	*Handbook for the Installation Restoration Program (IRP) Remedial Investigation and Feasibility Studies (RI/FS)*, September 1993
HCl	hydrochloric acid
HECD	(Hall) electrolytic conductivity detector
HpCDD	heptachlorodibenzo-p-dioxin
HpCDF	heptaclorordibenzofuran
HxCDD	hexachlorodibenzo-p-dioxin
HxCDF	hexachlorodibenzofuran
HMX	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HNO_3	nitric acid
HPLC	high-performance liquid chromatography
H_2SO_4	sulfuric acid
IAW	in accordance with
ICP	inductively coupled plasma
ICPES	inductively coupled plasma emission spectroscopy

ICP-MS	inductively coupled plasma - mass spectroscopy
ICS	interference check standard
ID	identification
IRP	Installation Restoration Program
IS	internal standard
LCL	lower control limit
LCS	laboratory control sample
MCPA	(4-chloro-2-methylphenoxy) acetic acid
MCPP	2-(4-chloro-2-methylphenoxy) propionic acid
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliter
mm	millimeter
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable
$Na_2S_2O_3$	sodium thiosulfate
NCP	National Contingency Plan
ng/L	nanograms per liter
ng/mL	nanograms per milliliter
NIST	National Institute of Standards and Technology
nm	nanometer
NO_2^-	nitrite
NO_3^-	nitrate
NTU	nephelometric turbidity unit
OCDD	octachlorodibenzo-p-dioxin
ORP	oxidation-reduction potential
OVA	organic vapor analyzer
P	polyethylene
PAH	polynuclear aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo-p-dioxin
PCDF	polychlorinated dibenzofuran

PE	performance evaluation
PeCDD	pentachlorodibenzo-p-dioxin
PeCDF	pentachlorodibenzofuran
PID	photoionization detector
PO_4^{-3}	phosphate
ppb	parts per billion
ppm	parts per million
ppmv	parts per million volume
PQL	practical quantitation limit
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
R	recovery
RCA	recommendations for corrective action
RCRA	Resource Conservation and Recovery Act
RDX	hexahydro-1,3,5-trinitro-1,3,5-triazine
RF	response factor
RI	remedial investigation
RI/FS	remedial investigation/feasibility study
RPD	relative percent difference
RSD	relative standard deviation
S	soil
SAP	sampling and analysis plan
SARA	Superfund Amendments and Reauthorization Act
SO_4^{-2}	sulfate
SOP	standard operating procedure
SOW	statement of work
SPCC	system performance check compound
SVOC	semivolatile organic compound
2,4,5-T	2,4,5-trichlorophenoxy acetic acid
T	California brass
TCA	trichloroethane
TCDD	tetrachlorodibenzo-p-dioxin
TCDF	tetrachlorodibenzofuran
TCE	trichloroethene

TCLP	toxicity characteristic leaching procedure
TCMX	tetrachlorometaxylene
TIC	tentatively identified compound
TNB	trinitrobenzene
TNT	trinitrotoluene
2,4,5-TP	2,4,5-trichlorophenoxy propanoic acid (silvex)
TPH	total petroleum hydrocarbon
UCL	upper control limit
VOC	volatile organic compound
v/v	volume to volume
W	water

SYMBOLS

µg/kg	micrograms per kilogram
µg/L	micrograms per liter
µg/mL	micrograms per milliliter
µL	microliter
µm	micrometer

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1.0 INTRODUCTION

The 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 Sampling and Analysis Plan (SAP) for the project. This detailed QAPP, (1) has been prepared for use by contractors who perform environmental services to ensure the data are scientifically valid and defensible, and (2) establishes the analytical protocols and documentation requirements to ensure the data are collected, reviewed, and analyzed in a consistent manner. This QAPP and a site specific Field Sampling Plan (FSP) shall constitute, by definition, an AFCEE Sampling and Analysis Plan (SAP).

The National Contingency Plan (NCP) specifies circumstances under which a QAPP is necessary for Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) response actions. For cleanup actions at the remedial investigation/feasibility study (RI/FS) stage, the NCP requires lead agents to develop sampling and analysis plans which provide a process for obtaining data of sufficient quality and quantity to satisfy data needs. Such sampling and analysis plans must include a quality assurance project plan "which describes policy, organization, and functional activities and the data quality objectives and measures necessary to achieve adequate data for use in selecting the appropriate remedy." 40 CFR 300.430 (b)(8)(ii).

The U.S. Environmental Protection Agency (EPA) QA policy requires a QAPP for every monitoring and measurement project mandated or supported by the EPA through regulations, contracts, or other formalized means not currently covered by regulation. Guidelines followed in the preparation of this plan are set out in *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans* (U.S. EPA, 1983a) and *U.S. EPA Region IX QAPP: Guidance for Preparing QAPPs for Superfund Remedial Projects* (U.S. EPA, 1989). Other documents that have been referenced for this plan include *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final* (U.S. EPA, 1988); *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, Draft Final, EPA QA/R-5* (U.S. EPA, 1993), *Compendium of Superfund Field Operations Methods* (U.S. EPA, 1987a); *Data Quality Objectives Process for Superfund, Interim Final Guidance* (U.S. EPA, 1993); *U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (U.S. EPA, 1994), *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (U.S. EPA, 1994), *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (U.S. EPA SW-846, Third Edition and its first, second and third update), and the *Handbook for Installation Restoration Program (IRP) Remedial Investigations and Feasibility Studies (RI/FS)* (Handbook), September 1993.

This QAPP is required reading for all staff participating in the work effort. The QAPP shall be in the possession of the field teams and in the laboratories performing all analytical methods. All contractors and subcontractors shall be required to comply with the procedures documented in this QAPP in order to maintain comparability and representativeness of the data produced.

Controlled distribution of the 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 the 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 the QAPP, a document control system shall be put into place to assure (1) all parties holding a controlled copy of the 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 the 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 THE U.S. AIR FORCE INSTALLATION RESTORATION PROGRAM

The objective of the U.S. Air Force Installation Restoration Project (IRP) is to assess past hazardous waste disposal and spill sites at U.S. Air Force installations and to develop remedial actions consistent with the NCP for sites that pose a threat to human health and welfare or the environment. This section presents information on the program origins, objectives, and organization.

The 1976 Resource Conservation Recovery Act (RCRA) is one of the primary federal laws governing the disposal of hazardous wastes. Sections 6001 and 6003 of RCRA require federal agencies to comply with local and state environmental regulations and provide information to the EPA concerning past disposal practices at federal sites. RCRA Section 3012 requires state agencies to inventory past hazardous waste disposal sites and provide information to the EPA concerning those sites.

In 1980, Congress enacted CERCLA (Superfund). CERCLA outlines the responsibility for identifying and remediating contaminated sites in the United States and its possessions. The CERCLA legislation identifies the EPA as the primary policy and enforcement agency regarding contaminated sites.

The 1986 Superfund Amendments and Reauthorization Act (SARA) extends the requirements of CERCLA and modifies CERCLA with respect to goals for remediation and the steps that lead to the selection of a remedial process. Under SARA, technologies that provide permanent removal or destruction of a contaminant are preferable to action that only contains or isolates the contaminant. SARA also provides for greater interaction with public and state agencies and extends the EPA's role in evaluating health risks associated with contamination. Under SARA, early determination of Applicable or Relevant and Appropriate Requirements (ARARs) is required, and the consideration of potential remediation alternatives is recommended at the initiation of an RI/FS. SARA is the primary legislation governing remedial action at past hazardous waste disposal sites.

Executive Order 12580, adopted in 1987, gave various federal agencies, including the Department of Defense (DOD), the responsibility to act as lead agencies for conducting investigations and implementing remediation efforts when they are the sole or co-contributor to contamination on or off their properties.

To ensure compliance with CERCLA, its regulations, and Executive Order 12580, the DOD developed the IRP, under the Defense Environmental Restoration Program, to identify potentially contaminated sites, investigate these sites, and evaluate and select remedial actions

for potentially contaminated facilities. The DOD issued the Defense Environmental Quality Program Policy Memorandum (DEQPPM) 80-6 regarding the IRP program in June 1980, and implemented the policies outlined in this memorandum in December 1980. The NCP was issued by EPA in 1980 to provide guidance on a process by which (1) contaminant release could be reported, (2) contamination could be identified and quantified, and (3) remedial actions could be selected. The NCP describes the responsibility of federal and state governments and those responsible for contaminant releases.

The DOD formally revised and expanded the existing IRP directives and amplified all previous directives and memoranda concerning the IRP through DEQPPM 81-5, dated 11 December 1981. The memorandum was implemented by a U.S. Air Force message dated 21 January 1982.

The IRP is the DOD's primary mechanism for response actions on U.S. Air Force installations affected by the provisions of SARA. In November 1986, in response to SARA and other EPA interim guidance, the U.S. Air Force modified the IRP to provide for an RI/FS program. The IRP was modified so that RI/FS studies could be conducted as parallel activities rather than serial activities. The program now includes ARAR determinations, identification and screening of technologies, and development of alternatives. The IRP may include multiple field activities and pilot studies prior to a detailed final analysis of alternatives. Over the years, requirements of the IRP have been developed and modified to ensure that DOD compliance with federal laws, such as RCRA, NCP, CERCLA, and SARA, can be met.

2.2 PURPOSE AND SCOPE

The purpose, scope, and use of this work effort is discussed in Section 1.2.2 and 1.2.3 of the Workplan.

2.3 PROJECT BACKGROUND

A project background description, including (1) the locations of sites at the base or facility, (2) a summary of the contamination history at each site and (3) the findings from previous investigations is included in Section 2 and 3 of the Workplan.

2.4 PROJECT SCOPE AND OBJECTIVES

A summary of the objectives and the proposed work for each site shall be included in Section 3.1, Section 3.2 and Section 3.3 of the FSP. The intended use of the data acquired during this project, the data quality objective process and a discussion of how the process specific decision rules were derived is described in Section 3.1 of the FSP.

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 is included in Section 4.0 of the FSP.

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

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4.0 QUALITY PROGRAM AND DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) specify the data type, quality, quantity, and uses needed to make decisions and are the basis for designing data collection activities. The DQOs for the project are specified in the FSP in Section 3.1.

4.1 DATA CATEGORIES

The two general categories of data used by the Air Force Center for Environmental Excellence (AFCEE) are defined as: (1) screening data and (2) definitive data.

Screening data are generated by rapid methods of analysis with less rigorous sample preparation, calibration and/or QC requirements than are necessary to produce definitive data. 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 and pH measurements, moisture content, turbidity, conductance, etc., have been designated by definition as screening methods (see Section 6).

Screening methods shall be confirmed, as required in Section 3.2 of the FSP, by analyses that generate definitive data. Confirmation samples shall be selected to include both detected and nondetected results from the screening method.

Definitive data are generated using rigorous analytical methods (see Section 7), such as approved EPA reference methods. The data can be generated in a mobile or off-site laboratory. Data are analyte-specific, and both identification and quantitation are confirmed. These methods have standardized QC and documentation requirements (Sections 7 and 8). Definitive data are not restricted in their use unless quality problems require data qualification.

4.2 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY

The basis for assessing each of these elements of data quality is discussed in the following subsections. Precision and accuracy QC limits for each method and matrix are identified in Sections 6 and 7.

4.2.1 Precision

Precision measures 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 conditions. *Analytical* precision is the measurement of the variability associated with duplicate (two) or replicate (more than two) analyses. AFCEE uses the laboratory control sample (LCS) to determine the precision of the analytical method. If the recoveries of analytes in the LCS are within established control limits, then precision is within limits. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch, rather the comparison is between the sample and samples analyzed in previous batches. *Total* precision is the measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate 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 analytical precision, and the precision measurement is determined 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. The formula for the calculation of RSD is provided in Table 4.2.1-1.

4.2.2 Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systemic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit. For volatile and semivolatile organic compounds, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed. Analysis of performance evaluation (PE) samples shall 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.

4.2.3 Representativeness

Objectives for representativeness are defined for each sampling and analysis task and are a function of the investigative objectives. Representativeness shall be achieved through use of the

standard field, sampling, and analytical procedures. Representativeness is also determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, and sampling locations. Decisions regarding sample/well/boring locations and numbers and the statistical sampling design are documented in Section 3.3 of the FSP.

4.2.4 Completeness

Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples. 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 (see Section 8 for an explanation of flagging criteria). The requirement for completeness is 95 percent for aqueous samples and 90 percent for soil samples. For any instances of samples that could not be analyzed for any reason (holding time violations in which resampling and analysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of valid results minus the number of possible results not reported.

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 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 performance evaluation (PE) 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.

Table 4.2.1-1 Statistical Calculations

Statistic	Symbol	Formula	Definition	Uses
Mean	$\overline{X}$	$\frac{\left(\sum_{i=1}^{n} x_i\right)}{n}$	Measure of central tendency	Used to determine average value of measurements
Standard Deviation	S	$\left(\frac{\Sigma(x_i-\overline{x})^2}{(n-1)}\right)^{1/2}$	Measure of relative scatter of the data	Used in calculating variation of measurements
Relative Standard Deviation	RSD	$(S/\overline{X}) \times 100$	Relative standard deviation, 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 pure matrix	Used to assess accuracy
Percent Recovery	%R	$\frac{\left(\begin{array}{c}\text{value of}\\ \text{spiked}\\ \text{sample}\end{array} - \begin{array}{c}\text{value of}\\ \text{unspiked}\\ \text{sample}\end{array}\right)}{\text{Value of added spike}} \times 100$	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

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

4.3.1 Method Detection Limits

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory shall establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. The laboratory shall revalidate these MDLs at least once per twelve month period. The laboratory shall provide the MDL demonstrations to AFCEE at the beginning of the project (i.e., before project samples are analyzed) and upon request in the format specified in Section 8. Results less than or equal to the MDL shall be reported as the MDL value and flagged with a "U" (see Section 8).

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, or
   b) the concentration equivalent of 3 times the standard deviation of replicate measurement of the analyte in reagent water, or
   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), or
   d) instrumental limitations.

(2) Prepare (i.e., extract, digest, etc.) and analyze seven samples of a matrix spike (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 equal to or in the same concentration range as the estimated method detection limit (recommended one to five times the estimated MDL).

(3) Determine the variance (S^2) for each analyte as follows:

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

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

(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:

$$MDL = 3.14(s)$$

(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 Reporting Limits

The laboratories participating in this work effort shall compare the results of the MDL demonstrations to the reporting limits (RLs) for each method that is listed in Section 7. 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). No results shall be reported below the MDL.

4.3.3 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. All results reported shall be within the calibration range. 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 for the method. This applies equally to multiresponse analytes (except as noted in Section 7). 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. Analyte concentrations are determined with either calibration curves or response factors (RFs). For gas chromatography (GC) and gas chromatography/mass spectroscopy (GC/MS) 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 laboratory control sample (LCS).

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 all 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 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 (not to exceed 20 environmental samples plus the associated laboratory QC samples) that are similar in composition (matrix) and analyzed sequentially. 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. All 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.

4.4.1 Laboratory Control Sample

The laboratory control sample (LCS) is analyte-free water for aqueous analyses or Ottawa sand for soil analyses (except metals where glass beads of 1mm diameter or smaller may be used) spiked with all analytes listed in the QC acceptance criteria table in Section 7 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 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. 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.

Whenever an analyte in an LCS is outside the acceptance limit, corrective action shall be performed. Low LCS results will prompt corrective action as outlined in Section 7, after the system problems have been resolved, the LCS and all samples in the affected AFCEE analytical batch will be reprepped and analyzed. When an analyte indicates a high recovery in an LCS, corrective action will be initiated to determine the cause. However, associated samples will be reanalyzed only for samples which show positive concentrations of the analytes in question. Samples showing non-detect for the analytes will not be reanalyzed. When an analyte in an LCS exceeds the upper or lower control limit and no corrective action is performed or the corrective action was ineffective, the appropriate validation flag, as described in Sections 7 and 8, shall be applied to all affected results.

4.4.2 Matrix Spike/Matrix Spike Duplicate

A matrix spike (MS) and matrix spike duplicate (MSD) is an aliquot of sample spiked with known concentrations of all analytes listed in the QC acceptance criteria table in Section 7 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. AFCEE does not use MSs and MSDs to control the analytical process.

A minimum of one MS and one MSD sample shall be analyzed for every 20 AFCEE samples.

The performance of the MS and MSD is evaluated against the QC acceptance limits given in the tables in Section 7. 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 and 8.

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, reprep and reanalyze the sample. If corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Sections 7 and 8, 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 and 8, shall be applied to the sample results.

4.4.5 Retention Time Windows

Retention time windows are used in GC and high performance liquid chromatography (HPLC) 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 8000B.

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 and 8, 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.

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 and 8, 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 in the analytical batch shall be repreped 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 and 8, shall be applied to the sample results.

4.4.8 Ambient Blank

The ambient blank consists of ASTM Type II reagent grade water poured into a volatile organic compound (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.

The frequency of collection for ambient blanks is specified in Section 3.2 of the FSP. Ambient blanks shall be collected downwind of possible VOC sources.

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 FSP. 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.

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.

One trip blank shall accompany each cooler of samples sent to the laboratory for analysis of VOCs.

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 an identification number in the field such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis. 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 FSP.

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 an identification number in the field such that they cannot be identified as replicate samples by laboratory personnel performing the analysis. 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 FSP.

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 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 is used. The result of the first column/detector shall be the result 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 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), EPA, American Association of Laboratory Accreditation (A2LA), procured from vendors who are ISO-9000 registered providing certified reference materials, or other equivalent AFCEE approved source, if available. If an NIST, EPA, A2LA, or ISO-9000 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.

4.5.4 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.

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5.0 SAMPLING PROCEDURES

5.1 FIELD SAMPLING

The field sampling procedures for collecting samples and sampling methods is included in Section 6.0 of the FSP.

5.1.1 Sample Containers

Sample containers are purchased precleaned and treated according to EPA specifications for the methods. Sampling containers that are reused are decontaminated between uses by the EPA-recommended procedures (i.e., EPA 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 which are not part of AFCEE QAPP methodology are included in Appendix B of this plan along with associated SOPs, RLs, and QC limits.

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

Name	Analytical Methods	Container[a]	Preservation[b,c]	Minimum Sample Volume or Weight	Maximum Holding Time
Alkalinity	E310.1	P, G	4°C	50 mL	14 days
Common anions	SW9056	P, G	None required	50 mL	28 days for Br^-, F^-, Cl^-, and SO_4^{-2}; 48 hours for NO_3^-, NO_2^- and PO_4^{-3}
Cyanide, total and amenable to chlorination	SW9010B SW9012A	P, G, T	4°C; NaOH to pH > 12, 0.6 g ascorbic acid	500 mL or 4 ounces	14 days (water and soil)
Filterable residue	E160.1	P, G	4°C	100 mL	7 days
~~Nonfilterable residue~~	~~E160.2~~	~~P, G~~	~~4°C~~	~~100 mL~~	~~7 days~~
Hydrogen ion (pH) (W, S)	SW9040B/ SW9045C	P, G	None required	N/A	Analyze immediately
Nitrogen, nitrate+nitrite	E353.1	P, G	4°C, H_2SO_4 to pH < 2	500 mL	28 days
Conductance	SW9050A	P, G	None required	N/A	Analyze immediately
Temperature	E170.1	P, G	None required	N/A	Analyze immediately
Dissolved oxygen	E360.1	G	None required	500 mL	Analyze immediately
Turbidity	E180.1	P, G	4°C	N/A	48 hours
Total organic carbon	SW9060	P, G, T	4°C, HCl or H_2SO_4 to pH < 2	500 mL	28 days
Chromium (VI)	SW7196A	P, G, T	4°C	500 mL or 8 ounces	24 hours (water); 30 days until extraction and 4 days after extraction (soil)
Mercury	SW7470A SW7471A	P, G, T	HNO_3 to pH < 2, 4°C	500 mL or 8 ounces	28 days (water and soil)
Metals (except chromium (VI) and mercury)	SW6010B SW6020 and SW-846 AA methods	P, G, T	HNO_3 to pH < 2, 4°C	500 mL or 8 ounces	180 days (water and soil)

a. Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).
b. No pH adjustment for soil.
c. Preservation with 0.008 percent $Na_2S_2O_3$ is only required when residual chlorine is present.

Table 5.1.2-1. Continued

Name	Analytical Methods	Container[a]	Preservation[b,c]	Minimum Sample Volume or Weight	Maximum Holding Time
~~Total petroleum hydrocarbons (TPH) volatile~~	~~SW8015 (modified)~~	~~G, Teflon-lined septum, T~~	~~4°C, HCl to pH < 2~~	~~2 x 40 mL or 4 ounces~~	~~14 days (water and soil); 7 days if unpreserved by acid~~
~~Total petroleum hydrocarbons (TPH) extractable~~	~~SW8015 (modified)~~	~~G, amber, T~~	~~4°C~~	~~1 liter or 8 ounces~~	~~7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)~~
~~Aromatic and Halogenated volatiles~~	~~SW8021B~~	~~G, Teflon-lined septum, T~~	~~4°C, HCl to pH < 2, 0.008% $Na_2S_2O_3$~~	~~2 x 40 mL or 4 ounces~~	~~14 days (water and soil); 7 days if unpreserved by acid~~
~~Nitrosamines~~	~~SW8070A~~	~~G, Teflon-lined cap, T~~	~~4°C~~	~~1 liter or 8 ounces~~	~~7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)~~
~~Chlorinated herbicides~~	~~SW8151A~~	~~G, Teflon-lined cap, T~~	~~4°C~~	~~1 liter or 8 ounces~~	~~7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)~~

a. Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).
b. No pH adjustment for soil.
c. Preservation with 0.008 percent $Na_2S_2O_3$ is only required when residual chlorine is present.

Table 5.1.2-1. Continued

Name	Analytical Methods	Container[a]	Preservation[b,c]	Minimum Sample Volume or Weight	Maximum Holding Time
~~Organochlorine pesticides~~	~~SW8081A~~	~~G, Teflon-lined cap, T~~	~~4°C~~	~~1 liter or 8 ounces~~	~~7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)~~
~~Polychlorinated biphenyls (PCBs)~~	~~SW8082~~	~~G, Teflon-lined cap, T~~	~~4°C~~	~~1 liter or 8 ounces~~	~~7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)~~
~~Organophosphorus pesticides/ compounds~~	~~SW8141A~~	~~G, Teflon-lined cap, T~~	~~4°C~~	~~1 liter or 8 ounces~~	~~7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)~~
~~Semivolatile organics~~	~~SW8270C~~	~~G, Teflon-lined cap, T~~	~~4°C, 0.008% $Na_2S_2O_3$~~	~~1 liter or 8 ounces~~	~~7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)~~
Volatile organics	SW8260B	G, Teflon-lined septum, T	4°C, 0.008% $Na_2S_2O_3$ (HCl to pH < 2 for volatile aromatics)[b]	2 x 40 mL or 4 ounces	14 days (water and soil); 7 days if unpreserved by acid

a. Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).
b. No pH adjustment for soil.
c. Preservation with 0.008 percent $Na_2S_2O_3$ is only required when residual chlorine is present.

Table 5.1.2-1. Concluded

Name	Analytical Methods	Container[a]	Preservation[b,c]	Minimum Sample Volume or Weight	Maximum Holding Time
~~Polynuclear aromatic hydrocarbons (PAHs)~~	~~SW8310~~	~~G, Teflon-lined cap, T~~	~~4°C, store in dark, 0.008% $Na_2S_2O_3$~~	~~1 liter or 8 ounces~~	~~7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)~~
~~Dioxins and furans~~	~~SW8280A SW8290~~	~~G, Teflon-lined cap, T~~	~~4°C, 0.008% $Na_2S_2O_3$ (kept dark)~~	~~1 liter or 8 ounces~~	~~30 days until extraction and 45 days after extraction (water and soil)~~
~~Ethylene dibromide (EDB)~~	~~SW8011~~	~~G, Teflon-lined cap, T~~	~~4°C, 0.008% $Na_2S_2O_3$~~	~~2 x 40 mL~~	~~28 days (water)~~
~~Explosive residues~~	~~SW8330~~	~~P, G, T~~	~~Cool, 4°C~~	~~1 liter or 8 ounces~~	~~7 days to extraction (water); 14 days to extraction (soil); analyze within 40 days after extraction~~
~~TCLP~~	~~SW1311~~	~~G, Teflon-lined cap, T~~	~~Cool, 4°C~~	~~1 liter or 8 ounces~~	~~14 days to TCLP extraction and 14 days after extraction (volatiles); 14 days to TCLP extraction, 7 days to prep extraction and 40 days after prep extraction (semivolatiles); 28 days to TCLP extraction and 28 days after extraction (mercury); 180 days to TCLP extraction and 180 days after extraction (metals)~~
~~Volatile Organics~~	~~TO-14~~	~~SUMMA® canister~~	~~none~~		~~14 days~~

a. Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).
b. No pH adjustment for soil.
c. Preservation with 0.008 percent $Na_2S_2O_3$ is only required when residual chlorine is present.

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 Quality Control (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
- 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 in accordance with (IAW) Section 6.2 and 6.3 of the FSP.

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 volatile organics compounds 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. 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°C ± 2°C. Acceptance criterion for the temperatures of the freezers shall be less than 0°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.

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

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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* (U.S. EPA SW-846, Third Edition, and its first, second and third update), *Methods for Chemical Analysis of Water and Waste* (U.S. EPA 1979), *ASTM Annual Book of Standards* (1993), and from manufacturers' literature. Additional screening methods not part of the AFCEE QAPP, including SOPs, RLs, QC limits, etc., to be employed for this LTM project can be found in Appendix B of this QAPP. These analytical parameters and methods include: TKN - EPA method 351.3; Ammonia – EPA method 350.2; COD – Standard Methods (SM) 5220C; BOD – SM 5210B; Phenols – EPA method SW846 9065; Total Hardness – EPA method 130.2; Color – EPA method 110.2.

Table 6-1. Screening Analytical Methods

Method	Parameter
SW9040B	pH (water)
SW9050A	Conductance
SW9060	Total organic carbon
E160.1	Filterable residue
E170.1	Temperature
E180.1	Turbidity
E310.1	Alkalinity
E360.1	Dissolved oxygen
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 EPA Method SW9040B (Water) –pH

pH measurements shall be performed for water samples using method SW9040. Measurements are determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode.

6.1.2 EPA Method SW9050A–Conductance

Standard conductivity meters are used. Temperature is also reported.

6.1.3 EPA Method SW9060–Total Organic Carbon

Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide by either catalytic combustion or wet chemical oxidation. The carbon dioxide formed is then either measured directly by an infrared detector or converted to methane and measured by a flame ionization detector. The amount of carbon dioxide or methane in a sample is directly proportional to the concentration of carbonaceous material in the sample.

Method	Analyte	Water	
		RL	Unit
SW9060	Total organic carbon	1	mg/L

6.1.4 EPA Method 160.1–Filterable Residue

A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180 °C.

Method	Analyte	Water	
		RL	Unit
E160.1	Total dissolved solids	10	mg/L

6.1.5 EPA Method 170.1–Temperature

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

6.1.6 EPA 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 deionized water.

6.1.7 EPA Method 310.1–Alkalinity

In this method, an unaltered sample is titrated to an end point of pH 4.5 using hydrochloric or sulfuric acid.

Method	Analyte	Water	
		RL	Unit
E310.1	Alkalinity[1]	10	mg/L

[1] alkalinity measured as calcium carbonate equivalence

6.1.8 EPA 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.

6.1.9 ASTM 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.

6.2 CALIBRATION AND QC 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 QC Procedures for Screening Methods

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Data Flagging Criteria[b]
~~SW-846[c]~~	~~Moisture~~	~~Duplicate sample~~	~~1 per 20 samples~~	~~% solid RPD ≤ 15%~~	~~Correct problem, repeat measurement. If still out, flag data~~	~~J if RPD >15% and ≤30%~~ ~~R if RPD >30%~~
~~SW9045C~~	~~pH (soil)~~	~~2-point calibration with pH buffers~~	~~1 per 10 samples analyzed~~	~~± 0.05 pH unit~~	~~Check with new buffers; if still out, repair meter; repeat calibration check~~	~~R~~
		~~pH 7 buffer~~	~~At each sample location~~	~~± 0.1 pH unit~~	~~Recalibrate~~	~~R~~
		~~Duplicate sample~~	~~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	R
		Field duplicate	10% of field samples	± 5%	Correct problem, repeat measurement	J
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	R
		pH 7 buffer	At each sample location	± 0.1 pH units	Correct problem, recalibrate	R
		Field duplicate	10% of field samples	± 0.1 pH units	Correct problem, repeat measurement	J
E170.1	Temperature	Field duplicate	10% of field samples	± 1.0°C	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 shall first be 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".
c. Described in method SW3550.

Table 6.2-1. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Data Flagging Criteria[b]
E180.1	Turbidity	Calibration with one formazin standard per instrument range used	Once per day at beginning of testing	± 5 units, 0–100 range ± 0.5 units, 0–0.2 range ± 0.2 units, 0–1 range	If calibration is not achieved, check meter; replace if necessary, recalibrate	R
		Field duplicate	10% of field samples	RPD ≤ 20%	Correct problem, repeat measurement	J
~~None~~	~~Organic vapor concentrations (FID and PID)~~	~~3 point calibration~~	~~Monthly~~	~~correlation coefficient ≥ 0.995~~	~~Recalibrate; check instrument and replace if necessary~~	~~R~~
		~~Calibration verification and check~~	~~Daily at beginning and end of day~~	~~Response ± 20% of expected value~~	~~Correct problem, recalibrate~~	~~R~~
SW9060	Total organic carbon	Method blank	Daily or one per batch, whichever is more frequent	< RL	Clean system; reanalyze blank. Repeat until analyte < RL	B
		Field duplicate	10% of field samples	RPD < 20%	Repeat measurement	J
E160.1	Filterable residue	Field duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
~~E160.2~~	~~Nonfilterable residue~~	~~Field duplicate~~	~~10% of field samples~~	~~RPD < 20%~~	~~Correct problem, repeat measurement~~	~~J~~
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	R
		Calibration with one standard	Once per day	Two successive readings ± 10 millivolts	Correct problem, recalibrate	R
		Field duplicate	10% of field samples	± 10 millivolts	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 shall first be 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".

Table 6.2-1. Concluded

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Data Flagging Criteria[b]
~~SW1110~~	~~Corrosivity~~	~~Duplicate~~	~~10% of field samples~~	~~RPD < 20%~~	~~Correct problem, repeat measurement~~	~~J~~
E310.1	Alkalinity	Field 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
~~SW4020~~	~~PCBs by immunoassay~~	~~Field duplicate~~	~~10% of field samples~~	~~RPD < 20%~~	~~Correct problem, repeat measurement~~	~~J~~
~~SW4030~~	~~Petroleum hydrocarbons by immunoassay~~	~~Field duplicate~~	~~10% of field samples~~	~~RPD < 20%~~	~~Correct problem, repeat measurement~~	~~J~~
~~ASTM D3416~~	~~Methane~~	~~Single point calibration~~	~~Daily, prior to sample analysis~~	~~Delineation from database average within ± 20%~~	~~Recalibrate~~	~~R~~
		~~Method blank~~	~~Daily or one per batch, whichever is more frequent~~	~~< RL~~	~~Clean system; reanalyze blank and Repeat until all analytes < RL~~	~~B~~
		~~Duplicate~~	~~1 per batch or 10%~~	~~RPD ≤ 20%~~	~~Analyze third aliquot: if still out, flag data~~	~~J~~

a. All corrective actions shall be documented, and the records shall be maintained by the prime contractor.
b. All screening results shall first be 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".

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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* (U.S. EPA SW-846, Third Edition, and its first, second and third update); *Handbook for the Installation Restoration Program (IRP) Remedial Investigations and Feasibility Studies (RI/FS)* (Handbook), September 1993; *U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, U.S. EPA, Office of Solid Waste and Emergency Response, Washington, D.C., Publication 9240.1-05-01, EPA-540/R-94-013, PB94-963502, February 1994; and *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, U.S. EPA, Office of Solid Waste and Emergency Response, Washington, D.C., Publication 9240.1-05, EPA-540/R-94-012, PB94-963501, February 1994. Definitions of terms are given in Section 4.0, and data validation procedures are presented in Section 8.0.

7.1 PREPARATION METHODS

Extraction and digestion procedures for liquid and solid matrices presented in this section are outlined in Table 7.1-1. The appropriate preparation method to be used (if applicable) for each analytical method is given in the RL tables.

Table 7.1-1. Extraction and Digestion Procedures

Method	Parameter
SW3005A	Acid Digestion of Water Samples for Metals Analysis
SW3010A	Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW3015	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW3020A	Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW5030B	Purge and Trap

7.1.1 Method SW3005A–Acid Digestion of Water Samples for Metals Analysis

This method is an acid digestion procedure used to prepare water samples for metals analysis. The digested samples are analyzed for total recoverable and dissolved metals determination by inductively coupled plasma (ICP).

For analysis of total recoverable metals, the entire sample is acidified at collection time. For analysis of dissolved metals, upon collection the samples are filtered then acidified.

7.1.2 Method SW3010A–Acid Digestion of Aqueous Samples and Extracts for Metals Analysis

Method SW3010A prepares aqueous or waste samples for total metals determination by ICP. The samples are vigorously digested with acid and then diluted.

7.1.3 Method SW3015–Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Metals Analysis

This method is used to prepare aqueous or waste samples that contain suspended solids, for total metals determination by graphite furnace atomic absorption spectroscopy (GFAA) or ICP. The samples are digested with acid and heated in a microwave.

7.1.4 Method SW3020A–Acid Digestion of Aqueous Samples and Extracts for Metals Analysis

Method SW3020A prepares aqueous or waste samples for total metals determination by GFAA or ICP. The samples are vigorously digested with acid and then diluted.

7.1.5 Method SW5030B-Purge and Trap

Method SW5030B describes sample preparation and extraction for the analysis of VOCs. The method is applicable to nearly all types of samples, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, water, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The success of this method depends on the level of interferences in the sample. Results may vary due to the large variability and complexity of matrices of solid waste 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 backflushed with inert gas to desorb the components onto a GC column.

7.1.6 Method SW3060A–Alkaline Digestion for Hexavalent Chromium

Method SW3060A is applicable to the preparation of sediment, sludge, and soil samples for analysis of hexavalent chromium by UV-VIS spectrophotometry. The samples are digested with sodium hydroxide.

7.2 ANALYTICAL PROCEDURES

The analytical procedures presented in this section are outlined in Table 7.2-1.

A brief description and three tables for each method are included in the following subsections. The first table presents the RLs for each analyte in the method. The RLs are presented for both soil and water matrices. The second table presents the acceptance criteria for the accuracy of spiked analyte and surrogate recoveries. This table also presents the acceptance criteria for the precision of matrix, field, and laboratory duplicate recoveries. The third table presents the calibration and QC procedures for each method. Corrective actions and data flagging criteria are also included in this table.

In the third 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 shall be applied in the event that the method-required calibration and QC acceptance criteria are not met.

Table 7.2-1. Analytical Procedures

Analytical Method	Parameter	Preparatory Methods
8260B	Volatile organics (water)	5030B
6010B	Trace metals by ICPES (water and soil)	3005A, 3010A, 3015, 3020A
7470A	Mercury (water)	(see analytical method)
9010B	Cyanide (water)	(see analytical method)
9056	Common anions	N/A
7196A	Hexavalent chromium	3060A

7.2.1 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). An inert gas is bubbled through the water samples (or a soil-water slurry for soil samples) to transfer the purgeable organic compounds from the liquid to vapor phase. Soil samples with higher contaminant levels are extracted using methanol before purging. 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.

Calibration—The mass spectrometer is tuned daily to give an acceptable spectrum for 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 added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.1-2 and 7.2.1-3.

Table 7.2.1-1. RLs for Method SW8260B

Parameter/Method	Analyte	Water	
		RL	Unit
VOCs	1,1,1,2-Tetrachloroethane	0.5	µg/L
SW8260B	1,1,1-TCA	0.8	µg/L
	1,1,2,2-Tetrachloroethane	0.4	µg/L
	1,1,2-TCA	1.0	µg/L
	1,1-DCA	0.4	µg/L
	1,1-DCE	1.2	µg/L
	1,1-Dichloropropene	1.0	µg/L
	1,2,3-Trichlorobenzene	0.3	µg/L
	1,2,3-Trichloropropane	3.2	µg/L
	1,2,4-Trichlorobenzene	0.4	µg/L
	1,2,4-Trimethylbenzene	1.3	µg/L
	1,2-DCA	0.6	µg/L
	1,2-DCB	0.3	µg/L
	1,2-Dibromo-3-chloropropane	2.6	µg/L
	1,2-Dichloropropane	0.4	µg/L
	1,2-EDB	0.6	µg/L
	1,3,5-Trimethylbenzene	0.5	µg/L
	1,3-DCB	1.2	µg/L
	1,3-Dichloropropane	0.4	µg/L
	1,4-DCB	0.3	µg/L
	1-Chlorohexane	0.5	µg/L
	2,2-Dichloropropane	3.5	µg/L
	2-Butanone	10*	µg/L
	2-Chlorotoluene	0.4	µg/L
	2-Hexanone	10*	µg/L
	4-Methyl-2-pentanone	10*	µg/L
	4-Chlorotoluene	0.6	µg/L
	Acetone	10*	µg/L
	Acrylonitrile	100*	µg/L
	Benzene	0.4	µg/L
	Bromobenzene	0.3	µg/L
	Bromochloromethane	0.4	µg/L
	Bromodichloromethane	0.8	µg/L
	Bromoform	1.2	µg/L
	Bromomethane	1.1	µg/L
	Carbon disulfide	5.0*	µg/L
	Carbon tetrachloride	2.1	µg/L
	Chlorobenzene	0.4	µg/L
	Chloroethane	1.0	µg/L
	Chloroform	0.3	µg/L
	Chloromethane	1.3	µg/L
	Cis-1,2-DCE	1.2	µg/L
	Cis-1,3-Dichloropropene	1.0	µg/L
	Dibromochloromethane	0.5	µg/L

Table 7.2.1-1. Concluded

Parameter/Method	Analyte	Water	
		RL	Unit
VOCs SW8260B (concluded)	Dibromomethane	2.4	µg/L
	Dichlorodifluoromethane	1.0	µg/L
	Ethylbenzene	0.6	µg/L
	Hexachlorobutadiene	1.1	µg/L
	Iodomethane	10*	µg/L
	Isopropylbenzene	0.5	µg/L
	m-Xylene	0.5	µg/L
	Methylene chloride	5.0	µg/L
	n-Butylbenzene	1.1	µg/L
	n-Propylbenzene	0.4	µg/L
	Naphthalene	0.4	µg/L
	o-Xylene	1.1	µg/L
	p-Isopropyltoluene	1.2	µg/L
	p-Xylene	1.3	µg/L
	Sec-Butylbenzene	1.3	µg/L
	Styrene	0.4	µg/L
	TCE	1.0	µg/L
	Tert-Butylbenzene	1.4	µg/L
	Tetrachloroethene	1.4	µg/L
	Toluene	1.1	µg/L
	Trans-1,4-Dichloro-2-butene	100*	µg/L
	Trans-1,2-DCE	0.6	µg/L
	Trans-1,3-Dichloropropene	1.0	µg/L
	Trichlorofluoromethane	0.8	µg/L
	Vinyl acetate	10*	µg/L
	Vinyl chloride	1.1	µg/L

* RL currently based on a 5 ml purge volume, the 25 ml volume will result in lower RLs, to be determined by the laboratory during time of sample analysis.

Table 7.2.1-2. QC Acceptance Criteria for Method SW8260B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Assoc. IS
SW8260B	1,1,1,2-Tetrachloroethane	72–125	≤ 20	2
	1,1,1-TCA	75–125	≤ 20	1
	1,1,2,2-Tetrachloroethane	74–125	≤ 20	3
	1,1,2-TCA	75–127	≤ 20	1
	1,1-DCA	72–125	≤ 20	1
	1,1-DCE	75–125	≤ 20	1
	1,1-Dichloropropene	75–125	≤ 20	1
	1,2,3-Trichlorobenzene	75–137	≤ 20	3
	1,2,3-Trichloropropane	75–125	≤ 20	3
	1,2,4-Trichlorobenzene	75–135	≤ 20	3
	1,2,4-Trimethylbenzene	75-125	≤ 20	3
	1,2-DCA	68–127	≤ 20	1
	1,2-DCB	75-125	≤ 20	3
	1,2-Dibromo-3-chloropropane	59–125	≤ 20	3
	1,2-Dichloropropane	70–125	≤ 20	1
	1,2-EDB	75-125	≤ 20	2
	1,3,5-Trimethylbenzene	72–112	≤ 20	3
	1,3-DCB	75-125	≤ 20	3
	1,3-Dichloropropane	75-125	≤ 20	2
	1,4-DCB	75-125	≤ 20	3
	1-Chlorohexane	75-125	≤ 20	2
	2,2-Dichloropropane	75-125	≤ 20	1
	2-Butanone	63-151	≤ 20	1
	2-Chlorotoluene	73–125	≤ 20	3
	2-Hexanone	52-168	≤ 20	2
	4-Methyl-2-pentanone	62-152	≤ 20	2
	4-Chlorotoluene	74–125	≤ 20	3
	Acetone	57-140	≤ 20	1
	Acrylonitrile	70-130	≤ 20	1
	Benzene	75–125	≤ 20	1
	Bromobenzene	75–125	≤ 20	3
	Bromochloromethane	73–125	≤ 20	1
	Bromodichloromethane	75–125	≤ 20	1
	Bromoform	75–125	≤ 20	2
	Bromomethane	72–125	≤ 20	1
	Carbon disulfide	73-140	≤ 20	1
	Carbon Tetrachloride	62–125	≤ 20	1
	Chlorobenzene	75–125	≤ 20	2
	Chloroethane	65–125	≤ 20	1
	Chloroform	74–125	≤ 20	1
	Chloromethane	75-125	≤ 20	1
	Cis-1,2-DCE	75–125	≤ 20	1
	Cis-1,3-Dichloropropene	74–125	≤ 20	1
	Dibromochloromethane	73–125	≤ 20	2

Table 7.2.1-2. Concluded

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Assoc. IS
SW8260B (Concluded)	Dibromomethane	69–127	≤ 20	1
	Dichlorodifluoromethane	75-125	≤ 20	1
	Ethylbenzene	75–125	≤ 20	2
	Hexachlorobutadiene	75–125	≤ 20	3
	Iodomethane	70-130	≤ 20	1
	Isopropylbenzene	75–125	≤ 20	3
	m-Xylene	75–125	≤ 20	2
	Methylene chloride	75–125	≤ 20	1
	n-Butylbenzene	75–125	≤ 20	3
	n-Propylbenzene	75–125	≤ 20	3
	Naphthalene	75–125	≤ 20	3
	o-Xylene	75–125	≤ 20	2
	p-Isopropyltoluene	75–125	≤ 20	3
	p-Xylene	75–125	≤ 20	2
	Sec-Butylbenzene	75–125	≤ 20	3
	Styrene	75–125	≤ 20	2
	TCE	71–125	≤ 20	1
	Tert-butylbenzene	75-125	≤ 20	3
	Tetrachloroethene	71–125	≤ 20	2
	Toluene	74–125	≤ 20	1
	Trans-1,4-Dichloro-2-butene	70-130	≤ 20	3
	Trans-1,2-DCE	75–125	≤ 20	1
	Trans-1,3-Dichloropropene	66–125	≤ 20	1
	Trichlorofluoromethane	67–125	≤ 20	1
	Vinyl acetate	42-164	≤ 20	1
	Vinyl Chloride	46–134	≤ 20	1
	Surrogates:			
	Dibromofluoromethane	75–125		
	Toluene-D8	75–125		
	4-Bromofluorobenzene	75–125		
	1,2-DCA-D4	62–139		
	Internal Standards:			
	Fluorobenzene			1
	Chlorobenzene-D5			2
	1,4-Dichlorobenzend-D			3

Table 7.2.1-3. Summary of Calibration and QC Procedures for Method SW8260B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8260B	Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.30[c] and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear- mean RSD for all analytes ≤15% with no individual analyte RSD >30%		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - least squares regression r > 0.995		
				option 3 non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample

Table 7.2.1-3. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.30[c]; and CCCs ≤ 20% difference (when using RFs)or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ±20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification
SW8260B	Volatile Organics	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.1-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		ISs	Immediately after or during data acquisition for each sample	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL. EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Apply R to all results for analytes associated with the IS
		Method blank	One per analytical batch	No analytes detected ≥ 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) in all samples in the associated analytical batch

Table 7.2.1-3. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.1-2	Correct problem then for low LCS results reprep and analyze the LCS and all samples in the affected AFCEE analytical batch; for high LCS results associated samples will be reanalyzed only for samples which show positive concentrations of the analytes outside the QC criteria	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.1-2	none	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

Table 7.2.1-3. Concluded

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8260B	Volatile Organics	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	Apply R to all results for all samples associated with the tune
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.1-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for a surrogate, apply J to all positive results if the %R < LCL for a surrogate, apply J to all positive results; apply R to all non-detect results If any surrogate recovery is <10%, apply R to all results
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 7.2.2-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		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-dichloroethane

7.2.2 Method SW6010B-Trace Elements (Metals) by Inductively Coupled Plasma Atomic Emission Spectroscopy for Water and Soil

Samples are analyzed for trace elements or metals using method SW6010B for water and soils. Analysis for most metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICPES). The elements and corresponding RLs for this method are listed in Table 7.2.2-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.2-2 and 7.2.2-3.

Table 7.2.2-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water	
		RL	Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L
	Antimony	0.05	mg/L
	Arsenic	0.03	mg/L
	Barium	0.005	mg/L
	Beryllium	0.005	mg/L
	Boron	0.10	mg/L
	Cadmium	0.007	mg/L
	Calcium	1.1	mg/L
	Chromium	0.01	mg/L
	Cobalt	0.006	mg/L
	Copper	0.01	mg/L
	Iron	0.20	mg/L
	Lead	0.025	mg/L
	Magnesium	0.10	mg/L
	Manganese	0.003	mg/L
	Molybdenum	0.015	mg/L
	Nickel	0.01	mg/L
	Potassium	0.50	mg/L
	Selenium	0.03	mg/L
	Silver	0.01	mg/L
	Sodium	1.0	mg/L
	Thallium	0.08	mg/L
	Vanadium	0.01	mg/L
	Zinc	0.01	mg/L

Table 7.2.2-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
SW6010B	Aluminum	75-125	≤ 20
	Antimony	75-125	≤ 20
	Arsenic	75-125	≤ 20
	Barium	75-125	≤ 20
	Beryllium	75-125	≤ 20
	Boron	70-130	≤ 20
	Cadmium	75-125	≤ 20
	Calcium	75-125	≤ 20
	Chromium	75-125	≤ 20
	Cobalt	75-125	≤ 20
	Copper	75-125	≤ 20
	Iron	75-125	≤ 20
	Lead	75-125	≤ 20
	Magnesium	75-125	≤ 20
	Manganese	75-125	≤ 20
	Molybdenum	75-125	≤ 20
	Nickel	75-125	≤ 20
	Potassium	75-125	≤ 20
	Selenium	75-125	≤ 20
	Silver	75-125	≤ 20
	Sodium	75-125	≤ 20
	Thallium	75-125	≤ 20
	Vanadium	75-125	≤ 20
	Zinc	75-125	≤ 20

Table 7.2.2-3. Summary of Calibration and QC Procedures for Method SW6010B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6010B	ICP Metals	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A	N/A	Apply R to all results for specific analyte(s) for all samples associated with the calibration if calibration not done
		Initial calibration verification (second source)	Daily after initial calibration	All analytes within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration blank	After every calibration verification	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) in all samples associated with the blank
		Calibration verification (Instrument Check Standard)	After every 10 samples and at the end of the analysis sequence	All analyte(s) within ±10% of expected value and RSD of replicate integrations <5%	Repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.2-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst

Table 7.2.2-3. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6010B	ICP Metals	Method blank	One per analytical batch	No analytes detected ≥ 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) in all samples in the associated analytical batch
		Interference check solution (ICS)	At the beginning of an analytical run	Within ±20% of expected value	Terminate analysis; correct problem; reanalyze ICS; reanalyze all affected samples	Apply R to all results for specific analyte(s) in all samples associated with the ICS
		LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.2-2	Correct problem then for low LCS results reprep and analyze the LCS and all samples in the affected AFCEE analytical batch; for high LCS results associated samples will be reanalyzed only for samples which show positive concentrations of the analytes outside the QC criteria	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test	Each new sample matrix	1:5 dilution must agree within ±10% of the original determination	Perform post digestion spike addition	Apply J to all sample results if either of following exist: (1) dilution test not run (2) RPD ≥10%

Table 7.2.2-3. Concluded

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6010B	ICP Metals	Post digestion spike addition	When dilution test fails	Recovery within 75-125% of expected results	Correct problem then reanalyze post digestion spike addition	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition If post digestion spike addition recovery is < 10%, apply R to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.2-2	none	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
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 7.2.2-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		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. b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

7.2.3 Method SW7470A/SW7471A–Mercury Manual Cold-Vapor Technique

Water and soil samples are analyzed for mercury using methods SW7470A and SW7471A, respectively. This method is a cold-vapor, flameless atomic absorption (AA) technique based on the absorption of radiation by mercury vapor. Mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an AA spectrophotometer. Mercury concentration is measured as a function of absorbance. The RLs for these methods are listed in Table 7.2.3-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.3-2 and 7.2.3-3.

Table 7.2.3-1. RLs for Method SW7470A

Parameter/Method	Analyte	Water	
		RL	Unit
SW7470A (W)	Mercury	0.001	mg/L

Table 7.2.3-2. QC Acceptance Criteria for Method SW7470A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
SW7470A	Mercury	77–120	≤ 15

Table 7.2.3-3. Summary of Calibration and QC Procedures for Method SW7470A/SW7471A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7470A SW7471A	Mercury	Initial multipoint calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within ±20% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.3-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ 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 in all samples in the associated analytical batch

Table 7.2.3-3. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7470A SW7471A	Mercury	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.3-2	Correct problem then for low LCS results reprep and analyze the LCS and all samples in the affected AFCEE analytical batch; for high LCS results associated samples will be reanalyzed only for samples which show positive concentrations of the analytes outside the QC criteria	For specific analyte in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test; five-fold dilution test	Each preparatory batch	Five times dilution sample result must be ±10% of the undiluted sample result	Perform recovery test	Apply J to all sample result if either of following exist: (1) dilution test not run (2) RPD ≥10%
		Recovery test	When dilution test fails	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range

Table 7.2.3-3. Concluded

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7470A SW7471A	Mercury	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.3-2	none	For the specific analyte 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
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 7.2.3-1	none	Apply R to all results for the specific analyte in all samples analyzed
		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.

7.2.4 Method SW9010B/SW9012A-Total Cyanide and Cyanide Amenable to Chlorination

Water and waste samples are analyzed for total cyanide using method SW9010B or SW9012A. These methods are equivalent in principle of analysis; SW9010B is a manual procedure, and SW9012A is an automated procedure.

Both methods are used to determine the concentration of inorganic cyanide in aqueous wastes and leachates. The methods detect inorganic cyanides that are present as either sample soluble salts or complex radicals. It is used to determine values for both total cyanide and cyanide amenable to chlorination. The cyanide is released by refluxing the sample with a strong acid and catalyst and distillation. Total cyanide in soils is determined after acidification of the soil and distillation. The cyanide ion in the absorbing solution is then determined by spectrophotometry for method SW9010B and by automated colorimetry for method SW9012A. RLs for cyanide are listed in Table 7.2.4-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.4-2 and 7.2.4-3.

Table 7.2.4-1. RLs for Method SW9010B/SW9012A

Parameter/Method	Analyte	Water	
		RL	Unit
SW9010B/SW9012A	Total cyanide	0.02	mg/L

Table 7.2.4-2. QC Acceptance Criteria for Method SW9010B/SW9012A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
SW9010B SW9012A	Total cyanide	79–114	≤ 20

Table 7.2.4-3. Summary of Calibration and QC Procedures for Method SW9010B/SW9012A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW9010B/ SW9012A	Cyanide	Multipoint calibration curve (six standards and a calibration blank)	Initial daily calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Distilled standards (one high and one low)	Once per multipoint calibration	Cyanide within ±10% of true value	Correct problem then repeat distilled standards	Apply R to all results for the specific analyte for all samples associated with the calibration
		Second-source calibration verification	Once per stock standard preparation	Cyanide within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to the result for the specific analyte for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.4-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to the specific analyte result for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to the result for the specific analyte in all samples in the associated analytical batch

Table 7.2.4-3. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW9010B/ SW9012A	Cyanide	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.4-2	Correct problem then for low LCS results reprep and analyze the LCS and all samples in the affected AFCEE analytical batch; for high LCS results associated samples will be reanalyzed only for samples which show positive concentrations of the analytes outside the QC criteria	For the specific analyte in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.4-2	none	For the specific analyte 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
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 7.2.4-1	none	Apply R to all results for the specific analyte in all samples analyzed
		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.

7.2.5 Method SW9056–Common Anions

This method addresses the sequential determination of the anions chloride, fluoride, bromide, nitrate, nitrite, phosphate, and sulfate in the collection solutions from the bomb combustion of solid waste samples, as well as water samples.

A small volume of combustate collection solution or other water sample is injected into an ion chromatograph to flush and fill a constant volume sample loop. The sample is then injected into a stream of elutent.

The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn (guard) column and a separator column, are packed with a low-capacity, strongly basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

RLs are listed in Table 7.2.5-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.5-2 and 7.2.5-3.

Table 7.2.5-1. RLs for Method SW9056

Parameter/Method	Analyte	Water	
		RL	Unit
Common Anions SW9056	Bromide	0.5	mg/L
	Chloride	1.0	mg/L
	Fluoride	1.0	mg/L
	Nitrate	1.0	mg/L
	Nitrite	1.0	mg/L
	Phosphate	1.0	mg/L
	Sulfate	1.0	mg/L

Table 7.2.5-2. QC Acceptance Criteria for Method SW9056

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
SW9056	Bromide	85–121	≤ 20
	Chloride	90–115	≤ 20
	Fluoride	84–117	≤ 27
	Nitrate	88–109	≤ 20
	Nitrite	85–114	≤ 20
	Phosphate	82–112	≤ 24
	Sulfate	82–116	≤ 20

Table 7.2.5-3. Summary of Calibration and QC Procedures for Method SW9056

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW9056	Common anions	Multipoint calibration for all analytes (minimum 3 standards and one calibration blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per multipoint calibration	All analytes within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time over 8 hour period	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis or when elutent is changed	All analytes within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within ±5% of expected response	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

Table 7.2.5-3. Continued

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW9056	Common anions	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.2.5-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ 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) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.5-2	Correct problem then for low LCS results reprep and analyze the LCS and all samples in the affected AFCEE analytical batch; for high LCS results associated samples will be reanalyzed only for samples which show positive concentrations of the analytes outside the QC criteria	For specific analyte(s) in all samples the associat analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Duplicate	One per every 10 samples	%D ≤10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results

Table 7.2.5-3. Concluded

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW9056	Common anions	MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.5-2	none	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
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 7.2.5-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		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.

7.2.6 Method SW7196A-Hexavalent Chromium (Colorimetric)

Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically. RLs for this method are listed in Table 7.2.6-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.6-2 and 7.2.6-3.

Table 7.2.6-1. RLs for Method SW7196A

Parameter/Method	Analyte	Water	
		RL	Unit
SW7196A	Hexavalent Chromium	0.5	mg/L

Table 7.2.6-2. QC Acceptance Criteria for Method SW7196A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
SW7196A	Hexavalent Chromium	86–117	≤ 15

Table 7.2.6-3. Summary of Calibration and QC Procedures for Method SW7196A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7196A	Hexavalent Chromium	Multipoint calibration curve (minimum three standards and a blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the specific analyte result for all samples associated with the calibration
		Second-source calibration verification	After each new stock standard preparation	Analytes within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to the specific analyte result for all samples associated with the calibration
		Calibration verification	After every 15 samples and at the end of the analysis sequence	Chromium within ±20% of expected value	Correct problem then repeat initial calibration and reanalyze all samples since last successful calibration	Apply R to the specific analyte result in all samples since the last acceptable 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 7.2.6-2	Recalculate results; locate and fix problem with system and then rerun demonstration	Apply R to the specific analyte result for all samples analyzed by the analyst
		Verification check to ensure lack of reducing condition and/or interference	Once for every sample matrix analyzed	Spike recovery between 85-115%	If check indicates interference, dilute and reanalyze sample persistent interference indicates the need to use and alternate method	Apply R to the specific analyte result for all samples analyzed since the last acceptable verification check
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 7.2.6-1	none	Apply R to all specific analyte results for all samples analyzed

Table 7.2.6-3. Concluded

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7196A	Chromium	Method blank	One per analytical batch	No analyte detected > RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to the specific analyte result for all samples in the associated analytical batch
		LCS	One LCS per analytical batch	QC acceptance criteria, Table 7.2.6-2	Correct problem then for low LCS results reprep and analyze the LCS and all samples in the affected AFCEE analytical batch; for high LCS results associated samples will be reanalyzed only for samples which show positive concentrations of the analytes outside the QC criteria	For specific analyte in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply to all positive results, apply R to all non-detects
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 7.2.6-2	none	For the specific analyte 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
		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.

8.0 DATA REDUCTION, REVIEW, VERIFICATION, REPORTING, VALIDATION, AND RECORDKEEPING

The data reduction, review, reporting, and validation procedures described in this section will ensure; (1) complete documentation is maintained, (2) transcription and data reduction errors are minimized, (3) the data are reviewed and documented, and (4) the reported results are qualified if necessary. Laboratory data reduction and verification procedures are required to ensure the overall objectives of analysis and reporting meet method and project specifications.

8.1 DATA REVIEW, VALIDATION, AND REPORTING REQUIREMENTS FOR SCREENING DATA

The analysts shall perform a 100 percent review of the screening data. The screening data methods are identified in Table 6-1 of Section 6. All screening data shall be qualified with an *S* flag and shall be further qualified if critical calibration and QC requirements are not acceptable. The calibration, QC requirements, corrective action requirements, and flagging criteria required are shown in Table 6.2-1 in Section 6. The flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed. "*S*" designator flags shall be maintained in the final data qualification. When the data are reviewed and qualified, the analyst shall apply a final qualifier to any data that has been affected by multiple qualifiers. This final qualifier shall reflect the most severe qualifier that was applied to the data. The allowable final data qualifiers for screening data and the hierarchy of data qualifiers, listed in order of the most severe through the least severe, are *SR, SJ, SB*, and *SU*. Therefore, the allowable final data qualifiers for screening data are *SR, SJ, SB, SU, and S.*

The definitions of the data qualifiers are shown in Table 8.2-1. A summary of the flagging conventions of field screening methods is given in Table 6.2-1.

Screening data report packages 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 review the entire screening data report package with the field records. The prime contractor (1) shall determine if the data quality objectives have been met, and (2) shall calculate the data completeness for the project. These results shall be included in the data package deliverable.

8.2 DATA REVIEW, VALIDATION, AND REPORTING REQUIREMENTS FOR DEFINITIVE DATA

MDLs and results shall be reported to one decimal place more than the corresponding RL. Soil/sediment 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 shall be taken for analysis (i.e., RLs and MDLs are NOT adjusted for dry weight). RLs and MDLs are adjusted for dilutions.

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 definitive data methods are identified in Section 7.2. The calibration, QC requirements, corrective action requirements, and flagging criteria required for definitive data are shown in the tables in Section 7.2, and in summary Tables 8.2-2, 8.2-3, and 8.2-4. The flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Data qualifiers shall be added or, if applied by a software package, reviewed by the laboratory supervisor of the respective analytical section, after the first and second level of laboratory data reviews have been performed. Analytical batch comments shall be added to the first page of the definitive data report packages to explain any nonconformance or other issues. When data are qualified, the laboratory supervisor shall apply a final qualifier to any data that have been affected by multiple qualifiers. This final qualifier shall reflect the most severe qualifier that was applied to the data, i.e., all data will have only one data qualifying flag associate with it. 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, F, J, B*, and *U*. The definitions of the data qualifiers are shown in Table 8.2-1.

The one exception to these data flagging criteria rules applies to the tentatively identified compounds (TICs) that are identified only in the GC/MS methods. These TICs numerical results will always be qualified with one and only one flag for any reason, and that is 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 manager shall perform a sanity check review on all the completed data packages.

The prime contractor's project manager shall review the entire definitive data report package, and with the field records, apply the final data qualifiers for the definitive data. The laboratory shall apply data qualifying flags to each environmental field QC sample, i.e., ambient blanks, equipment blanks,

trip blanks, field duplicates, matrix spike (MS) samples, and matrix spike duplicate (MSD) samples. The prime contractor shall review the field QC samples and field logs, and shall then appropriately flag any of the associated samples identified with the field QC sample, as explained in Table 8.2-2 and 8.2-3. Each matrix spike sample shall only be qualified by the laboratory, while the prime contractor shall apply the final 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.

The prime contractor (1) shall determine if the data quality objectives have been met, and (2) shall calculate the data completeness for the project. These results shall be included in the data package deliverable as described in Section 8.8.

Table 8.2-1 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.
F	The analyte was positively identified but the associated numerical value is below the RL.
R	The data are unusable due to deficiencies in the ability to analyze the sample and meet QC criteria.
B	The analyte was found in an associated blank, 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 (using GC/MS)

Table 8.2-2. General Flagging Conventions

QC Requirement	Criteria	Flag	Flag Applied To
Holding Time	Time exceeded for extraction or analysis	R	All analytes in the sample
LCS	% R > UCL	J for the positive results	The specific analyte(s) in all samples in the associated AAB
	%R < LCL	J for the positive results, R for the nondetects	
Method Blank	Analyte(s) detected ≥ RL	B	The specific analyte(s) in all samples in the associated AAB
Equipment Blank	Analyte(s) detected ≥ 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 R 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	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

	Criteria	Flag*
Quantitation	≤ MDL	U
	> MDL < RL	F
	≥ RL	as needed

* 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 8.2-3. Flagging Conventions Specific to Organic Methods

QC Requirement	Criteria	Flag	Flag Applied To
Ambient Blank (VOC samples only)	Analyte(s) detected ≥ RL	B	The specific analyte(s) in all samples with the same matrix and sampling date
Trip Blank (VOC samples only)	Analyte(s) detected ≥ 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	R	The specific analyte(s) in all samples associated with the initial calibration
Initial Five Point Calibration (GC/MS methods)	SPCC or CCC criteria not met	R	All analytes in all samples associated with the initial calibration
	Linearity criterion not met	R	The specific analyte(s) in all samples associated with the initial calibration
Second Source Calibration Verification	CL exceeded	R	The specific analyte(s) in all samples associated with the second source calibration verification
Initial Daily Calibration Verification (GC & HPLC methods)	CL exceeded	R	The specific analyte(s) in all samples associated with the initial calibration verification
Calibration Verification (GC/MS methods)	SPCC or CCC criteria not met	R	All analytes in all samples associated with the calibration verification
	CL exceeded	R	The specific analyte(s) in all samples associated with the calibration verification
Calibration Verification (GC & HPLC methods)	CL exceeded	R	The specific analyte(s) in the sample associated with the continuing calibration verification
Retention time	Retention time of analyte outside of established retention time window	R	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 R for the nondetects R 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 8.2-3. Concluded

QC Requirement	Criteria	Flag	Flag Applied To
Second Column/Second Detector Confirmation (GC & HPLC methods)	Not performed Agreement between results not within ±40%	R J	All analytes ≥RL All affected analytes
Internal Standard	Retention time not within ±30 seconds: EICP area not within -50% to +100% of last calibration verification	R	Apply R to all results for specific analytes associated with the IS
Lowest Calibration Standard	At or below RL in Initial Calibration	R	All results below the lowest calibration standard used
Tentatively Identified Compounds (TICs)		T	All TICs

Table 8.2-4. Flagging Conventions Specific to Inorganic Methods

QC Requirement	Criteria	Flag	Flag Applied To
Initial multipoint calibration	Correlation coefficient < 0.995	R	All results for specific analyte(s) for all samples associated with the initial calibration
Initial calibration verification/second source standard	CL exceeded	R	All results for specific analyte(s) for all samples associated with the calibration verification
Calibration blank	Analyte detected ≥ RL	B	All results for specific analyte(s) in all samples associated with the blank
Calibration verification (Instrument Check Standard)	CL exceeded	R	All results for specific analyte(s) in all samples since the last acceptable calibration verification
Interference check solution (ICS)	CL exceeded	R	All results for specific analyte(s) in all samples associated with the ICS
Dilution test	CL exceeded	J	Apply to all sample results if the new matrix check was not run or RPD ≥10%
Recovery test (GFAA methods)	CL exceeded	J	All samples in digestion batch if method of standard addition is not performed
Post digestion spike addition (ICP method)	CL exceeded	J	All sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
	% R < 10%	R	
Method of standard addition (GFAA methods)	Method of standard addition not done OR method of standard addition spike levels inappropriate OR correlation coefficient < 0.995	J	All positive sample results for specific analyte for all samples associated with the digestion batch

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

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 ERPIMS ELECTRONIC DATA REPORTS

The prime contractor shall provide an electronic deliverable report in the Environmental Restoration Program Information Management System (ERPIMS) format as specified by the SOW for the project.

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 RECORDKEEPING

The laboratory shall maintain electronic and hardcopy records sufficient to recreate each analytical event conducted pursuant to the SOW. 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) IS results, (5) surrogate spiking records and results (as applicable), (6) spike and spike duplicate records and results, (7) laboratory records, (8) raw data,

including instrument printouts, bench work sheets, and/or chromatograms with compound identification and quantitation reports, (9) corrective action reports, (10) other method and project required QC samples and results, and (11) laboratory-specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples.

8.8 HARDCOPY DATA REPORTS FOR SCREENING AND DEFINITIVE DATA

The hardcopy data reports shall conform to the formats identified in this section.

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-4, I-5, I-6, I-7, I-8 and I-9 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 or O-3A, O-4, O-5 or O-5A, O-6, O-7, O-8, O-9 and O-10 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/MS analyses, forms O-3A and O-5A shall be used and form O-11 shall be added to the organic report package. Additional exceptions to the forms format are AFCEE approved variances to the standard reporting forms. For this project AFCEE approved forms can be found in Appendix C and can be substituted for the standard forms.

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-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-2, O-2 or W-2). The results of the analysis of the diluted sample shall be reported with the dilution noted on the report form and the MDL and RL adjusted for 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)

Field Sample ID: enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

FORM I-1 (continued)

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 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., mg/L or 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)

Qualifier: 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 96)

Initial Calibration ID: enter the unique identifying number given to the initial calibration event

Concentration Units: enter the appropriate units (i.e., mg/L or mg/kg)

Analyte: enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

RF1, RF2, RF3: enter the response factor corresponding to the standard with the same number

Std 1, Std2, Std3: enter the concentration of the standard

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 (Mercury analyses only)

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 96)

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)

RF1, RF2, RF3, RF4, RF5: enter the response factor corresponding to the standard with the same number

Std 1, Std 2, Std 3, Std 4, Std 5: enter the concentration of the standard

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 (Cyanide analyses only)

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 96)

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)

RF1, RF2, RF3, RF4, RF5, RF6: enter the response factor corresponding to the standard with the same number

Std 1, Std 2, Std 3, Std 4, Std 5, Std 6: enter the concentration of the standard

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 (continued)

Expected: enter the expected result (i.e., the concentration of the calibration material).

Found: enter the measured result.

%D: enter the per cent 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., 2S960603)

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., CCV960603-1)

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., CCV960603-2)

Concentration Units: enter the appropriate units (i.e., mg/L or 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, Found 2: enter the measured result. Found 1 corresponds to the first CCV run, Found 2 corresponds to the second CCV run, etc.

FORM I-4 (continued)

%D: enter the per cent 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#: 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., mg/L or 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., CB960603)

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., MB960603)

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 analysis) enter the identification number for the first CCB (the same ID number will be found in the run sequence log, e.g., CCB960603-1)

CCB #2 ID: (used for 6010B analysis) enter the identification number for the second CCB (the same ID number will be found in the run sequence log, e.g., CCB960603-2)

CCB #3 ID: (used for 6010B analysis) enter the identification number for the third CCB (the same ID number will be found in the run sequence log, e.g., CCB960603-3)

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

FORM I-5 (continued)

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-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., LCS960603)

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 (i.e., mg/L or 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 per cent recovery

Control Limits: enter the control limits 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-7

Concentration Units: enter the appropriate units (i.e., mg/L or 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., MS960603)

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., MSD960603)

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 per cent recovery

Duplicate Spiked Sample Result: enter the numeric result of the MSD

%RPD: enter the relative per cent difference between the spike (MS) and spike duplicate (MSD)

Control Limits %R: enter the control limits required to be met (see QAPP Section 7)

Control Limits %RPD: enter the control limits required to be met (see QAPP Section 7)

Q: enter the qualifier flag as needed (see QAPP Sections 7 and 8)

FORM I-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 96)

Date Received: enter the date the sample was received at the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

Date Analyzed: enter the date the sample was analyzed by the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

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-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 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., 6 Jun 96)

FORM I-9 (continued)

Time Analysis Completed: enter the time the sample analysis was completed in 24-hour format (e.g., 0900, 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)

FORM O-2 (continued)

Concentration Units: enter the appropriate units (i.e., µg/L or 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 per cent recovery of the surrogate

Control Limits: enter the control limits 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 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

FORM O-3 and 3A (continued)

Concentration Units: enter the appropriate units (i.e., µg/L or 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 non-linear 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 non-linear calibrations)

%RSD: enter the per cent 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

FORM O-4 (continued)

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 per cent 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)

FORM O-5 and O-5A (continued)

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 per cent difference

% D or % drift: (form O-5) enter the per cent 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

FORM O-6

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., µg/L or 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)

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 per cent recovery of the surrogate

FORM Q-7

Control Limits: enter the control limits 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., µg/L or 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 per cent recovery

Control Limits: enter the control limits 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 per cent 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., µg/L or 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 per cent recovery

Duplicate Spiked Sample Result: enter the numeric result of the MSD

%RPD: enter the relative per cent difference between the spike (MS) and spike duplicate (MSD)

Control Limits %R: enter the control limits required to be met (see QAPP Section 7)

Control Limits %RPD: enter the control limits 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)

FORM O-10 (continued)

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 per cent 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 W-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 W-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., mg/L or mg/kg dry weight)

Analyte: enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

FORM W-2 (continued)

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)

Qualifier: 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 96)

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)

RF1, RF2, RF3: enter the response factor corresponding to the standard with the same number

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., 2S960603)

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)

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., CCV960603-1)

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., CCV960603-2)

Concentration Units: enter the appropriate units (i.e., mg/L or 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 per cent 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 (i.e., mg/L or 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., CB960603)

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., MB960603)

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., LCS960603)

FORM W-6 (continued)

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 (i.e., mg/L or 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 per cent recovery

Control Limits: enter the control limits 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., MS960603)

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., MSD960603)

Concentration Units: enter the appropriate units (i.e., mg/L or mg/kg)

Analyte: enter the name of the analytes (use the same name as used in the tables in Section 7 of the QAPP)

FORM W-7 (continued)

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 per cent recovery

Duplicate Spiked Sample Result: enter the numeric result of the MSD

%RPD: enter the relative per cent difference between the spike (MS) and spike duplicate (MSD)

Control Limits %R: enter the control limits required to be met (see QAPP Section 7)

Control Limits %RPD: enter the control limits 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 96)

Date Received: enter the date the sample was received at the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

Date Analyzed: enter the date the sample was analyzed by the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

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

FORM W-8 (continued)

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 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., 6 Jun 96)

Time Analysis Completed: enter the time the sample analysis was completed in 24-hour format (e.g., 0900, 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 96)

FORM S-1 (continued)

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, soil)

Date Analyzed: enter the appropriate dates in the format DD-MMM-YY (e.g., 3 Jun 96)

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

Qualifier: 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 per cent or difference relative per cent 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, 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 96)

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 seven replicates

MDL: enter the calculated MDL

CHAIN OF CUSTODY FORM

COC#: enter a unique number for each chain of custody form

Ship to: enter the laboratory name and address

Carrier: enter the name of the transporter (e.g., FedEx) or handcarried

Airbill#: enter the airbill number or transporter tracking number (if applicable)

Project Name: enter the project name (e.g., Banks AFB RI/FS)

Sampler Name: enter the name of the person collecting the samples

Sampler Signature: signature of the person collecting the samples

Send Results to: enter the name and address of the prime contractor

Field Sample ID: enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)

CHAIN OF CUSTODY FORM (continued)

Date: enter the year and date the sample was collected in the format M/D (e.g., 6/3)

Time: enter the time the sample was collected in 24-hour format (e.g., 0900)

Matrix: enter the sample matrix (e.g., water, soil)

Pres: enter the preservative used (e.g., HNO3) or "none"

Filtered/Unfilt.: enter "F" if the sample was filtered or "U" if the sample was not filtered

# of Containers: enter the number of containers (i.e., jars, bottles) associated with the sample

MS/MSD: enter "X" if the sample is designated the MD/MSD

Analyses Requested: enter the method name of the analysis requested (e.g., SW6010B)

Comments: enter comments

Sample Condition Upon Receipt at Laboratory: enter any problems with the condition of any sample(s)

Cooler Temperature: enter the internal temperature of the cooler, upon opening, in degrees C

Special Instructions/Comments: enter any special instructions or comments

Released by: (SIG): enter the signature of the person releasing custody of the samples

Company Name: enter the company name employing the person releasing/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., 6/3/96) when the samples were released/ received

Time: enter the time in 24-hour format (e.g., 0900) when the samples were released/received

AFCEE
INORGANIC 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 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:

_____

_____

_____

_____

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 (mg/L or mg/kg): _____

Analyte	Std 1	RF 1	Std 2	RF 2	Std 3	RF 3	r	Q

r = correlation coefficient

Comments:

_____

_____

_____

AFCEE FORM I-3

AFCEE
INORGANIC ANALYSES DATA SHEET 3
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	RF 1	Std 2	RF 2	Std 3	RF 3	Std 4	RF 4	Std 5	RF 5	r	Q
Mercury												

r = correlation coefficient

Comments:

_____

_____

_____

AFCEE FORM I-3A
AFCEE
INORGANIC ANALYSES DATA SHEET 3
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	RF 1	Std 2	RF 2	Std 3	RF 3	Std 4	RF 4	Std 5	RF 5	Std 6	RF 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 (mg/L or mg/kg): _____

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
BLANKS

Analytical Method: _____ AAB #: _____

Lab Name: _____ Contract #: _____

Concentration Units (mg/L or mg/kg): _____

Initial Calibration Blank ID: _____ Initial Calibration ID: _____

CCB #1 ID: _____ CCB #2 ID: _____ CCB #3 ID: _____

Method Blank ID: _____ Initial Calibration ID: _____

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

Comments:

_____

_____

AFCEE
INORGANIC 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	Control Limits	Q

Comments:

_____

_____

_____

AFCEE FORM I-6

AFCEE
INORGANIC ANALYSES DATA SHEET 7
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLE RECOVERY

Analytical Method: _____

Lab Name: _____ Contract #: _____

Concentration Units (mg/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	Control Limits %R	Control Limits %RPD	Q

Comments:

_____

_____

_____

AFCEE
INORGANIC 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
INORGANIC ANALYSES DATA SHEET 9
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 FORM O-1

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	Control Limits	Qualifier

Internal Std	Qualifier

Comments:

_____
_____
_____
_____

AFCEE
ORGANIC ANALYSES DATA SHEET 3A
INITIAL MULTIPOINT CALIBRATION-GC/MS 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
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 3A
INITIAL MULTIPOINT CALIBRATION-GC/MS 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
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 3
INITIAL MULTIPOINT CALIBRATION-GC/MS 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 MULTIPOINT CALIBRATION-GC/MS 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:

_____

_____

_____

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/MS 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		
	RF	% D	RF	% D	RF	% D	Q
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	Control Limits	Qualifier

Internal Std	Qualifier

Comments:

_____

_____

_____

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	Control Limits	Q

Surrogate	**Recovery**	**Control Limits**	**Qualifier**

Internal Std	**Qualifier**

Comments:

_____

_____

_____

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	Control Limits %R	Control Limits %RPD	Q

Comments:
_____
_____
_____

AFCEE FORM O-8 Page ___ of _____

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:

_____

_____

_____

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 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 FORM W-1

AFCEE
WET CHEM ANALYSES DATA SHEET 2
RESULTS

Analytical 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:

_____

_____

_____

_____

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	RF 1	Std 2	RF 2	Std 3	RF 3	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 FORM W-4

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	Control Limits	Q

Comments:

_____

_____

_____

AFCEE FORM W-6

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	Control Limits %R	Control Limits %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: _____

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:

_____

_____

_____

AFCEE FORM S-3 Page ___ of _____

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		

MDL FORM Method ___________ Page ____ of ____

AFCEE
CHAIN OF CUSTODY RECORD

COC#: _____

Ship to:	Project Name:	Send Results to:
	Sampler Name:	
Carrier: Airbill #:	Sampler Signature:	

Field Sample ID	Date 19__	Time	Matrix	Pres	Filtered /Unfilt.	# of Containers	MS/ MSD	Analyses Requested											Comments

Sample Condition Upon Receipt at Laboratory:	Cooler temperature:
Special Instructions/Comments:	

#1 Released by: (Sig)	Date:	#2 Released by: (Sig)	Date:	#3 Released by: (Sig)	Date:
Company Name:	Time	Company Name:	Time	Company Name:	Time
#1 Received by: (Sig)	Date	#2 Received by: (Sig)	Date	#3 Received by: (Sig)	Date
Company Name:	Time:	Company Name:	Time:	Company Name:	Time:

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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 validation is also a quantitative check of the analytical process, where all documentation and calculations are evaluated and verified. Data validation is 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 shall be reviewed by the prime contractor to determine whether data produced by the analytical contractor shall fulfill the objectives of the program.

Audit findings shall be transmitted form 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 correct the deficiencies identified during the state/federal audits(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 activity is being performed in compliance with the Sampling and Analysis Plan (SAP) specifications. Sampling and field procedures, and the 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 are outstanding, 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 outstanding corrective actions have been addressed.

Critical items for a laboratory or field 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 a sampling systems audit include: (1) calibration procedures and documentation for field equipment, (2) documentation in field logbooks and sampling data sheets, (3) organization and minimization of potential contamination sources while in the field, (4) proper sample collection, storage, and transportation procedures, and (5) 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 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.

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 performance evaluation (PE) samples for analysis for each analytical method used in the project. The prime contractor shall submit project specific PE samples once per quarter per project. The project-specific PE 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 control limits and whether the data produced meet the analytical QA specifications.

The project-specific PE 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 PE results include: (1) correct identification and quantitation of the PE sample analytes, (2) accurate and complete reporting of the results, and (3) measurement system operation within established control limits for precision and accuracy.

The concentrations reported for the PE 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. If the accuracy criteria are not met, the cause of the discrepancy shall be investigated and a second PE 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 up to date regarding corrective actions and subsequent PE 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 assess 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 PE results, on-site audit results, or by other state/federal investigations.

9.1.5 Performance Evaluation Sample Programs

All laboratories shall participate in the U.S. EPA PE Water Supply and Water Pollution Studies programs or equivalent programs for state certifications. Satisfactory performance in these nonproject-specific PE programs also demonstrate proficiency in methods used to analyze AFCEE samples. The laboratory shall document the corrective actions to unacceptable PE results to demonstrate resolution of the problems.

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.

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10.0 PREVENTIVE 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 Recommendations for Corrective Action (RCA) exists for addressing significant and systematic problems. Recommendations for corrective actions 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.

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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.

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Table B

AFCEE QAPP
ANALYTICAL VARIANCE REQUEST

ITEM	E & E VARIANCE
General—LCS criteria. Pertinent "Summary of Calibration and QC Procedures..." tables in Section 7.2.	E & E proposes a variance to the requirement that an LCS failure be automatically followed by the repreparation of all affected samples. When the LCS is high and the sample does not contain the affected compound(s), then the result should be considered fully usable.
General— Standard Materials. Section 4.5.3 Standard materials used in calibration shall be traceable to NIST, EPA, or A2LA.	E & E will use the following vendors for this project: Supleco, Restek, Accustandard, Absolute Standards, Ultra Scientific, and Chem Service. All vendors are ISO9000-registered and provide certified reference materials.
General—Dilutions. AFCEE QAPP Section 8.8 implies a requirement that all samples should be analyzed undiluted.	E & E proposes a variance to allow all samples to be run at a dilution suitable for analysis (i.e., such that all components are within the calibration range). If required to satisfy project objectives and at the specific request of the prime contractor, the sample will then be run a further dilution at 10 times more concentrated than the first analysis. Every effort will be made to run samples undiluted, but some samples are so contaminated that undiluted analysis will cause instruments to fail.
General—MDL spiking levels. AFCEE QAPP Section 4.3.1 requires the analysis of seven samples containing analytes at a concentration 3 to 5 times the estimated MDL.	E & E proposes that 40 CFR Part 136 Appendix B be followed: namely, that the spiking level considered for use be obtained from an estimate that is 1 to 5 times the *estimated* MDL and that criteria for acceptance of the *determined* MDL is that it be ≥ 1/10 the spiking level used.
AFCEE Reporting Forms. AFCEE QAPP Section 8.8. Analysis sequence log Form I-9, O-10, W-9.	These forms will include one column for date of analysis and one column for time of analysis, instead of separate start and completion dates and times. For GCMS, E & E proposes combining Forms O-10 and O-11. For all methods, E & E proposed Lab ID to be substituted for Field ID. We currently do not have separate fields in our database to store this information. To produce the specified report, our database would have to be modified and more manual entry would be required for each sample.
AFCEE Reporting Forms. AFCEE QAPP Section 8. Inorganic forms I-4 and I-5.	E & E proposes splitting these two forms into separate forms for different calibrations and blanks. Our current reporting system will not allow us to combine this information on a single form without having to perform manual entry for each analytical batch.

Table B (Cont.)

ITEM	E&E VARIANCE
AFCEE Reporting Forms. AFCEE QAPP Section 7 and 8. Reporting order in QAPP is alphabetical.	E & E proposes to report all compounds in elution order so that they can be easily reviewed and entered from the raw data.
AFCEE Reporting Forms. AFCEE QAPP Section 7 and 8.2. Reporting units.	E & E proposes to report all organic soil units in ug/kg instead of mg/kg and metal waters in ug/L instead of mg/L. If E &E uses the higher reporting limits and the AFCEE reporting rules in Section 8.2, many MDLs will be reported as zero instead of the actual value.

Table C

AFCEE QAPP
VARIANCES ON LABORATORY LIMITS

E & E has reviewed the AFCEE 1998 QAPP 3.0 target compounds, reporting limits (RL) and QC Acceptance Criteria. The ASC will meet the AFCEE 1998 QAPP 3.0 requirements for all analyses, with the exceptions listed below:

9056/LCS/Bromide	Water	85-121	86-112	85-121
9056/LCS/Chloride	Water	86-121	91-111	90-115
9056/LCS/Fluoride	Water	84-117	86-114	84-117
9056/LCS/Nitrate	Water	88-109	90-110	88-109
9056/LCS/Nitrite	Water	85-114	88-116	85-114
9056/LCS/Phosphate	Water	78-118	87-110	82-112
9056/LCS/Sulfate	Water	78-116	88-115	82-116
9056/MS/MSD/Bromide	Water	85-121	86-112	85-121
9056/MS/MSD/Chloride	Water	86-171	91-111	90-115
9056/MS/MSD/Fluoride	Water	84-117	86-114	84-117
9056/MS/MSD/Nitrate	Water	88-109	90-110	88-109
9056/MS/MSD/Nitrite	Water	85-114	88-116	85-114
9056/MS/MSD/Phosphate	Water	78-118	87-110	82-112
9056/MS/MSD/Sulfate	Water	78-116	88-115	82-116
9056/MS/MSD/Fluoride	Water	27	20	27
9056/RPD/Phosphate	Water	24	20	24

8260/Methylene Chloride	Water	0.005mg/L	0.002mg/L

Ecology and Environment Inc.

Quality Control Limits, wet chemistry tests.

TEST	PQL	TYPE	PARAMETER	LCL	UCL	RPD
Alkalinity	1 mg/L	DUP	Alkalinity			10.0
		LCS	Alkalinity	90.0	110	
		MS	Alkalinity	85.0	115	
Ammonia – W	0.2 mg/L	DUP	Ammonia-Distilled			15.0
		LCS	Ammonia-Distilled	83.0	117	
		MS	Ammonia-Distilled	82.2	114	
COD - W	5 mg/L	DUP	COD			15.0
		LCS	COD	80.0	120	
		MS	COD	75.0	125	
Color	< 5 ntu	DUP	Color			10.0
Cyanide TOT 9010B - W	0.01 mg/L	DUP	Cyanide Total			20.0
		LCS	Cyanide Total	80.0	120	
		MS	Cyanide Total	75.0	125	
Hardness - W	4 mg/L	DUP	Hardness			10.0
		LCS	Hardness	90.0	110	
		MS	Hardness	90.0	110	
TOC - W	1.0 mg/L	DUP	TOC			20.0
		LCS	TOC	80.0	120	
		MS	TOC	75.0	125	
Total Dissolved Solids	10 mg/L	DUP	Total Dissolved Solids			10.0
		LCS	Total Dissolved Solids	80.0	120	

AOC LTM BASELINE STUDY
GRIFFISS AFB, NY
AFCEE QAPP 3.0
REVISED ANALYTICAL VARIANCE REQUEST

1. E & E proposes a variance to the requirement that an LCS failure be automatically followed by the reanalysis of all affected samples. Low LCS results will prompt corrective action as outlined in each analytical table (QAPP Section 7). When an analyte is high in an LCS, corrective action will be initiated. Corrective action will include an investigation into the causes of the high LCS result and will include reanalysis of all samples which showed positive concentrations of the analyte in question. Samples showing non-detect for the analyte will not be reanalyzed. Note that this corresponds to the flagging criteria in each analytical table (QAPP Section 7) and the general flagging conventions in Table 8.2-2.

2. E & E's understanding is that the concept of traceability requires that standards used to calibrate a process must be analyzed against standards prepared by the ultimate agency -- EPA, NIST or A2LA. This can be direct (i.e., a one to one comparison) or indirect (i.e., through one or more intermediates, each of which has been tested against a standard which is itself directly or indirectly traceable to the agency. EPA and A2LA do not prepare standards so traceability to these agencies is not possible. NIST prepares and sells standards which correspond to a small fraction of the analytes of interest, and for the majority of analytes, traceability is not possible. E & E's approach has been to use standard vendors whose product preparation processes have been approved by a third party agency (e.g., ISO 9000), and a system of second source verification procedures. These vendors' products are used throughout the environmental testing industry. All vendors used by E & E use NIST traceable weights and volumetric glassware for standard preparation.

3. Original variance is withdrawn as per AFCEE's interpretation of the QAPP.

4. E & E's procedure for determining method detection limits is that promulgated in 40CFR Part 136, Appendix B. These procedures are acceptable to USEPA and the New York State Department of Environmental Conservation (NYSDEC) and will allow project data quality objectives (DQOs) to be met. For E & E to determine MDLs separately for AFCEE using a non standard technique will add significantly to E & E's costs.

5. Modified forms have been sent separately by fax.

6. Modified forms have been sent separately by fax.

7. Original variance is withdrawn. The reporting order of analytes will be as per the QAPP.

8. This variance is not applicable to the LTM project since only water samples are scheduled to be sampled. If soil samples are taken, the reporting units will be mg/kg, however a variance from the QAPP Section 8.2 requiring that MDLs and results to be reported to one decimal place more than the corresponding RL will be increased to as many decimal places necessary to take into account rounding effects.

9. AFCEE reporting limit (RL) by method 8260B for methylene chloride in water is 0.3 ug/L. If blank contamination is found above this level, the corrective action required by the QAPP is to reanalyze all affected samples. E & E proposes a RL of 5 ug/L, which is lower than the NYSDEC Part 360 recommended PQL of 10 ug/L by method 8260. Methylene chloride is a ubiquitous laboratory contaminant, and while E & E has been very successful in minimizing it through good laboratory design and analytical practice, we prefer to commit to a slightly higher RL. It's E & E understanding is that methylene chloride is not an analyte of concern at most of the sites at Griffiss.

10. E & E proposes to use the QC limits for the Anions analysis, method 9056, as previously presented in Table C of the original variance submission. The limits vary slightly from the QAPP 3.0 limits and will not adversely impact the DQO of the LTM project.

QA CHART SUMMARY

Test	Type	Parameter	LCL	UCL	RPD	Last Update
AATEST	LCS	AATEST	87.92	124.88		1/29/98
ACIDITY	DUP	ACIDITY			10.00	3/9/98
	LCS	ACIDITY	90.00	110.00		3/9/98
ACR6	DUP	CR VI			20.00	3/2/98
	LCS	CR VI	85.00	115.00		2/5/98
ALKALINITY	DUP	ALKALINITY			10.00	3/9/98
	LCS	ALKALINITY	90.00	110.00		3/9/98
	MS	ALKALINITY	85.00	115.00		2/5/98
AMMONIA - S	DUP	AMMONIA - DISTILLED			15.00	2/5/98
	MS	AMMONIA - DISTILLED	85.00	115.00		3/9/98
AMMONIA - W	DUP	AMMONIA - DISTILLED			15.00	2/5/98
	LCS	AMMONIA - DISTILLED	83.00	117.00		3/9/98
	MS	AMMONIA - DISTILLED	82.16	113.60		1/29/98
AMMONIA -nessler	DUP	AMMONIA - DISTILLED			30.00	3/31/98
	LCS	AMMONIA - DISTILLED	70.00	130.00		3/31/98
	MS	AMMONIA - DISTILLED	70.00	130.00		3/31/98
AOX - W	LCS	AOX	85.00	115.00		2/5/98
	MS	AOX	80.02	119.98		3/2/98
ASH	DUP	ASH			10.00	3/9/98
BOD5 - W	LCS	BOD5	88.00	126.00		3/11/98
BROMIDE - W	DUP	BROMIDE			30.00	3/31/98
	LCS	BROMIDE	85.00	115.00		3/9/98
BROMIDE-W	MS	BROMIDE	85.00	115.00		3/9/98
BTU - S	DUP	BTU			20.01	3/9/98
BTU - SW	LCS	BTU	85.00	115.00		3/9/98
BTU - W	DUP	BTU			19.98	3/2/98
BULK DENSITY		BULK DENSITY			9.99	2/5/98
CHLORIDE - S		CHLORIDE			30.00	6/9/98
	MS	CHLORIDE	79.99	120.01		3/11/98
CHLORIDE - W	DUP	CHLORIDE			15.00	2/5/98
	LCS	CHLORIDE	85.00	115.00		2/5/98
	MS	CHLORIDE	79.99	120.01		3/11/98
CHLORINE - W	DUP	RESIDUAL			30.00	6/9/98
CHLORINE DEMAND - W		CHLORINE DEMAND			15.00	2/5/98
COD - W	Dup	COD			15.00	2/5/98
	LCS	COD	79.99	120.01		3/11/98
	MS	COD	75.01	124.99		3/9/98
COD 120-W	LCS	COD	70.00	130.00		3/18/98
COD 1200-W		COD	70.00	130.00		3/18/98
COD 30-W		COD	70.00	130.00		3/18/98

Test	Type	Parameter	LCL	UCL	RPD	Last Update
COD 30-W	MS	COD	70.00	130.00		3/19/98
COD 300-W	LCS	COD	70.00	130.00		3/18/98
	MS	COD	70.00	130.00		3/19/98
COLOR - W	DUP	COLOR			10.00	3/9/98
CONDUCTIVITY - W		CONDUCTIVITY			10.00	3/9/98
	LCS	CONDUCTIVITY	90.00	110.00		3/9/98
CR6 - W	DUP	CR VI			30.00	3/19/98
	LCS	CR VI	87.20	110.00		2/5/98
	MS	CR VI	80.02	119.98		3/2/98
CR6 7196A - W	DUP	CR VI			15.00	3/9/98
	LCS	CR VI	81.15	114.45		3/9/98
	MS	CR VI	70.00	130.00		3/2/98
CR6-S	DUP	CR VI			30.00	3/19/98
	LCS	CR VI	85.00	115.00		3/10/98
	MS	CR6	70.00	130.00		3/2/98
CYANIDE AMINABLE - S	LCS	CYANIDE AMINABLE	79.97	120.03		3/11/98
CYANIDE AMINABLE - W		CYANIDE AMINABLE	79.99	120.01		3/10/98
CYANIDE RELEASABLE -W	DUP	CYANIDE RELEASABLE			19.98	3/2/98
	LCS	CYANIDE RELEASABLE	80.02	119.98		3/2/98
CYANIDE RELEASABLE-REF		CYANIDE RELEASABLE	70.00	130.00		3/2/98
CYANIDE TOT 335.2-W		CYANIDE TOTAL	79.97	120.03		3/11/98
	MS	CYANIDE TOTAL	75.00	125.01		3/10/98
CYANIDE TOT 335.4-W	DUP	CYANIDE TOTAL			20.03	3/11/98
	LCS	CYANIDE TOTAL	79.97	120.03		3/11/98
	MS	CYANIDE TOTAL	75.00	125.01		3/11/98
CYANIDE TOT 9010B-W	DUP	CYANIDE TOTAL			20.03	3/11/98
	LCS	CYANIDE TOTAL	79.97	120.03		3/11/98
	MS	CYANIDE TOTAL	75.00	125.01		3/11/98
CYANIDE TOT 9013/9010B-S	DUP	CYANIDE TOTAL			30.00	2/2/98
	LCS	CYANIDE TOTAL	70.00	130.00		2/2/98
	MS	CYANIDE TOTAL	70.00	130.00		2/2/98
CYANIDE TOT CLP-S	DUP	CYANIDE TOTAL			20.03	3/11/98
	LCS	CYANIDE TOTAL	79.99	120.01		3/13/98
	MS	CYANIDE TOTAL	75.00	125.01		3/10/98
CYANIDE TOT CLP-W	DUP	CYANIDE TOTAL			20.03	3/11/98
	LCS	CYANIDE TOTAL	79.97	120.03		3/11/98
	MS	CYANIDE TOTAL	75.00	125.01		3/11/98
CYANIDE TOT MIDI 9012A-S	DUP	CYANIDE TOTAL			20.00	3/30/98
	LCS	CYANIDE TOTAL	85.00	115.00		3/30/98
	MS	CYANIDE TOTAL	93.01	109.99		2/5/98
CYANIDE TOT MIDI 9012A-W	DUP	CYANIDE TOTAL			20.03	3/11/98
	LCS	CYANIDE TOTAL	85.00	115.00		3/30/98
	MS	CYANIDE TOTAL	75.01	124.99		2/5/98
FILTER WEIGHT	DUP	FILTER WEIGHT			10.00	3/10/98

Test	Type	Parameter	LCL	UCL	RPD	Last Update
FLASHPOINT - L	DUP	FLASHPOINT			10.00	3/10/98
	LCS	FLASHPOINT	80.00	120.00		3/10/98
FLASHPOINT - S	DUP	FLASHPOINT			9.99	3/10/98
	LCS	FLASHPOINT	95.04	107.58		1/29/98
FLASHPOINT - SW		FLASHPOINT	70.00	130.00		3/2/98
FLASHPOINT - W	DUP	FLASHPOINT			9.99	3/10/98
FLUORIDE - S		FLUORIDE			20.00	3/10/98
	LCS	FLUORIDE	79.97	120.03		3/11/98
FLUORIDE - W	DUP	FLUORIDE			15.00	3/10/98
	LCS	FLUORIDE	85.00	115.00		3/10/98
	MS	FLUORIDE	85.00	115.00		3/10/98
FORMALDEHYDE - W	DUP	FORMALDEHYDE			20.00	3/10/98
GLYCOL - W		GLYCOL			20.00	3/10/98
	LCS	GLYCOL	79.97	120.03		3/11/98
HARDNESS - W	DUP	HARDNESS			10.00	3/10/98
	LCS	HARDNESS	90.00	110.00		3/10/98
	MS	HARDNESS	90.00	110.00		3/10/98
LBTU	DUP	BTU			20.03	3/11/98
LSULFD		LSULFD			20.03	3/11/98
MBAS - W		MBAS			20.00	3/10/98
	LCS	MBAS	79.99	120.01		3/10/98
	MS	MBAS	70.00	130.00		3/31/98
NITRATE - S	DUP	NITRATE			20.01	3/10/98
	LCS	NITRATE	85.00	115.00		3/10/98
	MS	NITRATE	80.00	120.00		3/10/98
NITRATE - W	DUP	NITRATE			15.00	3/10/98
	LCS	NITRATE	85.00	120.70		3/10/98
	MS	NITRATE	79.99	120.01		3/10/98
NITRITE - S	LCS	NITRITE	80.00	120.00		2/5/98
NITRITE - W	DUP	NITRITE			15.00	2/5/98
	LCS	NITRITE	85.00	120.00		3/10/98
	MS	NITRITE	79.99	120.01		3/10/98
OIL AND GREASE - S	DUP	OIL AND GREASE			20.00	2/5/98
OIL AND GREASE - W	LCS	OIL AND GREASE	55.34	121.04		9/8/98
	MS	OIL AND GREASE	75.00	125.01		3/11/98
ORGANIC CHLORIDE - S	DUP	ORGANIC CHLORIDE			20.02	1/29/98
ORGANIC CHLORINE - L		ORGANIC CHLORINE			19.98	3/2/98
ORGANIC CHLORINE - W	LCS	ORGANIC CHLORINE	79.97	120.03		3/11/98
ORGANIC MATTER	DUP	ORGANIC MATTER			20.00	3/10/98
ORGANIC MATTER - S		ORGANIC MATTER			20.01	3/13/98
pH - L		pH			10.00	3/11/98
pH - S		pH			10.00	3/10/98
pH - W		pH			10.00	3/10/98
PHENOL - S		PHENOL			20.00	3/26/98

Test	Type	Parameter	LCL	UCL	RPD	Last Update
PHENOL - S	LCS	PHENOL	70.00	130.00		3/2/98
	MS	PHENOL	85.00	115.00		2/5/98
PHENOLS - W	DUP	PHENOLS			15.00	3/11/98
	LCS	PHENOLS	72.69	117.33		3/11/98
	MS	PHENOLS	70.00	130.00		3/11/98
PHOSPHATE ORTHO - S	DUP	PHOSPHATE ORTHO			20.01	3/11/98
PHOSPHATE ORTHO - W		PHOSPHATE ORTHO			20.01	3/11/98
	LCS	PHOSPHATE ORTHO	85.00	115.00		3/11/98
	MS	PHOSPHATE ORTHO	85.00	115.00		3/11/98
PHOSPHATE TOTAL - S	DUP	PHOSPHATE TOTAL			20.00	2/5/98
PHOSPHATE TOTAL - W		PHOSPHATE TOTAL			20.01	3/11/98
	LCS	PHOSPHATE TOTAL	85.00	115.00		3/11/98
	MS	PHOSPHATE TOTAL	79.97	120.03		3/11/98
RSULRA 1001	LCS	RSULRA 1001	70.00	130.00		3/2/98
SULFATE - W	DUP	SULFATE			20.01	3/11/98
	LCS	SULFATE	76.81	119.47		3/11/98
	MS	SULFATE	75.00	125.01		3/11/98
SULFIDE - S	DUP	SULFIDE			20.01	3/11/98
SULFIDE - W		SULFIDE			12.85	3/11/98
	LCS	SULFIDE	85.00	115.00		3/11/98
SULFIDE RELEASABLE - S	DUP	SULFIDE			20.00	3/2/98
SULFIDE RELEASABLE - W		SULFIDE RELEASABLE			4.03	1/29/98
	LCS	SULFIDE RELEASABLE	-9.80	77.08		1/29/98
SULFIDE RELEASABLE-REF		SULFIDE RELEASABLE	80.00	120.00		3/2/98
SUSPENDED SOLIDS	DUP	SUSPENDED SOLIDS			20.00	3/2/98
TCIP TRPH		TCIP TRPH			20.00	3/2/98
THIOSULPHATE-REF	LCS	THIOSULPHATE	80.00	120.00		3/2/98
TKN - S	DUP	TKN			15.00	3/11/98
	MS	TKN	85.00	115.00		3/11/98
TKN - W	DUP	TKN			15.00	3/11/98
	MS	TKN	85.00	115.00		3/11/98
TKN-REF	LCS	TKN	85.00	115.00		3/11/98
TOC - S	DUP	TOC			20.00	3/11/98
	LCS	TOC	79.99	120.01		3/11/98
	MS	TOC	60.01	139.99		8/26/98
TOC - W	DUP	TOC			20.01	3/11/98
	LCS	TOC	79.99	120.01		3/11/98
	MS	TOC	75.00	125.01		3/11/98
TOTAL DESOLVED SOLIDS	DUP	TOTAL DESOLVED SOLIDS			10.00	3/11/98
TOTAL DISOLVED SOLIDS		TOTAL DISOLVED SOLIDS			10.00	3/11/98
	LCS	TOTAL DISOLVED SOLIDS	80.00	120.00		3/2/98
TOTAL SOLIDS	DUP	TOTAL SOLIDS			10.00	3/11/98
TOTAL SOLIDS - W		TOTAL SOLIDS			10.00	3/11/98
TOTAL SUSPENDED SOLIDS - W		TOTAL SUSPENDED SOLIDS			10.00	3/11/98

Ecology and Environment Inc.

Quality Control Limits, wet chemistry tests.

TEST	PQL	TYPE	PARAMETER	LCL	UCL	RPD
Alkalinity	1 mg/L	DUP	Alkalinity			10.0
		LCS	Alkalinity	90.0	110	
		MS	Alkalinity	85.0	115	
Ammonia – W	0.2 mg/L	DUP	Ammonia-Distilled			15.0
		LCS	Ammonia-Distilled	83.0	117	
		MS	Ammonia-Distilled	82.2	114	
COD - W	5 mg/L	DUP	COD			15.0
		LCS	COD	80.0	120	
		MS	COD	75.0	125	
Color	< 5 ntu	DUP	Color			10.0
Cyanide TOT 9010B - W	0.01 mg/L	DUP	Cyanide Total			20.0
		LCS	Cyanide Total	80.0	120	
		MS	Cyanide Total	75.0	125	
Hardness - W	4 mg/L	DUP	Hardness			10.0
		LCS	Hardness	90.0	110	
		MS	Hardness	90.0	110	
TOC - W	1.0 mg/L	DUP	TOC			20.0
		LCS	TOC	80.0	120	
		MS	TOC	75.0	125	
Total Dissolved Solids	10 mg/L	DUP	Total Dissolved Solids			10.0
		LCS	Total Dissolved Solids	80.0	120	

TITLE: Chemical Oxygen Demand (COD)			
ORIGINAL AUTHOR	F. Lindauer	REVISION AUTHOR	K. Demarco
IMPLEMENTATION DATE	5/18/1998	ANNUAL REVIEW DATE	5/18/1999
FILE INFORMATION	W:\SOPs\Final\GAC\GAC12.ene-11/30/98 4:29 PM		
REVISION: 2	STATUS: Final	METHOD: SM 5220 C	APPROVAL DATE: 4/14/98

1.0 SCOPE AND APPLICATION

1.1 COD at levels of 5 to 1500 mg/L can be analyzed without dilution. Higher levels require dilutions. Premixed vials of reagents are used for all three levels of COD analysis. The ranges for these levels are as follows:

Low level	< 5 - 150 mg/L
Med level	< 5 - 1500 mg/L
High level	< 50 - 15000 mg/L

Dilutions are only made at high level.

2.0 METHOD SUMMARY

2.1 This SOP describes the procedure to determine the quantity of oxygen required to oxidize all organic and inorganic material present within the sample with potassium dichromate in a sulfuric acid solution. The excess dichromate is titrated with standard ferrous ammonium sulfate using ferroin as an indicator. Method modification: premixed vials of reagents for this analysis are purchased and not made in house.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E & E, Inc. Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 Use Extreme Caution - This test uses sulfuric acid and mercuric salts at high temperatures.

4.0 REFERENCES

4.1 Standard Methods, 18th Edition 1992, Method 5220 C

4.2 HACH COD - Microdigestion Procedure

4.3 Use checklist number C-071 for analyst and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 COD – Chemical Oxygen Demand

5.2 F.A.S. –Ferrous Ammonium Sulfate($Fe(NH_4)_2(SO_4)_2 \bullet 6\ H_2O$).

5.3 Ferroin - Orthophenzntroline Ferrous Complex

5.4 ASC Control Chart Database - Access ™ database used for control charting by ASC. Control limits are tabulated by lab, method, and by matrix is the "Control Limit Summary".

6.0 INTERFERENCES/POTENTIAL PROBLEMS

8.3 For low level FAS Titrant, divide the result of the above equation by ten.

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	NA	28 Days	Poly or Glass H_2SO_4 pH< 2, 2-5°C
Water	NYSDEC	NA	26 Days	Poly or glass, 2-5°C H_2SO_4 pH < 2
REVISION: 2	STATUS: Final	METHOD: SM 5220 C		APPROVAL DATE: 4/14/98

Hold Time

10.0 PROCEDURE

10.1 Standards (analyze 1200 mg/L KHP standard for high and med level vials, and 120 mg/L for low level vials): pipet 2 mL of each standard into the appropriate level vial.
Blanks: pipet 2 mL of ASTM type II water into each level vial used. Prepare in duplicate.

10.2 Pipette 2 mL of sample into a low level vial. If sample turns green, use a medium level vial. If that turns green use a high level vial. If 2 mL of sample turns green using a high level vial, prepare a dilution to get into high level vial range.

10.3 Cap and invert each vial a few times.

10.4 Place vials into block digester at 150 ± 5° C and digest for at least 2 hours.

10.5 If a green color appears after digestion, the titration should be attempted for mid level and high level samples, titrate with 0.125 N FAS. If the titration does not require at least 0.25 mL of titrant, the sample must be redigested using a higher level vial.

10.6 Remove vials, cool, add 2 drops Ferroin, and titrate using the appropriate FAS until the endpoint is reached. For low level samples titrate with 0.0125 N FAS. For med and high level samples use 0.125 N FAS. The endpoint goes from blue-green to orange-brown.

11.0 DATA REDUCTION/EVALUATION/REPORTING

Method Calculations

$$\text{COD mg/L} = \frac{(B - S) \times N \times 8{,}000}{V}$$

where: B = mL of FAS to reach endpoint on the blank (Average of 2 titrations)
S = mL of FAS to reach endpoint on the sample
V = milliliters of sample used for the test
N = FAS Normality

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Water
COD low level and medium level	T	5 mg/L
COD high level	T	50 mg/L

REVISION: 2	STATUS: Final	METHOD: SM 5220 C	APPROVAL DATE: 4/14/98

Key Type:

T = Compound/Analyte is target compound routinely reported.
M= Compound/Analyte is listed in the method but is not routinely reported by E & E.
C = Compound/Analyte is specified by the client and can be analyzed under this method.
S = Compound/Analyte is routinely used as a matrix spike.
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as a surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Reference Standard: 120 mg/L (low level LCS)	1 per batch[1]	Pipette 2.0 mL 120 mg/L KHP into low level vial	Current lab limits	Reanalyze standards and blank, Reprepare Batch
Reference Standard: 1200 mg/L (med & high level LCS)	1 med and/or 1 high per batch[1]	Pipette 2.0 mL 1200 mg/L KHP into med and/or high level vial	Current lab limits	Reanalyze standards and blank, Reprepare Batch
Duplicate (DUP)	1 per batch or 10% (ELAP)	2nd aliquot of sample analyzed	Current lab limits	Reanalyze

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Blank	2 per level per batch	Pipette 2.0 mL ASTM Type II water in appro-priate level vials	NA	NA
Matrix Spike (MS)	1 per batch or 10%	1 mL 300 mg/L + 1 mL sample into medium or high level vial or 1 mL 30 mg/L + 1 mL sample in low level vial.	Current lab limits	Reanalyze Comment

REVISION: 2	STATUS: Final	METHOD: SM 5220 C	APPROVAL DATE: 4/14/98

[1] LCS frequency is 10% for each batch (ELAP).

Control Limits for Routine Quality Control Samples

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

See attached.

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
Duplicate (DUP)	COD	Water	In-House	Annually
LCS	COD	Water	In-House	Annually
Spike (MS)	COD	Water	In-House	Annually

REVISION: 2	STATUS: Final	METHOD: SM 5220 C	APPROVAL DATE: 4/14/98

Table 12-3

ACCEPTANCE CRITERIA FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameter	Recovery (%)	RPD
LCS	COD	See Control Limit Summary, COD	NA
Matrix spike	COD	See Control Limit Summary, COD	NA
Duplicate	COD	NA	See Control Limit Summary, COD

REVISION: 2	STATUS: Final	METHOD: SM 5220 C	APPROVAL DATE: 4/14/98

TITLE: Total Kjeldahl Nitrogen (TKN) By Method 351.3			
ORIGINAL AUTHOR	F. Lindauer	REVISION AUTHOR	K. Demarco
IMPLEMENTATION DATE	5/18/1998	ANNUAL REVIEW DATE	5/18/1999
FILE INFORMATION	\\LMSRV1\LABORATORY\SOPs\Final\GAC\GAC4.ene-11/30/98 4:29 PM		
REVISION: 2	STATUS: Final	METHOD: 351.3	APPROVAL DATE: 5/8/98

1.0 SCOPE AND APPLICATION

This SOP directs the procedure for determining the concentration of organic nitrogen plus ammonia nitrogen, total Kjeldahl Nitrogen (TKN), in drinking water and surface waters, domestic and industrial wastes.

2.0 METHOD SUMMARY

2.1 The applicable range of this method is 0.1 to 20 mg/L TKN. The range may be extended with sample dilution.

2.2 Modification; An autodistillation apparatus (Büchi 316) is used.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E&E, Inc. Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.2 ***Toxic Sulfur Trioxide is released during digestion. Perform in a fume hood.***

4.0 REFERENCES

4.1 EPA Method 351.3.

4.2 Standard Methods 4500-NH_3 B, E, 18th edition, 1992.

4.3 Standard Methods 4500-N_{org}B, 18th edition, 1992.

4.4 Use checklist C-095 for analyst and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 TKN - Total Kjeldahl Nitrogen is the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2 SO_4$, under the conditions of digestion.

5.2 ASC Control Chart Database – Access ™ Database use for control charting by ASC control limits are tabulated by laboratory, method and by matrix in the "Control Limit Summary."

6.0 INTERFERENCES/POTENTIAL PROBLEMS

NA

7.0 INSTRUMENTATION AND EQUIPMENT

Büchi 316 Distillation Unit

Table 8-2

STANDARD AND REAGENT PREPARATION

Stock	Standard Name	____ Amount of Stock Added to ____	Final Concentration
		water.	
Methyl Red Methylene Blue in Ethyl Alcohol	Mixed Indicator	Mix two volumes of 0.2% Methyl Red in 95% Ethyl Alcohol with one volume 0.2% Methylene Blue in 95% Ethyl Alcohol. This solution should be prepared fresh every 30 days.	NA

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8.1 Standardization of 0.02 Normal Sulfuric Acid

Standardize against 40.00 mL 0.05N Na_2CO_3 solution, with about 60 mL water in a beaker by titrating potentiometrically to pH of about 5. Lift out electrodes, rinse into the same beaker and boil gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature, rinse cover glass into beaker, and finish titrating to the pH inflection point. Calculate normality:

$$\text{Normality, N} = \frac{A \bullet B}{53.00 \bullet C}$$

where:

A = g Na_2CO_3 weighed into 1-L flask
B = mL Na_2CO_3 solution taken for titration, and
C = mL acid used

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Holding Time

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
H_2O	Std	NA	28	Concentrated Sulfuric Acid 2-5° C
Soil	Std	NA	28	2-5° C No preserve

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Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment Instrument	Symptom	Operation	Frequency
Glassware	Star cracks, chips, damaged	Do Not Use	Replace as needed
Fumehood	Not Drawing	Notify Supervisor or Lab Manager	As necessary
Büchi 316	Contaminated blank	Clean system with 1:1 HCl	As needed
Büchi 316	No distillate collected.	Fill reservoir with ASTM type II water	As needed

REVISION: 2	STATUS: Final	METHOD: 351.3	APPROVAL DATE: 5/8/98

10.4 Analytical Run Sequence

- Prep Blank
- LCS
- Up to 10 Samples
- DUP
- MS

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water	Soil
TKN	T	< 0.3 mg/L for 100 mL sample	< 6.0 mg/kg for a 5 g sample

REVISION: 2	STATUS: Final	METHOD: 351.3	APPROVAL DATE: 5/8/98

Key Type:

T = Compound/Analyte is target compound routinely reported.
M= Compound/Analyte is listed in the method but is not routinely reported by E & E.
C = Compound/Analyte is specified by the client and can be analyzed under this method.
S = Compound/Analyte is routinely used as a matrix spike.
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as a surrogate spike.

11.2 METHOD CALCULATION

$$TKN\,(mg/L \text{ or } mg/kg) = \frac{N \bullet (V - B)\ x\,14{,}000}{C}$$

where:

B = Volume of 0.02 N H_2SO_4 necessary to titrate blank
C = Volume of sample (mL) or weight in grams
N = Normality of H_2SO_4 titrant used
V = Volume of 0.02 N H_2SO_4 used (mL) to titrate sample.
14,000 = Conversion from meq N/mL to mg N/L

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
LCS/ICV 10 mg/L	1 per batch of 20 or fewer samples.	1 mL of 1000 mg/L ammonia spiking solution added to 100 mL ASTM type II water and processed.	See Control Limit Summary, TKN	Repeat digestion . Make new standard.
Duplicate	1 per batch of 10 or fewer samples.	A second portion of sample processed.	See Control Limit Summary, TKN	Comment
Matrix Spike 200 mg/kg soil, 10 mg/L water	1 per batch of 20 or fewer samples.	1 mL of 1000 mg/L Ammonia Spiking Solution added to another 100 mL aliquot of sample and processed a 5 g portion of soil sample, the resulting concentration is 200 mg/kg.	See Control Limit Summary, TKN	Comment
Blank	1 per batch of 20 or fewer samples.	100 mL of ASTM type II water processed.	<0.3 mg/L waters <6.0 mg/kg soils	Repeat

See attached

Control Limits for Routine Quality Control Samples

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
LCS	TKN	Water, Soil as Water	In-House	Annually
DUP	TKN	Water, Soil as Water	In-House	Annually
Spike	TKN	Water, Soil as Water	In-House	Annually
REVISION: 2	STATUS: Final	METHOD: 351.3	APPROVAL DATE: 5/8/98	

Table 12-3

ACCEPTANCE CRITERIA FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameter	Spike Amount	Recovery (%)	RPD
LCS	TKN	10 mg/L	See Control Limit Summary, TKN	NA
DUP	TKN	NA	NA	See Control Limit Summary, TKN
Spike	TKN	Water 10 mg/L, Soil 200 mg/kg	See Control Limit Summary, TKN	NA
REVISION: 2	STATUS: Final	METHOD: 351.3	APPROVAL DATE: 5/8/98	

13.0 SPECIAL PROJECT REQUIREMENTS

NA

14.0 SAMPLE DISPOSAL

14.1 Residue in the digestion/distillation has to be dumped in the hazardous waste mercury salts drum located in the General Analytical Lab.

14.2 The Boric Acid solution (that was used for titration) can be flushed down the sink with copious amounts of H_2O.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Biological Oxygen Demand (BOD5 and CBOD5)			
ORIGINAL AUTHOR	R. Piccione	REVISION AUTHOR	P. Najm
IMPLEMENTATION DATE	6/11/1998	ANNUAL REVIEW DATE	06/11/99
FILE INFORMATION	M:\SOPs\Final\GAC\GAC11.ene-11/30/98 4:30 PM		
REVISION: 3	STATUS: Final	METHOD: 5210B	APPROVAL DATE: 05/08/98

1.0 SCOPE AND APPLICATION

1.1 The 5-day biochemical oxygen demand test is used to determine, among others, the oxygen required for the biochemical degradation of organic material. It is applicable to wastewaters, effluents and polluted waters. Carbonaceous BOD ($CBOD_5$), the BOD attributable to carbon containing organic matter is also addressed here by chemical nitrification inhibition.

2.0 METHOD SUMMARY

Nutrient enriched water along with a specific amount of seed and sample are added to an air tight bottle to overflowing. These bottles are then incubated in the dark for five days. The BOD is then computed by calculating the difference between the initial and final DO. If CBOD is desired, dilutions are prepared using a nitrificiation inhibitor.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E&E, Inc. Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

4.0 REFERENCES

4.1 Standard Methods, 18th edition, Method 5210B, 1992

4.2 Use checklist C-078 for analyst and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 BOD - biochemical oxygen demand.

5.2 CBOD - carbonaceous biochemical oxygen demand.

5.3 Seed – a water source containing a sufficient microbial population which is fairly consistent day to day.

5.4 ASC Control Chart Database - Access™ database used for control charting by ASC. Control limits are tabulated by Lab, Method and by Matrix in the "Control Limit Summary".

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1.1 Samples containing caustic alkalinity or acidity —Neutralize samples to pH of 6.5 – 7.5 with a solution of sulfuric acid (H_2SO_4) or sodium hydroxide (NaOH) of such strength that the quantity of reagent does not dilute the sample by more than 0.5%. The pH of seeded dilution water should not be affected by the lowest sample dilution.

6.2 Samples containing oxidizing agents ~~residual chlorine compounds~~: if possible, avoid samples containing residual chlorine by sampling ahead of chlorination processes. If the sample has been chlorinated

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis	Container Type and Preservative
Water	Standard	NA	48 hours after collection	1-L Glass or poly unpreserved 2-5°C

REVISION: 3	STATUS: Final	METHOD: 5210B	APPROVAL DATE: 05/08/98

10.0 PROCEDURE

10.1 Winkler Calibration of Dissolved Oxygen (D.O.) Meter.

10.1.1 Allow 15 min. warm-up time. Set redline and zero with respective controls, check time and temperature and record.

10.1.2 Fill 2 replicate BOD bottles with dilution water. Read D.O. on 0 to 10 scale and record readings for both bottles.

10.1.3 Determine D.O. of one of the replicates as follows:

10.1.3.1 Add contents of manganous sulfate and alkali iodide azide pillows to full bottle, stopper carefully allowing no air bubbles in bottle.

10.1.3.2 Invert bottle several times to mix, allow floc to settle, then repeat mixing.

10.1.3.3 Add contents of sulfamic acid pillow, stopper and mix well, allowing no trapped air.

10.1.3.4 Measure 200 mL of this solution into 500 mL Erlenmeyer flask and titrate with sodium thiosulfate to pale straw color while stirring.

10.1.3.5 Add 5 drops starch indicator solution and continue titration dropwise until blue color vanishes.

10.1.3.6 Record volume titrant to 0.05 mL; 1 mL = 1 mg/L D.O. This is the initial D.O.

10.1.4 Calibrate meter using the D.O. determined in Step 10.1.3 using the other replicate sample.

10.1.5 Always make sure stirrer is on, range selection is correct, and reading has stabilized when using D.O. meter. Then set the meter using the adjustment dial on the front to the winkler titration result (which is equivalent to the volume of titrant used). NOTE: If the Winkler test reads less than 7.50, aerate dilution water until a minimum of 7.50 is obtained.

10.2 Sample Analysis

10.2.1 Acid-wash all bottles with 25% HCl solution. Rinse thoroughly with ASTM Type II water.

10.2.2 Record BOD bottle numbers allowing 2 bottles for blanks, 2 bottles for seeded blanks, 2 bottles for standard, and 3 to 5 bottles for each set of sample dilutions. See Tables 12-4 ~~and 12-5~~ for dilution guidance.

10.2.3 While seed solution is still stirring, transfer 25 mL (or suitable amount to deplete at least 50% of oxygen) to each seeded blank bottle and 4 mL to each sample bottle. (Sample should be 20°C before use to avoid supersaturation). Do not add to blanks.

10.2.4 Add 6 mL of glucose/glutamic acid standard solution to the two standard bottles per batch. (This is the LCS).

10.2.5 Check samples for residual chlorine with KI - starch paper. Remove residual if found. (Section 6.2)

10.2.6 Record pH of sample, adjust to be within pH 6.5 to 7.5 with 10% H_2SO_4 or 10% NaOH solution. (Section 6.1) The sample should not be diluted by the acid or base by more than 0.5%. Add sample to cor-

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Water (Units)
BOD_5 or $CBOD_5$	TL	2.4 mg/L at a sample volume of 250 mL
REVISION: 3	STATUS: Final	METHOD: 5210B	APPROVAL DATE: ~~05/08/98~~

Key Type:

T = Compound/Analyte is target compound routinely reported.
M= Compound/Analyte is listed in the method but is not routinely reported by E & E
C = Compound/Analyte is specified by the client and can be analyzed under this method.
S = Compound/Analyte is routinely used as a matrix spike
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as s surrogate spike.

11.2 Method Calculations

11.2.1 Seed correction: $mg/L = (\Delta D.O.) \times 4/25$

where:

Δ D.O. = The average change (initial to final readings) in DO of the seeded blank samples. Seed correction factor to be between 0.6/1.0 mg/L.
4/25 = Ratio of seed in samples to seed in control

Do not subtract seed correction from blanks

11.2.2 Determine BOD or CBOD for each sample dilution.

$$\text{Sample } BOD_5 \text{ or } CBOD_5, \text{ mg/L} = \frac{(\text{D.O. initial} - \text{D.O. final}) - \text{seed correction}}{} \times \frac{300}{V}$$

where:

D.O. initial = D.O. of diluted sample immediately after preparation.
D.O. final = D.O. of diluted sample after incubation.
V = Aliquot of sample used, mL.
300 = Size of BOD bottle, mL.

11.2.3 Criteria used to determine results to accept for averaging:

- Exclude all dilutions where the D.O. depletion is < 2 mg/L after seed correction.
- Exclude all dilutions where < 1 mg/L D.O. remains in sample.
- Average BOD readings for each acceptable dilution for that sample and report result in mg/L.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Blank	2 per batch	300 mL of dilution water in a capped BOD bottle.	< 2.0 mg/L	Wash bottles with acid prior to use. check DO meter calibration.
Standard (LCS)	2 per batch	6 mL of Glucose/Glutomic Acid standard solution plus 4 mL of seed solution to 300 mL in a BOD bottle.	170 - 230 mg/L	Qualify all BOD determinations made with this seed and dilution water.
Seed Control	2 per batch	25 mL of seed solution to 300 mL in a BOD bottle.	0.6 - 1.0 mg/L (result of seed correction calculation)	Use more seed. Replace seed if necessary.

REVISION: 3	STATUS: Final	METHOD: 5210B	APPROVAL DATE: 05/08/98

Control Limits for Routine Quality Control Samples

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES See attached

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
LCS	BOD_5 or $CBOD_5$	Water	Method	Annually

REVISION: 3	STATUS: Final	METHOD: 5210B	APPROVAL DATE: 05/08/98

Table 12-3

ACCEPTANCE CRITERIA FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameter	Spike Amount	Recovery (%)	RPD
LCS	BOD_5 or $CBOD_5$	200 mg/L	200 ± 30	NA
REVISION: 3	STATUS: Final	METHOD: 5210B	APPROVAL DATE: 05/08/98	

Table 12-4

BOD RANGE TABLE

Dilution Factor	Sample (mL)	BOD Range (mg/L)	Dilution Factor	Sample (mL)	BOD Range (mg/L)
~~1~~ 1.2	250	2.4-8.4	50	.4	1500-5200
~~2~~ 1.67	200	9-10.5	800	.375	1600-5600
3	100	6-21	900	.333	1800-6300
4	75	8-28	1000	.3	2000-7000
5	60	10-35	2000	.15	4000-14000
6	50	12-42	3000	.1	6000-21000
7.5	40	15-52.5	4000	.075	8000-28000
10	30	20-70	5000	.06	10000-35000
12	25	24-84	10000	.03	20000-70000
15	20	30-105	20000	.015	40000-140000
20	15	40-140	30000	.01	60000-210000
25	12	50-175	50000	.006	100000-350000
30	10	60-210	100000	.003	200000-700000
33	9	66-231	200000	.0015	400000-1400000
38	8	76-266			
43	7	86-301			
50	6	100-350			
60	5	120-420			
75	4	150-525			
100	3	200-700			
150	2	300-1050			
300	1	600-2100			
400	.75	800-2800			
500	.6	1000-3500			
600	.5	1200-4200			
REVISION: 3	STATUS: Final	METHOD: 5210B	APPROVAL DATE: 05/08/98		

13.0 SPECIAL PROJECT REQUIREMENTS

Client-Specific Quality Control Requirements

13.1 West Valley: 250 mL and 300 mL sample volume.

13.2 West Valley: Oxidizer test and pretreatment

13.2.1 The sample is checked for oxidizing agents by adding 1.0 mL 1:1 acetic acid and 1.0 mL potassium iodide solution to 100 mL of sample.

TITLE: Phenols by Method 9065 (4AAP)			
ORIGINAL AUTHOR	F. Lindauer	REVISION AUTHOR	P. Rosiek
IMPLEMENTATION DATE	8/6/1998	ANNUAL REVIEW DATE	8/6/1999
FILE INFORMATION	\\LMSRV1\LABORATORY\SOPs\Final\GAC\GAC13.ene-11/30/98 4:31 PM		
REVISION: 1	STATUS: Final	METHOD: 9065	APPROVAL DATE: 8/6/98

1.0 SCOPE AND APPLICATION

1.1 To determine the amount of phenolic compounds in drinking, saline, surface waters, domestic and industrial wastes. The procedure is also extended to include soils. It is not possible to use this method to differentiate between different kinds of phenols.

2.0 METHOD SUMMARY

2.1 Phenolic materials react with 4-aminoantipyrine in the presence of Potassium Ferricyanide at a pH of 10 to form a stable reddish-brown colored antipyrine dye. The amount of color produced is a function of the concentration of phenolic material.

3.0 HEALTH AND SAFETY

3.1 If volatile/distillable organic compounds are known to be present, reform the distillation in a hood. All work with chloroform must be done in a hood.

3.2 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E & E, Inc., Analytical Services Center, Chemical Hygiene Plan located in the QA Library Island shelf 2.

3.3 All sample analysis to be performed under a fume hood.

4.0 REFERENCES

4.1 SW-846, Method 9065 (Rev 0, 9/86), 3rd ed., June 1996.

4.2 "Methods for the Chemical Analysis of Water and Wastes" (EPA-600/4-79-020) March 1979, Method 420.1.

4.3 Use checklist C-061 for analyst and peer review.

4.5 GAC.57 Operation of Spectronic 601 for definitive analysis.

5.0 DEFINITIONS/ACRONYMS

5.1 ASC Control Chart Database - Access™ database used for control charting by ASC. Control limits are tabulated by Lab, Method, and by Matrix in the "Control Limit Summary".

5.2 Laboratory Control Sample (LCS)– distilled reference standard.

5.3 Matrix Spike (MS) – prepared along with samples in batch, but with addition of known concentration.

5.4 CCV - Distilled standard of known concentration from an independent source different from calibration stock. Prepared with batch.

5.5 Distilled Blank – Prepared the same as samples in batch.

5.6 Duplicate (DUP) – A second portion of a sample prepared with batch.

5.7 Batch – Consists of 20 samples or less.

Table 8-2

STANDARD AND REAGENT PREPARATION

Stock	Standard	Amount of Stock Added to	Final Concentration
4-Aminoantipyrin (4-AAP)	4-Aminoantipyrine Solution	Dissolve 2.0 4-AAP in ASTM water and dilute to 100 mL. Prepare daily.	NA
$K_3Fe(CN)_6$	Potassium Ferricyanide solution	Dissolve 8.0 g $K_3Fe(CN)_6$ in ASTM type II water and dilute to 100 mL. Prepare daily.	NA
REVISION: 1	STATUS: Final	METHOD: 9065	APPROVAL DATE: 8/6/98

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation	Analysis	Container Type and Preservative
Water	Standard	NA	28 days	Glass, 1 g $CuSO_4 \cdot 5H_2O$ per L, pH < 4 w/ H_2SO_4, 2-5°C.
Soil	Standard	NA	28 days	Glass, 2-5°C
Water	NYSDEC	NA	28 days	Glass, H_2SO_4 to pH < 2, 2-5°C
REVISION: 1	STATUS: Final	METHOD: 9065		APPROVAL DATE: 8/6/98

10.0 PROCEDURE

NOTE: Spectrophotometer should be set and warmed up at the proper wavelength for at least one half hour before use to avoid wavelength fluctuations.

10.1 CURVE PREPARATION – Direct Method

10.1.1 Pipette 0, 0.5, 1, 2, 5, 8, and 10 mL of working calibration standard A into 1-L Teflon separatory funnel. Bring up to 500 mL with ASTM type II water. This gives 0, 50, 100, 200, 500, 800, and 1000 µg/L standards.

10.1.2 Continue with the Sample Analysis section (10.5) Direct Method (10.4).

10.1.3 Zero the spectrophotometer with an undigested blank. See SOP GAC.57 for operating the spectrophotometer.

10.1.4 Enter standard concentrations in µg/L, the corresponding absorbances and the correlation coefficient in logbook. (≥ 0.995).

10.1.5 Calibration curve good for 6 months.

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Direct Method

$$\text{Phenols}\ \frac{\text{mg}}{\text{L}} = \frac{(\mu\text{g/L curve})\left(\frac{100}{\text{mL dev}}\right)(\text{mL distilate})}{(1000)(\text{mL distilled})}$$

where:

mL dev = mL developed
mL distilled = for soils use gram weight

11.12 Chloroform Extraction

$$\text{Phenols}\frac{\text{mg}}{\text{L}} = \frac{(\mu\text{g/L curve})(\text{mL distillate})(500)}{(1000)(\text{mL distilled})(\text{mL dev.})}$$

where:

mL dev = mL developed
mL distilled = for soils, use gram weight

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water (mg/L)	Soil (mg/kg)
Phenols, Direct Method	TSL	0.05	2.5
Phenols, Extraction Method	TSL	0.005	0.5
REVISION: 1	STATUS: Final	METHOD: 9065	APPROVAL DATE: 8/6/98

Key Type:

T = Compound/Analyte is target compound routinely reported.
M= Compound/Analyte is listed in the method but is not routinely reported by E & E.
C = Compound/Analyte is specified by the client and can be analyzed under this method.

S = Compound/Analyte is routinely used as a matrix spike.
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as a surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	~~Preparation~~ Instructions	Acceptance Criteria	Corrective Action
Distilled Blank	1 per batch	500 mL ASTM Type II water	< 0.005 mg/L	Reanalyze the batch
Undistilled Blank (to zero the sepctro-photometer)	1 per batch	500 mL ASTM Type II water	NA	NA
Undistilled ICV/LCS (0.5 mg/L)	1 per batch Direct Method	5.0 mL working reference standard A into 100 mL ASTM Type II water	± 15%	Reanalyze
Distilled LCS/ICV (0.03 mg/L)	1 per Batch	15 mL of working reference standard B into 500 mL ASTM Type II water.	See Control Limit Summary, Phenol	Reanalyze remake stock make new curve reanalyze samples
CCV Undistilled (0.20 mg/L) Direct Method	1 per Batch	2.0 ~~20~~ mL of working calibration standard A into 100 mL ASTM Type II water.	± 15%	Reanalyze remake stock make new curve reanalyze samples
CCV 0.03 mg/L Undistilled (Chloroform Extraction)	1 per 20 analyzed	15 mL of working calibration standard B into 500 mL ASTM Type II water	± 15%	Reanalyze remake stock make new curve reanalyze samples
Duplicate (DUP)	1 per 10 or less	A second portion of sample	See Control Limit Summary, Phenol	~~Reanalyze~~ Comment
Matrix Spike 0.03 mg/L (MS) (Chloroform Extraction)	1 per 20 or less	Add 15 mL working reference standard B into an aliquot of sample for sample distillation.	See Control Limit Summary, Phenol	~~Reanalyze; remake stock~~ Comment

REVISION: 1	STATUS: Final	METHOD: 9065	APPROVAL DATE: 8/6/98

Control Limits for Routine Quality Control Samples

See attached

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
LCS	Phenols	Water & Soil	In-house	Annual
Spike (MS)	Phenols	Water & Soil	In-house	Annual
DUP	Phenols	Water & Soil	In-house	Annual
REVISION: 1	STATUS: Final	METHOD: 9065	APPROVAL DATE: 8/6/98	

Table 12-3

ACCEPTANCE CRITERIA FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameter	Spike Amount	Recovery (%)	RPD
LCS	Phenols H_2O/Soil	0.03 mg/L 1.3 mg/kg	See Control Limit Summary, Phenol	NA
Spike	Phenols H_2O/Soil	0.03 mg/L 1.3 mg/kg	See Control Limit Summary, Phenol	NA
DUP	Phenols H_2O/Soil	NA	NA	See Control Limit Summary, Phenol
LCS Direct Method*	Phenols H_2O/Soil	0.03 mg/L 1.3 mg/kg	See Control Limit Summary, Phenol	NA
Spike Direct Method*	Phenols H_2O/Soil	0.03 mg/L 1.3 mg/kg	See Control Limit Summary, Phenol	NA
DUP Direct Method*	Phenols H_2O/Soil	NA	NA	See Control Limit Summary, Phenol
REVISION: 1	STATUS: Final	METHOD: 9065	APPROVAL DATE: 8/6/98	

*Direct Method may not have control chart completed due to not enough data points. Use chloroform extraction values until enough data points have been accumulated.

13.0 SPECIAL PROJECT REQUIREMENTS

NA

14.0 SAMPLE DISPOSAL

14.1 Chloroform extracts are placed in the waste solvent storage drum.

14.2 Water based extracts can be flushed down the drain with copious amounts of water.

TITLE: Total Hardness by EPA Method 130.2			
ORIGINAL AUTHOR	F. Lindauer	REVISION AUTHOR	P. Najm
IMPLEMENTATION DATE	5/5/1998	ANNUAL REVIEW DATE	5/5/1999
FILE INFORMATION	M:\SOPs\Final\GAC\GAC27.ene-11/30/98 4:31 PM		
REVISION: 2	STATUS: Final	METHOD: 130.2	APPROVAL DATE: 4/11/98

1.0 SCOPE AND APPLICATION

1.1 This SOP directs the determination of magnesium and calcium salts in water as "Hardness" and reported as mg/L $CaCO_3$ in drinking, surface, saline waters, domestic or industrial wastes.

2.0 METHOD SUMMARY

Calcium and Magnesium ions in the sample are sequestered upon the addition of the sodium EDTA (disodium ethylene diamine tetraachtate) as a titrant. The endpoint of the test is visually detected by means of Eriochrome Black T indicator. (which is red in the presence of Calcium and Magnesium and blue when the cations are sequestered).

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E&E, Inc. Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

4.0 REFERENCES

4.1 Methods for the Chemical Analysis of Water and Wastes, USEPA, August 1993 (revision 2.0) Method 130.2.

4.2 Use checklist C-085 for analyst and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 Hardness - The hardness of a water or waste characterized by the level of Magnesium or Calcium salts.

5.2 ASC Control Chart Database used for control charting by ASC. Control limits are tabulated by laboratory method and by matrix in the "Control Limit Summary".

6.0 INTERFERENCES/POTENTIAL PROBLEMS

Excessive amounts of heavy metals can interfere. This can be overcome by the addition of sodium cyanide (Caution: deadly poison). Record any special pretreatment in the logbook.

7.0 INSTRUMENTATION AND EQUIPMENT

- Micro burette (10-mL size)
- Flasks
- Magnetic stir plate and stir bar
- Class A pipettes

Table 8-2

STANDARD AND REAGENT PREPARATION

Stock	Standard Name	____ Amount of Stock Added to ____	Final Concentration
Tetraacetate (EDTA)		standard calcium solution. Standardize at each use. Good for 6 months.	
Ammonium Hydroxide	Ammonium Hydroxide Solution	Dilute 70-mL of concentrated NH_4OH to 1-L with ASTM type II water. Good for 6 months.	1 N
EDTA, Magnesium sulfate, Ammonium chloride, Ammonuim hydroxide	Buffer Solution	Dissolve 1.179 g of Disodium EDTA and 0.780 g $MgSO_4 \bullet 7H_2O$ in 50 mL ASTM type II water. Add this to a 250-mL volumetric containing 16.9 g NH_4Cl and 143 mL of NH_4OH. Dilute up to volume with ASTM type II water. Good for one month.	NA
Sodium Chloride and Eriochrome Black T	Indicator	100 g NaCl and 0.50 g Eriochrome Black T, mix well, stopper and shake into jar.	NA

REVISION: 2	STATUS: Final	METHOD: 130.2	APPROVAL DATE: 4/11/98

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Analysis (Days)	Container Type and Preservative
Water	All	180	2 - 5°C poly container preserve with Nitric Acid to pH < 2.

REVISION: 2	STATUS: Final	METHOD: 130.2	APPROVAL DATE: 4/11/98

10.0 PROCEDURE

10.1 Standardization

10.1.1 Place 10.0 mL standard calcium solution in a beaker containing about 40 mL of ASTM type II water.

10.1.2 Titrate using Normal Hardness Procedure.

Calculate normality of EDTA titrant as follows:

$$\text{Normality of EDTA} = \frac{0.2}{\text{Volume (mL) of EDTA}}$$

10.2 Normal to High Hardness.

10.2.1 Sample should require <15 mL EDTA titrant and titration should be completed within 5 minutes of buffer addition.
10.2.2 Place 25.0 mL sample in titration vessels, adjust pH to 7 (if pH > 7, bring down with 1 n HNO_3) with 1N ammonium hydroxide and dilute to about 50 mL with ASTM type II water.
10.2.3 Add 1 to 2 mL buffer solution.
10.2.4 Add a small scoop of indicator formulation.
10.2.4.1 Titrate slowly, continuously stirring, with standard EDTA titrant until last reddish tint disappears. Solution is normally blue at end point.
10.2.4.2 If end ~~point is not sharp~~ (as determined by practice run) See Section 6.

10.3 Low Hardness (< 5 mg/L)
10.3.1 ~~Use a larger sample~~ (100 mL).
10.3.2 Use proportionately larger amounts (5 times) of buffer and indicator.
10.3.3 Use a microburet and run a blank using ASTM type II water.

11.0 DATA REDUCTION/EVALUATION/REPORTING

$$\frac{A \ (N) \ 50{,}000}{\text{mL sample}} = \text{mg } CaCO_3/L$$

where:

A = mL EDTA titrant
N = normality titrant

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Water (Units)
Total Hardness	T, L	1 mg/L for 100 mL sample 4 mg/L for 25 mL sample PQL = 4 for 25 mL PQL = 1 for 100 mL
REVISION: 2	STATUS: Final	METHOD: 130.2	APPROVAL DATE: 4/11/98

Key Type:

T = Compound/Analyte is target compound routinely reported.
M= Compound/Analyte is listed in the method but is not routinely reported by E & E.
C = Compound/Analyte is specified by the client and can be analyzed under this method.
S = Compound/Analyte is routinely used as a matrix spike.
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as a surrogate spike.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	~~Preparation~~ Instructions	Acceptance Criteria	Corrective Action
Prep Blank	1 per batch	50 mL ASTM type II water.	<1.0 mg/L	Reanalyze, if results still high, notify the GAC supervisor (water system may need checking).
LCS	1 per 10 ELAP	Pipette 5.0 mL of standard Calcium solution to 50 mL with ASTM type II water. True value = 100 mg/L.	In-House	Reanalyze
Dupli-cate (DUP)	1 per batch or 10%	Second aliquot of sample.	In-House	Reanalyze
REVISION: 2	STATUS: Final	METHOD: 130.2	APPROVAL DATE: 4/11/98	

Spikes can be done by client request.

Control Limits for Routine Quality Control Samples

See attached

Table 12-2

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
LCS	Total Hardness	Waters	In-House	Annually
Duplicate	Total Hardness	Waters	In-House	Annually

REVISION: 2	STATUS: Final	METHOD: 130.2	APPROVAL DATE: 4/11/98

Table 12-3

ACCEPTANCE CRITERIA FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameter	Spike Amount	Recovery (%)	RPD
LCS	Total Hardness	100 mg/L	See Control Limit Summary, Hardness – W	NA
Duplicate	Total Hardness	NA	NA	See Control Limit Summary, Hardness – W

REVISION: 2	STATUS: Final	METHOD: 130.2	APPROVAL DATE: 4/11/98

13.0 SPECIAL PROJECT REQUIREMENTS

NA

14.0 SAMPLE DISPOSAL

Titrated samples may be poured down lab drain with copeous tap water.

15.0 EXAMPLE FORMS

NA

END OF SOP

TITLE: Ammonia-Nitrogen by Method 350.2			
ORIGINAL AUTHOR	D. Piekarek, K. Woodward	REVISION AUTHOR	K. DeMarco
IMPLEMENTATION DATE	5/18/1998	ANNUAL REVIEW DATE	5/18/1999
FILE INFORMATION	M:\SOPs\Final\GAC\GAC2.ene-12/01/98 8:40 AM		
REVISION: 1	STATUS: Final	METHOD: 350.2	APPROVAL DATE: 5/7/98

1.0 SCOPE AND APPLICATION

1.1 This SOP covers the determination of ammonia-nitrogen, in drinking, surface and saline waters, domestic and industrial wastes. The method has been modified to determine ammonia-nitrogen in soils and waste. The macro distillation option of the method is used in this SOP.

2.0 METHOD SUMMARY

2.1 The sample is buffered at pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanides and organic nitrogen compounds, and is then distilled into a solution of boric acid.

2.2 The ammonia in the distillate is then determined titrimetrically with standard sulfuric acid with the use of a mixed indicator.

2.3 Modification: An automated distillation system (Büchi 316) is used to distill the samples.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E&E, Inc. Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

4.0 REFERENCES

4.1 Method 350.2, Methods for Chemical Analysis of Water and Wastes. EPA –600/4-79-020, March 1979.

4.2 Use checklist C_084 for analyst and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 NH_3-N = Ammonia as nitrogen.

5.2 ASC Control Chart Database – Access ™ database used for control charting by ASC. Control limits are tabulated by lab, method, and by matrix in the Control Limit Summary.

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Volatile alkaline compounds, such as certain ketones, aldehydes and alcohols, may cause an off color in this distillation method.

6.2 Residual chlorine must be removed by pretreatment with sodium thiosulfate prior to distillation.

8.1 STANDARDIZATION

Standardize against 40.00 mL 0.05N Na_2CO_3 solution, with about 60 mL water in a beaker by titrating potentiometrically to pH of about 5. Lift out electrodes, rinse into the same beaker and boil gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature, rinse cover glass into beaker, and finish titrating to the pH inflection point. Calculate normality:

$$\text{Normality, N} = \frac{A \bullet B}{53.00 \bullet C}$$

where:

A = g Na_2CO_3 weighed into 1-L flask
B = mL Na_2CO_3 solution taken for titration, and
C = mL acid used

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1
HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Water	Standard	NA	28	Glass, H_2SO_4 to pH <2, 2-5°C
Soil	Standard	NA	28	Glass jar – no preservative
REVISION: 1	STATUS: Final	METHOD: 350.2	APPROVAL DATE: 5/7/98	

10.0 PROCEDURE

10.1 Turn Büchi 316 on, making sure that you have the cold tap water running through the condenser tube. Make sure the Büchi water reservoir jug is at least half full with ASTM type II water. Let the Büchi 316 warm up for at least 5 minutes.

10.2 Set the Büchi 316 at 4 minutes.

10.3 To clean out distillation apparatus, add approximately 15 mL 1:1 HCl to 200 mL ASTM type II water and run. Then distill an instrument blank to insure the apparatus is clean making sure asparation dial is set at "yes".

10.4 Water samples: Check for residual chlorine with KI paper.

10.5 Remove any residual chlorine in the sample by adding dechlorinating agent equivalent to the residual chlorine.

10.6 Water sample, use up to 200 mL in distillation tube.

10.7 Soil sample use 10 g and 200 mL ASTM type II H_2O in a distillation tube.

10.8 Adjust pH by adding 1 N NaOH, until pH is 9.5, checking pH during addition with a calibrated pH meter.
10.9 Distillation: add approximately12 - 13 mL of the Borate Buffer.
10.10 Turn knob to aspiration "yes" for waters "no" for soils.
10.11 Distill into 25 mL of 2% Boric acid contained in a 250-mL Erlenmeyer flask.
10.12 Note: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.
10.13 Remove distillation tube from apparatus. **Tube is hot**, use rubber grip tongs. Add a few drops of the mixed indicator to the distillate and titrate with 0.02 N H_2SO_4 to a purple end point. The color change goes from green to an end point of purple.
10.14 Turn off Büchi 316 distillation unit.
10.15 Turn off cold water for the condenser.
10.16 **Analytical Run Sequence**
Prep Blank
LCS
Up to 10 Analyses
DUP
MS

Table 10-1

ROUTINE MAINTENANCE PROCEDURES

Equipment Instrument	Symptom	Operation	Frequency
Büchi 316 Distillation apparatus.	Apparatus Contamination	Clean instrument by running a solution of 15 mL 1:1 HCl and 200 mL ASTM type II water. Distill a blank to ensure it is clean.	Before use.
Büchi 316.	No distillate being collected.	Add ASTM type II water to reservoir.	As needed.

REVISION: 1	STATUS: Final	METHOD: 350.2	APPROVAL DATE: 5/7/98

11.0 DATA REDUCTION/EVALUATION/REPORTING

Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL	
		Water	Soil
NH_3-N	T	0.2 mg/L for 200 mL sample based on 0.1 mL titrant	3.0 mg/kg for 10 g sample based on 0.1 mL titrant

REVISION: 1	STATUS: Final	METHOD: 350.2	APPROVAL DATE: 5/7/98

Key Type:

T = Compound/Analyte is target compound routinely reported.
M= Compound/Analyte is listed in the method but is not routinely reported by E & E.
C = Compound/Analyte is specified by the client and can be analyzed under this method.
S = Compound/Analyte is routinely used as a matrix spike.
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as a surrogate spike.

11.1 Method Calculations

$$\text{Ammonia Nitrogen (mg/L, mg/kg)} = \frac{A \times B \times 14{,}000}{C}$$

where: A = mL 0.02N H_2SO_4 used
B = Normality of H_2SO_4 titrant used
C = Volume of sample (mL or g)

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Blank	1 Batch	200 mL ASTM type II H_2O and 15 mL Borate buffer	< 0.2 mg/L	Repeat
LCS Ref. Std.	At begin-ning and after every 20 analyses	1 mL of Ammonium Chloride stock solution (1000 mg/L) into 200 mL ASTM type II water. (5 mg/L)	See Control Limit Summary, Ammonia distilled	Repeat
Duplicate (DUP)	1/10 or less	Up to 200 mL sample for water, 10 g sample for soil.	See Control Limit Summary, Ammonia distilled	Repeat sample
Matrix Spike	1/20 or less	1 mL Ammonium Chloride stock solution (1000 mg/L) into a total of 200 mL sample for water and 10 g sample for soils. (5 mg/L; 100 mg/kg)	See Control Limit Summary, Ammonia distilled	Comment on event

Table 12-1 ROUTINE QUALITY CONTROL SAMPLES				
QC Type	**Frequency**	**Preparation Instructions**	**Acceptance Criteria**	**Corrective Action**
REVISION: 1	STATUS: Final	METHOD: 350.2	APPROVAL DATE: 5/7/98	

Control Limits for Routine Quality Control Samples

Table 12-2 CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES				
QC Type	**Parameters Charted**	**Type of Samples Charted**	**Source of Limits**	**Frequency Updated**
LCS	NH_3 - N	Water, Soil as Water	In-House	Annually
DUP	NH_3 – N	Water, Soil	In-House	Annually
SPIKE	NH_3 – N	Water, Soil	In-House	Annually
REVISION: 1	STATUS: Final	METHOD: 350.2	APPROVAL DATE: 5/7/98	

See attached

Table 12-3 ACCEPTANCE CRITERIA FOR ROUTINE QUALITY CONTROL SAMPLES				
QC Type	**Parameter**	**Spike Amount**	**Recovery (%)**	**RPD**
LCS	NH_3 – N Ammonia Distilled	5 mg/L	See Control Limit Summary, Ammonia	NA
DUP	NH_3 – N Ammonia Distilled	NA	NA	See Control Limit Summary, Ammonia
MS	NH_3 – N Ammonia Distilled	5 mg/L	See Control Limit Summary, Ammonia	NA
REVISION: 1	STATUS: Final	METHOD: 350.2	APPROVAL DATE: 5/7/98	

13.0 SPECIAL PROJECT REQUIREMENTS

NA

9.0 PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Table 9-1

HOLDING TIMES

Matrix	Client/Project	Preparation (Days)	Analysis (Days)	Container Type and Preservative
Waters	Standard	NA	48 Hours	Store at 2 – 5 °C, poly or glass bottles.

REVISION: 2	STATUS: Final	METHOD: 110.2	APPROVAL DATE: 5/20/98

10.0 PROCEDURE

10.1 Prepare standards having colors of 5, 10, 15, 20, 25, 40 and 50 units by diluting 0.5, 1.0, 2.0, 2.5, 4.0 and 5.0 mL stock color standard to 50-mL in Nessler tubes with ASTM type II water. Cover the tubes to protect against evaporation and contamination when not in use. They are good for 6 months.

10.2 Arrange standards in order of color units.

10.3 To observe the color of a sample, place 50 mL of sample into a matching Nessler tube and look vertically down the tubes of standards and sample against a white background such that light is reflected upward through the columns of liquid. If the color exceeds 50 units (the highest standard), dilute the sample with distilled water in known proportions until the color is within the range of the standards. Report as Apparent Color. Measure the pH with pH paper and record in lab notebook.

10.4 Samples that show visible signs of turbidity should centrifuged prior to reading the color. The sample should be compared to ASTM type II water to ensure that the sample is no longer turbid. Compare the sample to the standards. Record any pre-treatment (centrifugation) in the laboratory notebook. Report as True Color. Measure pH with pH paper and record.

11.0 DATA REDUCTION/EVALUATION/REPORTING

11.1 Target Compounds and Reporting Limits

Table 11-1

TARGET COMPOUNDS/ANALYTES AND QUANTITATION LIMITS

Compound/Analyte	Type	PQL Water
Color	T, C	< 5 NTU

REVISION: 2	STATUS: Final	METHOD: 110.2	APPROVAL DATE: 5/20/98

Key Type:

T = Compound/Analyte is target compound routinely reported.
M= Compound/Analyte is listed in the method but is not routinely reported by E & E.
C = Compound/Analyte is specified by the client and can be analyzed under this method.
S = Compound/Analyte is routinely used as a matrix spike.
L = Compound/Analyte is routinely used as a LCS spike.
Q = Compound/Analyte is used as a surrogate spike.

11.2 Method Calculations

$$\text{Color units} = \frac{A \bullet 50}{B}$$

Where: A = estimated color of a diluted sample, (color units)
B = volume (mL) of sample taken for dilution.

12.0 QUALITY CONTROL/QUALITY ASSURANCE/CORRECTIVE ACTION

Routine Quality Control Samples

Table 12-1

ROUTINE QUALITY CONTROL SAMPLES

QC Type	Frequency	Preparation Instructions	Acceptance Criteria	Corrective Action
Duplicate	1 per batch or 10% of samples.	2nd aliquot of sample.	± 5 units	Repeat
REVISION: 2	STATUS: Final	METHOD: 110.2	APPROVAL DATE: 5/20/98	

Control Limits for Routine Quality Control Samples

Table 12-2

See attached

CONTROL LIMITS FOR ROUTINE QUALITY CONTROL SAMPLES

QC Type	Parameters Charted	Type of Samples Charted	Source of Limits	Frequency Updated
Duplicate	Color	Waters	In-House Arbitrary	Annually
REVISION: 2	STATUS: Final	METHOD: 110.2	APPROVAL DATE: 5/20/98	

TITLE: Color, Platinum-Cobalt			
ORIGINAL AUTHOR	P. Najm	REVISION AUTHOR	P. Rosiek
IMPLEMENTATION DATE	6/11/1998	ANNUAL REVIEW DATE	6/11/1999
FILE INFORMATION	\\LMSRV1\LABORATORY\SOPs\Final\GAC\GAC24.ene-12/01/98 8:41 AM		
REVISION: 2	STATUS: Final	METHOD: 110.2	APPROVAL DATE: 5/20/98

1.0 SCOPE AND APPLICATION

1.1 To determine the color of water resulting from naturally occurring materials. This method is not applicable to highly colored or unusually colored industrial wastes.

2.0 METHOD SUMMARY

2.1 Color is measured by the comparison of the sample with platinum – cobalt standards of a known range. One unit of color is that produced by 1 mg/L platinum in the form of the Chloroplatinate ion.

2.3 High color standard in this SOP is 50 units instead of 70 units in the method.

3.0 HEALTH AND SAFETY

3.1 All employees should protect themselves at a minimum with safety glasses, protective gloves and a lab coat. For more information see the E&E, Inc. Analytical Services Center Chemical Hygiene Plan located in the QA Library Island shelf 2.

4.0 REFERENCES

4.1 EPA Method 110.2

4.2 Method 2120B Standard Methods, 18th Edition, 1992.

4.3 Use checklist number C-101 for analyst and peer review.

5.0 DEFINITIONS/ACRONYMS

5.1 Apparent color is the color due to substances in solution and to suspended matter. It is determined on the original sample without filtration or centrifugation.

5.2 True color is the color of the sample after the turbidity has been removed from the sample by centrifuging or filtering.

5.3 NTU = color unit

6.0 INTERFERENCES/POTENTIAL PROBLEMS

6.1 Since turbidity interferes with the determination, samples having turbidity it should be clarified by centrifugation.

6.2 This method is pH dependent.

6.3 Since biological activity may change the color characteristics of a sample, the determination should be made as soon as possible.

7.0 INSTRUMENTATION AND EQUIPMENT

7.1 Nessler tubes, matched 50-mL tall form.

AFCEE
INORGANIC ANALYSES DATA SHEET 9
INSTRUMENT ANALYSIS SEQUENCE LOG

Analytical Method: SW6010A

Lab Name : Ecology and Environment, Inc. Contract #:: F41689-96-D0711

Instrument ID: Optima

Field Sample ID/Std ID/ Blank ID/QC Sample ID	Date Analyzed	Time Analyzed
BLK	27-Jul-98	16:33
STD1A	27-Jul-98	16:36
STD1B	27-Jul-98	16:39
STD FE	27-Jul-98	16:42
STD FE2	27-Jul-98	16:45
STD2A	27-Jul-98	16:48
STD3A	27-Jul-98	16:50
ICV07271654	27-Jul-98	16:54
ICB07271703	27-Jul-98	17:03
CCV07271710	27-Jul-98	17:10
CCB07271713	27-Jul-98	17:13
ICSAB 10X	27-Jul-98	17:16
PBW830-1291	27-Jul-98	17:19
LCS830-1291	27-Jul-98	17:23
10616	27-Jul-98	17:26
10617	27-Jul-98	17:34
10617L	27-Jul-98	17:37
10618	27-Jul-98	17:40
10619	27-Jul-98	17:43
CCV07271749	27-Jul-98	17:49
CCB07271752	27-Jul-98	17:52
ICSAB 10X	27-Jul-98	17:56
10620	27-Jul-98	17:59
10621	27-Jul-98	18:01
10622	27-Jul-98	18:05
10623	27-Jul-98	18:08
10624	27-Jul-98	18:11
CCV07271817	27-Jul-98	18:17
CCB07271820	27-Jul-98	18:20
ICSAB 10X	27-Jul-98	18:24

Comments: 19980727204 STD1A - CA,K,NA. STD1B - MG

STD2A - 1A + 1B

STD3A - 2A DILUTIONS FOR CA, NA.

0-10
0-11

AFCEE
ORGANIC ANALYSES DATA SHEET 11
INSTRUMENT TUNE PERFORMANCE CHECK
4-Bromofluorobenzene

Analytical Method: SW8260b AAB#: 19980715431

Lab Name: Ecology & Environment, Inc. Contract#: F41689-96-D-0711 MS 8/19/98

Instrument ID: CLYDE_II Fraction: VOA

Tune File ID: C0330 Date Analyzed: 15-JUL-98

Initial Cal. ID: CGW02 Time Analyzed: 12:45

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE	
95	Base Peak, 100% relative abundance	100.00	
50	15.00 - 40.00% of mass 95	23.13	
75	30.00 - 60.00% of mass 95	45.88	
96	5.00 - 9.00% of mass 95	6.18	
173	Less than 2.00% of mass 174	0.41 (0.67)	a
174	50.00 - 100.00% of mass 95	60.68	
175	5.00 - 9.00% of mass 174	5.03 (8.29)	a
176	95.00 - 101.00% of mass 174	59.65 (98.32)	a
177	5.00 - 9.00% of mass 176	4.43 (7.43)	b

a = Value is relative to mass 174
b = Value is relative to mass 176

This tune check applies to the following Standards, Blanks, Samples and QC

	Sample ID	File ID	Date Analyzed	Time Analyzed
1	VSTD010	C0331	15-JUL-98	13:22
2	BLK 1251-11	C0334	15-JUL-98	16:59
3	10616.01	C0335	15-JUL-98	17:47
4	10617.01	C0336	15-JUL-98	18:23
5	10620.01	C0339	15-JUL-98	20:14
6	10621.01	C0340	15-JUL-98	20:50
7	10622.01	C0341	15-JUL-98	21:26
8	10623.01	C0342	15-JUL-98	22:02
9	10624.01	C0343	15-JUL-98	22:38
10	MS 10616.02	C0345	15-JUL-98	23:50
11	MSD10616.03	C0346	16-JUL-98	00:26
12	LCS 1251-11	C0347	16-JUL-98	01:02

AFCEE

INORGANIC ANALYSES DATA SHEET 4

INITIAL CALIBRATION VERIFICATION

Analytical Method: SW 6010A Total Metals **AAB #:** 19980723205

Lab Name: Ecology and Environment, Inc. **Contract #:** F41689-96-D-0711

Instrument ID: OPTIMA **Initial Calibration ID:** 980727203R

2nd Source ID or ICV ID: ICV07271428

Units: ug/L

	2nd Source or Initial Calibration Verification			
Analyte	**Expected**	**Found**	**%D**	**Q**
Antimony	2000.00	1994.00	0.3	
Arsenic	1000.00	997.20	0.3	
Barium	1000.00	987.60	1.2	
Beryllium	1000.00	1007.00	-0.7	
Cadmium	1000.00	998.10	0.2	
Chromium	1000.00	991.70	0.8	
Cobalt	1000.00	985.20	1.5	
Copper	1000.00	980.30	2.0	
Lead	2000.00	1975.00	1.2	
Manganese	1000.00	989.80	1.0	
Nickel	1000.00	984.20	1.6	
Selenium	1000.00	1001.00	-0.1	
Silver	1000.00	986.10	1.4	
Thallium	1000.00	987.80	1.2	
Vanadium	1000.00	997.50	0.2	
Zinc	1000.00	998.90	0.1	

Comments:

262
KH 9/3/6

AFCEE

INORGANIC ANALYSES DATA SHEET 4

INITIAL CALIBRATION VERIFICATION

Analytical Method: SW 6010A Total Metals **AAB #:** 19980723205

Lab Name: Ecology and Environment, Inc. **Contract #:** F41689-95-D-0711

Instrument ID: OPTIMA **Initial Calibration ID:** 980727203R

2nd Source ID or ICV ID: ICV07271654

Units: ug/L

	2nd Source or Initial Calibration Verification			
Analyte	Expected	Found	%D	Q
Calcium	50000.00	50790.00	-1.6	
Magnesium	50000.00	49680.00	0.6	
Potassium	50000.00	50040.00	-0.08	
Sodium	50000.00	50140.00	-0.3	

Comments:

2163
RMD 9/3/98

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INORGANIC ANALYSES DATA SHEET 4 A

CALIBRATION VERIFICATION

Analytical Method: SW 6010A Total Metals **AAB #:** 19980723205

Lab Name: Ecology and Environment, Inc. **Contract #:** F41689-96-D-0711

Instrument ID: OPTIMA **Initial Calibration ID:** 980727203R

CCV ID: CCV07271439

Units: ug/L

	Continuing Calibration Verification			
Analyte	Expected	Found	%D	Q
Aluminum	5000.00	4966.00	0.7	
Antimony	1000.00	961.80	3.8	
Arsenic	500.00	484.60	3.1	
Barium	500.00	468.70	6.3	
Beryllium	500.00	483.20	3.4	
Cadmium	500.00	473.10	5.4	
Chromium	500.00	474.20	5.2	
Cobalt	500.00	465.60	6.9	
Copper	500.00	496.50	0.7	
Iron	5000.00	4675.00	6.5	
Lead	1000.00	932.80	6.7	
Manganese	500.00	474.20	5.2	
Nickel	500.00	471.00	5.8	
Selenium	500.00	489.40	2.1	
Silver	500.00	489.30	2.1	
Thallium	500.00	482.90	3.4	
Vanadium	500.00	485.00	3.0	
Zinc	500.00	484.30	3.1	

Comments:

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INORGANIC ANALYSES DATA SHEET 5

INITIAL CALIBRATION BLANK

Analytical Method: SW 6010A Total Metals **AAB #:** 19980723205

Lab Name: Ecology and Environment, Inc. **Contract #:** F41689-96-D-0711

Units: ug/L

Initial Calibration Blank ID: ICB07271435 **Initial Calibration ID:** 980727203R

Analyte	Initial Calibration Blank	RL	Qualifier
Aluminum	40.80 U	200.0	
Antimony	6.60 U	50.0	
Arsenic	2.30 U	30.0	
Barium	1.40 U	5.0	
Beryllium	0.07 U	5.0	
Cadmium	0.29 U	7.0	
Chromium	0.40 U	10.0	
Cobalt	0.39 U	6.0	
Copper	1.09 U	10.0	
Iron	32.80 U	200.0	
Lead	2.70 U	25.0	
Manganese	2.00 U	3.0	
Molybdenum	1.00 U	15.0	
Nickel	0.70 U	10.0	
Selenium	5.10 U	30.0	
Silver	1.90 U	10.0	
Thallium	2.80 U	80.0	
Vanadium	1.29 U	10.0	
Zinc	5.96 U	10.0	

Comments:

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INORGANIC ANALYSES DATA SHEET 5

INITIAL CALIBRATION BLANK

Analytical Method: SW 6010A Total Metals **AAB #:** 19980723205

Lab Name: Ecology and Environment, Inc. **Contract #:** F41689-96-D-0711

Units: ug/L

Initial Calibration Blank ID: ICB07271703 **Initial Calibration ID:** 980727203R

Analyte	Initial Calibration Blank	RL	Qualifier
Calcium	31.80 U	1100.0	
Magnesium	20.20 U	100.0	
Potassium	213.30 F	500.0	
Sodium	445.10 F	1000.0	

Comments:

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