QUALITY ASSURANCE PROJECT PLAN RUNWAY STOCKPILES CHARACTERIZATION

HAMILTON ARMY AIRFIELD

NOVATO, CALIFORNIA

Final Submittal

Prepared by:

US Army Corps of Engineers ®

Sacramento District
Environmental Design Section

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LIST OF ACRONYMS AND ABBREVIATIONS

bgs	below ground surface
BRAC	Base Realignment and Closure
CCB	Continuing Calibration Blank
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CDQAR	Chemical Data Quality Assessment Report
CESPK	Corps of Engineers, Sacramento District
CL	Control Limit
COC	Chain of Custody
CRWQCB	California Regional Water Quality Control Board
cy	cubic yards
DL	Detection Limit
DQO	Data Quality Objectives
DTSC	California Department of Toxic Substances Control
EPA	Environmental Protection Agency
FSP	Field Sampling Plan
GC	Gas Chromatograph
GC/MS	Gas Chromatography/Mass Spectrometry
HAAF	Hamilton Army Airfield
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma (Spectroscopy)

ICS	Interference Check Standard
ICV	Initial Calibration Verification
IS	Internal Standard
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LUFT	Leaking Underground Fuel Tank
MDL	Method Detection Limit
MS	Matrix Spike
MSA	Method of Standard Addition
MSD	Matrix Spike Duplicate
µg/kg	micrograms per kilogram
mg/kg	milligrams per kilogram
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
P.E.	Professional Engineer
PM	Project Manager
PNAs	Polynuclear Aromatic Hydrocarbons
QL	Quantitation Limit
QA	Quality Assurance
QAC	Quality Assurance Chemist
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCSR	Quality Control Summary Report
RF	Response Factor
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SD	Serial Dilution
SIM	Selective Ion Monitoring
SOPs	Standard Operating Procedures

SPCC	System Performance Check Compounds
TCE	Trichloroethene
TPH-E	Total Petroleum Hydrocarbons - Extractable
TPH-P	Total Petroleum Hydrocarbons - Purgeable
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
VOC	Volatile Organic Compound

QUALITY ASSURANCE PROJECT PLAN
RUNWAY STOCKPILES CHARACTERIZATION
HAMILTON ARMY AIRFIELD

1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents functions, procedures, and specific quality assurance (QA) and quality control (QC) activities designed to achieve the data quality goals for determining the disposition options for the runway stockpiles at Hamilton Army Airfield. This project is conducted by the Environmental Design Section of the U.S. Army Corps of Engineers, Sacramento District (CESPK) on behalf of the Army Base Realignment and Closure (BRAC) environmental office and the Formerly Used Defense Sites (FUDS) program. This QAPP is prepared in accordance with EPA QA/R-5, EPA Requirements for Quality Assurance Project Plans (U.S. EPA, 2001). This document accompanies the Work Plan and the Field Sampling Plan.

1.1 Site Location and Project Objectives

The site location is illustrated in Figure 1-1 of the Work Plan. For this effort, stockpiled soil from various excavations at Hamilton Army Airfield must be characterized to determine if the soil stockpiled on the runways may be used unrestricted, restricted, or must be disposed of off-site. Historical data for the majority of stockpiles includes contaminant concentrations for many parameters, but not pesticides or metals. To achieve the objective, samples will be collected from the stockpiles and analyzed for pesticides and metals. In addition, the samples may be analyzed for other parameters either not available or parameters that may have degraded since the historical data was collected. The results will be compared to inboard area action goals.

1.2 QAPP Objectives and Use

Standard procedures and specifications are established to ensure that all laboratories produce comparable data, and that data quality is consistently assessed and documented. The specific objectives of this QAPP are to:

- provide standardized references and quality specifications for all anticipated field sampling, analysis, and data review procedures required for the project sites;
- provide guidance and criteria for selected field and analytical procedures; and
- establish procedures for reviewing and documenting compliance with field and analytical

procedures.

The fieldwork will include: hand auger drilling to 2 feet below the surface of the stockpiles, soil sample collection, packaging, and shipping to offsite laboratory for analysis.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 Corps of Engineers

The following Sacramento District, Corps of Engineers personnel have been assigned to accomplish the sampling design and execution required supporting this project. The USACE Project Manager is Jim McAlister for FUDS portion and Ray Zimny for the BRAC portion of the stockpile sampling and disposal. The project execution will be performed under the general supervision of Rick Meagher P.E., Chief of Environmental Design Section. The technical team consists of the following personnel:

Technical Team Leader:	Kathy Siebenmann	(916) 557-7180
Sampling Team Leader:	Paige Caldwell	(916) 557-6903
Chemist:	Kathy Siebenmann	(916) 557-7180
Health & Safety:	Donna Maxey	(916) 557-7437

USACE fax number: (916) 557-7465

2.2 Project Management

2.2.1 Project Leader

The Project Leader will be responsible for reviewing the sampling plans and associated field activities, and ensuring that all sampling activities conform to the QAPP. The Project Leader will oversee quality assurance of field activities. Prior to the start of field activities, preparatory meetings will be held with the field crew. If field conditions require modifications to protocol outlined in the SAP or if questions arise, the Sampling Team Leader or field crew will contact the Project Leader for direction. The Project Leader will also be responsible for overseeing the project and subcontractors, directing field crews, and the compilation of data. The Project Leader reports to the Section Chief.

2.2.2 Project Chemist

The Project Chemist will have a “hands on” role in management of project tasks associated with sampling and analysis. These tasks include:

- Coordination with the analytical laboratory to ensure readiness to implement project specific requirements,
- Review of analytical data as it becomes available to ensure conformance with quality standards, and
- Implementation of corrective actions in accordance with QAPP specifications when review of data uncovers deficiencies.

2.2.3 Health and Safety Manager

The certified industrial hygienist is responsible for the general health and safety plan development and training for field personnel. This individual is also responsible for ensuring that health and safety procedures are understood and followed by all field personnel, and for reporting and correcting any violations of policy or regulations.

2.2.4 Sampling Team Leader

The Sampling Team Leader will be responsible for quality assurance of field activities and for executing all work elements related to the sampling program, including documenting field activities, maintaining field notes and photographs, maintaining a record of onsite personnel and visitors, and implementing the sampling plan. These tasks include instruction of field personnel in sampling and preservation requirements and general oversight of field personnel involved in sampling activities.

2.2.5 Field Crew

Field crew personnel will be responsible for performance of project mobilization, demobilization, sample collection and oversight. Field personnel will report to the Sampling Team Leader. Field personnel will include members of the USACE Environmental Design Section, Sacramento District.

3.0 QUALITY OBJECTIVES FOR ENVIRONMENTAL DATA

3.1 Characteristics of Data Quality

The term “data quality” refers to the level of uncertainty associated with a particular data set. Data quality associated with environmental measurement is a function of the sampling plan rationale and procedures used to collect the samples, as well as of the analytical methods and instrumentation used in making the measurements. Uncertainty cannot be eliminated entirely from environmental data. However, quality assurance programs effective in measuring uncertainty in data are employed to monitor and control excursions from the desired data quality objectives (DQOs). The DQO process and data needs are specified in Attachment A. Sources of uncertainty that can be traced to the sampling component are poor sampling plan design, incorrect sample handling, faulty sample transportation, and inconsistent use of standard operating procedures. The most common sources of uncertainty that can be traced to the analytical component of the total measurement system are calibration and contamination.

The purpose of this QAPP is to ensure that the data collected are of known and documented quality and useful for the purposes for which they are intended. The procedures described are designed to obtain data quality indicators for each field procedure and analytical method. Data quality indicators include the PARCC parameters (i.e., Precision, Accuracy, Representativeness, Comparability, and Completeness). To ensure that quality data continues to be produced, systematic checks must show that test results and field procedures remain reproducible and that the analytical methodology is actually measuring the quantity of analytes in each sample.

A laboratory certified by the State of California and validated by the USACE will generate all laboratory chemical data. Laboratories must have an in-place program for data reduction, validation, and reporting as discussed in Section 7.0. The reliability and credibility of analytical laboratory results can be corroborated by the inclusion of a program of scheduled replicate analyses, analyses of standard or spiked samples, and analysis of split samples with QA laboratories for some projects. Regularly scheduled analyses of known duplicates, standards, and spiked samples are a routine aspect of data reduction, validation, and reporting procedures.

All data that will be collected for this project will be definitive data for organics/inorganics using EPA procedures and should be usable in stockpile characterization and engineering design. The data obtained will conform to the quality control requirements specified in the following text and the tables accompanying this document.

4.0 SAMPLE ACQUISITION, CUSTODY, MANAGEMENT, AND DECONTAMINATION

Sample acquisition, custody, management, and decontamination procedures are described in the Field Sampling Plan (FSP).

The samples will be sent to a State of California and USACE certified or National Environmental Laboratory Accreditation Conference (NELAC) audited laboratory. The USACE certification includes in-depth audits to determine if quality assurance and quality control measures are in place and adequate. These audits are based upon many of the same elements as the NELAC audits. The address and point of contact are listed below.

POC: Anna Pajarillo
Curtis and Tompkins, Ltd.
2323 Fifth Street
Berkeley, California 94710
Phone: (510) 486-0925 #103
Fax: (510) 486-0532

5.0 ANALYTICAL METHODS AND CALIBRATION

This section contains brief descriptions of preparation and analytical methods that will be used to analyze soil samples collected for this project. These methods are listed in Table 5-1.

Table 5-1. Summary of Analytical Methods

Analytes	Preparatory	Analytical Methods
Metals	SW3050B	SW6010B/SW7471A
TPH-purgeable (gasoline range – C5-C12)	SW5035/SW5030	SW8015B
TPH-extractable (diesel - C12-C24) (motor oil - C24-C36)	SW3550B, SW3630C	SW8015B
Organochlorine Pesticides	SW3550B, SW3630C	SW8081A
Polynuclear Aromatic Hydrocarbons (PNAs)	SW3550B	SW8270C Modified
Trichloroethene and breakdown products	SW5035/SW5030	SW8260B

Unless authorized by the Project Chemist, the most current promulgated method shall be utilized. If during the course of a project, it becomes necessary to apply a different quantitation limit because of changes in instrument capabilities, the Project Chemist will be notified and approval must first be obtained in instances where higher quantitation limits result. Methodology references contain specific QC criteria associated with the particular methods. These specific requirements include calibration and QC samples, and are described in detail within the methods. Daily performance tests and demonstrations of precision and accuracy are required.

The laboratory methods identified in this document were published by the United States Environmental Protection Agency (U.S. EPA) in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846*, Third Edition (November 1986; Revision 1, July 1992; and Revision 2, November 1992, Update I, August 1993, Update II, September 1994, Update III, 1998). Preservation and holding times for these analytical procedures are presented in Table 5-2. Attachment A summarizes the calibration and the internal quality control procedures; Attachment B lists the quantitation limits and Action Goals that will be used for this project.

Table 5-2. Preservation and Holding Times

Method	Chemical Preservation	Holding Time	Temperature Preservation
SW8015B (TPH-Purgeable)	None	2 days to preservation or freezing, 7 days from preservation to analysis	Cool to 4°C
SW8015B (TPH-Extractable)	None	14 days before extraction, 40 days after extraction	Cool to 4°C
SW8081A	None	14 days before extraction, 40 days after extraction	Cool to 4°C
Modified SW8270C	None	14 days before extraction, 40 days after extraction	Cool to 4°C
SW8260B	None	2 days to preservation or freezing, 7 days from preservation to analysis	Cool to 4°C
SW6010B	None	40 days before digestion, 6 months after digestion	None
SW7471A	None	28 days to analysis	Cool to 4°C

5.1 Sample Preparation and Analytical Methods - Organic

The following sections briefly summarize the sample preparation and analytical methods to be performed for the determination of organic analytes. Various cleanup methods may be used, depending upon the interferences encountered following extraction. Not all potential cleanup methods are included below. The Project Chemist should be advised of any alternative cleanup methods proposed by the laboratory.

5.1.1 Method SW3550B: Sonication Extraction

Method 3550B is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, wastes, and sludges. The sonication process ensures intimate contact of the sample matrix with the extraction solvent. A weighted portion of the solid material is mixed with the anhydrous sodium sulfate, ground to form a free-flowing powder, and then dispersed into the methylene chloride. The extract is separated from the sample by vacuum or gravity filtration, or centrifugation, and then dried with anhydrous sodium sulfate and concentrated to an appropriate volume for analysis.

5.1.2 Method SW3630C: Silica Gel Cleanup

Generally, solid-phase extraction cartridges filled with silica gel are used. Aliquots of sample extract are loaded onto the cartridges that are then eluted with suitable solvents, depending upon the analysis method. The collected fractions are analyzed by the appropriate method.

5.1.3 Method SW3640A: Gel-Permeation Cleanup

The extract is passed through a column containing a hydrophobic gel absorbent. The column is then flushed with clean organic solvents to separate the interferences from the analytes of interest by retention time.

5.1.4 Method SW5035: Closed System Purge and Trap Method

Method SW5035 is used to determine the concentration of VOCs and TPH-P in solid matrices. It is a closed-system purge-and-trap gas chromatographic procedure. The success of this method depends on the level of interference in the sample. For this project, EnCore™ samplers will be used to sub-sample stainless steel sleeves for VOCs. Three EnCore™ samplers will be collected for each sample and test required - one sampler for low-level analysis, one for back up, and one for methanol preservation (medium level) upon receipt. The methanol-preserved sample will be used if VOC concentrations in the low-level analysis exceed 200 µg/kg. The low concentration soil samples are weighed in the field to approximately 5 grams, and are preserved with sodium bisulfate usually in the laboratory. The vial is sealed and sent to the laboratory. Prior to analysis, organic-free reagent water, surrogates and internal standards (if applicable) are added to the sample, without opening the vial. The vial is heated, and the vapors are purged into an appropriate trap. For expected concentrations greater than 200 µg/kg, the high concentration method is used. For the high concentration sample, 5 milliliters (mLs) of methanol is added to the sample vial. At the laboratory, surrogates (and internal standards) are added, and then an aliquot of the extract is purged using Method SW5030. An inert gas is bubbled through the sample 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 gas chromatographic column for analysis.

5.1.5 Method SW8015B: Total Petroleum Hydrocarbons – Purgeable and Extractable

Method SW8015 is used to determine gasoline, diesel, and residual range organics quantitated as gasoline or diesel. The purgeable component of TPH consists of those hydrocarbons in the gasoline boiling or carbon range. The extractable component of TPH consists of those hydrocarbons in the diesel fuel and motor oil boiling or carbon range.

For analysis of the TPH-P component, the sample is collected and prepared for analysis using Method SW5035 for soils as described above. For analysis of the TPH-E component, the sample is first extracted following Method SW3540 or SW3550 for soil or sludge matrices. Analysis is accomplished on a gas chromatograph (GC) equipped with a capillary or megabore column and flame ionization detector (FID) and photoionization detector (PID).

The chromatograms consist of groups of peaks that have a general shape or pattern and fall within a noted carbon range (i.e., number of carbon atoms in the molecule). Diesel fuel will be used to calibrate the instruments and determine response factors for quantitation of TPH-E (C12 through C26) and motor oil (C26 through C40). Gasoline will be used to calibrate the instruments and determine response factors for quantitation of TPH-P (C6 through C12). No second-column confirmation will be performed because identification is based on pattern recognition and not retention time (where false positives due to interference are likely). In addition, the patterns and carbon ranges of other petroleum hydrocarbons listed above will be used to compare to sample chromatograms for identification. Often, unknown or un-calibrated hydrocarbons are encountered; therefore, the concentration reported is considered estimated. Carbon ranges and significant deviations of the pattern from the patterns of reported analytes will be described in the analytical report.

Quantitation of both standards and samples will be performed by adding the area from all peaks from the baseline of the entire chromatogram. In cases where the range of the pattern in the sample extends outside of the gasoline or diesel fuel standard ranges, the area throughout the range of the sample pattern will be quantitated (relative to gasoline or diesel).

5.1.6 Method SW8081A: Organochlorine Pesticides

Method SW8081A is used to determine the concentration of various organochlorine pesticides. Prior to analysis, the sample is extracted into solution. An aliquot of solution is injected into an open-tubular capillary column, and detected by an electron capture detector (ECD) or electrolytic conductivity detector (ELCD). Any compounds identified tentatively in the primary analysis are confirmed on a second GC column.

5.1.7 Modified Method SW8270C: Polynuclear Aromatic Hydrocarbons by GC/MS Selective Ion Monitoring

Method SW8270C is used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride. Such compounds include polynuclear aromatic hydrocarbons (PAHs). The concentrated extract is injected into a gas chromatograph for separation and detected by mass spectrometry. Mass spectrometry provides a characteristic ion pattern for fragmented target analytes, providing a high level of confidence in compound identification. Compounds are quantitated by comparing the response of a characteristic ion to the average response from a 5-point calibration. The internal standard technique is used for calibration. The instrument will be modified for selective ion monitoring (SIM) to reduce interferences and lower the quantitation and detection limits of PAHs for this project. Aliquot of the extract is injected into a GC/MS that is set up to detect only specific ions found in the PAH analytes.

5.1.8 Method SW8260B: Trichloroethene and breakdown products

Method SW8260 is used to determine volatile organic compounds in a variety of matrices. The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection. Purged sample components are trapped in a tube containing suitable sorbent materials as described in Method SW5030. Once the components are desorbed onto the capillary column, the column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer interfaced to the gas chromatograph. Usually the average response factor is used for quantitation but linear regression is also acceptable. Method SW8260 will be used to analyze soil for TCE and breakdown products only.

5.2 Sample Preparation and Analysis Methods - Inorganic

The following sections briefly summarize the sample preparation and analysis methods to be performed for the determination of inorganic analytes.

5.2.1. Method SW3050B: Acid Digestion of Sediments, Sludges, and Soils

This digestion procedure is used for the preparation of solid samples for analysis by inductively coupled plasma/atomic emission spectroscopy (ICP). A mixture of nitric acid, and the material to be analyzed is refluxed in a covered Griffin beaker or equivalent. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. Hydrogen peroxide is then added and the mixture warmed. The digestate is then cooled and brought to a low volume with water. If the digestate contains suspended solids, it must be centrifuged, filtered, or allowed to settle before analysis.

5.2.2 Method SW6010B: Inductively Coupled Plasma-Atomic Emission Spectrometry

ICP determines elements in solution. The sample requires digestion by Method SW3050B for soil prior to analysis.

The method provides a simultaneous or sequential multi-element determination of elements by ICP. Element-emitted light is measured by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic line emission spectra are produced by radio frequency inductively coupled plasma. The spectra are dispersed and photo-multiplier tubes monitor the intensities of the lines. The spectra are the physical property of the element and the intensity is proportional to the concentration of the element in solution.

5.2.3 Method SW7471A: Cold Vapor Atomic Absorption Spectroscopy

Method SW7471A is based on the absorption of radiation at the 253.7 nm wavelength by mercury vapor. The 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 atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Quantitation is accomplished by comparing the absorbance to a five-point calibration curve prepared from standards of known mercury concentration.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

6.1 Calibration Procedures and Frequency

All instruments and equipment used during sample analysis are operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations, as well as criteria set forth in the applicable analytical methods. Personnel properly trained in these procedures will operate, calibrate, and maintain the instruments. Laboratory capabilities will be demonstrated initially for instrument and reagent/standards performance as well as accuracy and precision of analytical methodology.

Calibration of instruments is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established quantitation limits. Each instrument will be calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method presented in Section 5.0. The frequency of calibration and calibration verification and the concentration of calibration standards are determined by the manufacturer's guidelines and the analytical method. Calibration procedures for all instruments are summarized in the method-specific tables in Attachment A. All samples must be bracketed by passing calibration check samples for the majority of methods. Failure to bracket all samples with acceptable calibration checks may result in the reanalysis of affected samples.

6.1.1 Gas Chromatography

The field of chromatography involves a variety of instrumentation and detection systems. While calibration standards and acceptance criteria vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply uniformly. As outlined in EPA SW-846 procedures, each chromatographic system is calibrated prior to performance of analyses using five concentrations by external standard technique for all columns. The lowest calibration standard shall be within a factor of two relative to the QL, and the others corresponding to the expected range of concentrations or defining the working range of the detector. This is done on each chromatographic column and each instrument at the beginning of the contract period and each time a new column is installed. The results are used to determine a calibration curve and response factors for each analyte. Initial calibration consists of determining the working range, establishing limits of detection, and establishing retention time windows. The calibration is checked on a daily basis to ensure that the system remains within specifications. Second column confirmation is required for single compound analytes.

Continuing calibration standards are analyzed to check the instrument response relative to the initial calibration curve at the beginning and end of each analytical run. Calibration checks are also performed for overall system performance and for retention time shifts, as specified in SW-846. Individual and standard mixes are analyzed to establish response factors and absolute retention time. The response factors and retention times are verified throughout the analytical run and at the end of the analytical sequence. Each analyte must be within its retention time window or the analyst shall take corrective action. For GC analyses conducted on this project, the response factor must agree with the factor determined during the initial 5-point calibration withiṇ 15% for quantitation analysis utilizing SW-846 methodology.

The instrumental detection limit, the linear range of the instrument, and interference effects must be established for each individual analyte on that particular instrument. The calibration is verified initially prior to sample analysis using an independent second source standard. Calibration verification standards are analyzed after every 10 samples using a midrange calibration check standard and must be withiṇ 15% of the expected value.

6.1.2 GC/MS analysis

Each day prior to analysis of samples, the instrument is tuned with bromofluorobenzene for volatile compounds and decafluorotriphenylphosphine for semivolatile compounds or other tuning criteria as specified by the method used. Mass spectral peaks must conform both in mass numbers and relative intensity to method-specified requirements before analyses can proceed.

The instrument is then calibrated for all target compounds. An initial calibration curve is produced to define the working range to establish criteria for identification. All GC/MS instruments are calibrated at five different concentrations for analytes of interest, using the procedures outlined in SW-846. Method system performance check compounds (SPCC's) must show a minimum mean response factor and method calibration check compounds (CCC) must show a relative standard deviation (RSD) less than the method specified standard for the initial calibration to be considered valid. On a daily basis, SPCC's must meet the same criteria relevant for the initial calibration and CCCs must show a minimum percent drift relative to the expected concentration of the CCC to be considered valid. This initial calibration is evaluated on a daily basis to ensure that the system is within calibration. If the daily standard does not meet the established criteria, the system is recalibrated. These procedures will be modified for selective ion monitoring.

Following a successful tune, the initial five-point calibration is verified by a single mid-range concentration standard. The calibration is verified daily prior to sample analysis using an independent second source standard. This initial calibration can be utilized as long as the calibration verification remains valid.

6.1.3 Inductively Coupled Argon Plasma-Atomic Emission Spectrometry (ICPES) Metals

Plasma emission spectrophotometry, also termed inductively coupled argon plasma (ICP) spectrometry, is calibrated daily using either one standard solution and one blank or a four-point calibration (3 levels plus blank). For the single standard calibration, the calibration standard must be within the demonstrated linear range of the instrument. The instrumental detection limit, the linear range of the instrument, and interference effects must be established for each individual analyte on that particular instrument. The linear range is verified at the time of the analysis by analyzing the highest calibration standard as a sample, the results of which must be within ± 5% of its true value. The calibration is verified initially prior to sample analysis using an independent second source standard at a concentration mid-range of the calibration. Continuing calibration checks are analyzed after every 10 samples using a mid-range calibration check standard and must be within ±10% of the expected value. Sensitivity is established at the lower calibration level by analyzing a low level standard at the QL (3 to 5 times the MDL). Calibration blanks are analyzed after all calibration check standards and no analytes may be detected above one-half the QL. An interelement check standard is analyzed at the beginning and end of each analytical run, to verify that interelement and background correction factors have remained constant. Results outside of the established criteria trigger reanalysis of samples.

6.1.4 Atomic Absorption Spectroscopy

The instrument must be calibrated and checked for contamination before each set of samples. An initial calibration (ICAL) consists of a minimum of a blank and three calibration standards. The least concentrated standard will be at a concentration corresponding to the QL. The remaining standards will define the working range of the instrument. A linear regression fit of the calibration data must yield a correlation coefficient must be at least 0.995. Failure to meet these criteria will require recalibration and possible preparation of a new set of standards. Prior to sample analysis, an initial calibration verification (ICV), consisting of a second source standard, and an initial calibration blank (ICB) will be analyzed to verify the quantitation and to detect any contamination. A continuing calibration verification (CCV) at a mid-curve concentration and CCB will be analyzed very 10 samples and at the end of analytical sequence. If the CCV value varies from the predicted concentration by more than + 10% then the analysis must be stopped. The problem must be identified and corrected, and rerun the impacted samples. All samples must be bracketed by calibration standards that meet the stated criteria.

6.2 Standard and Reagent Preparation

A critical element in the generation of quality data is the purity and traceability of the standard solutions and reagents used in the analytical operations. The preparation and maintenance of standards and reagents will be performed per the specified analytical methods presented in Section 5.0. The laboratory shall continually monitor the quality of reagents and standard solutions through a series of well-documented standard operating procedures (SOPs). In general, SOPs for standards preparation should incorporate the following items:

- Documentation and labeling of date received, lot number, date opened, and expiration date;
- Documentation of tracability;
- Preparation, storage, and labeling of stock and working solutions; and
- Establishing and documenting expiration dates and disposal of unusable standards.

Primary reference standards and standard solutions used by the laboratory are to be obtained from the National Institute of Standards and Technology, or other reliable commercial sources to ensure the highest level of purity possible. All standards and standard solutions shall be catalogued to identify the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information included in the specific SOP.

6.3 Standard Solutions and Reagents

Standard solutions and reagents are validated prior to use. Validation procedures can range from a check for chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time, concentration or source. Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory. Stock and working standards are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is to be exercised in the proper storage and handling of standard solutions, and all containers are labeled as to compound, concentration, solvent, expiration date, and preparation data (initials of the preparer/date of preparation).

6.4 Field Quality Control Checks

Quality control checks in the field will include the collection of field duplicate, equipment rinsate and temperature blank samples. These QC checks are described in Section 4.2 of the FSP.

6.5 Laboratory Quality Control Checks

The Project Laboratories will have a QA/QC program that monitors data quality with internal QC checks. Internal QC checks are used to answer two questions:

1) Are laboratory operations in-control, (i.e., operating within acceptable QC guidelines), during data generation?

2) What effect does the sample matrix have on the data being generated?

Laboratory performance QC is based on the use of a standard control matrix to generate precision and accuracy data that are compared, on a daily basis, to control limits. This information, in conjunction with method blank data, is used to assess daily laboratory performance.

The second question is addressed with Matrix-Specific QC. Matrix-Specific QC is based on the use of an actual environmental sample for precision and accuracy determinations and commonly relies on the analysis of matrix spikes and matrix spike duplicates. This information, supplemented with field blank results, is used to assess the effect of the matrix and field conditions on analytical data.

Laboratory Performance QC will be provided as a standard part of every routine analysis. Matrix-Specific QC will be required per the guidance documents presented in Section 5.0. A brief summary of the required QC samples follows. The type and frequency of QC samples performed by the laboratory will be according to the specified analytical method.

6.5.1 Analytical Batch (Preparation Batch)

The analytical batch is defined as a preparation batch. The analytical batch will not exceed 20 samples and is defined as a set of samples that are extracted/analyzed concurrently or sequentially. Significant gaps (greater than two hours) in the analytical sequence will result in the termination of the previous sequence and the initiation of a new analytical sequence. The analytical batch shall be analyzed sequentially on a single instrument. The practice of "holding a batch open" and performing a single set of batch QC samples for all analyses performed during that period is unacceptable.

The laboratory shall, at a minimum, analyze internal QC samples at the frequency specified in this QAPP for all analytical methods. These QC samples for each analytical batch shall include method blanks (MB), MS/MSD analyses, and laboratory control samples (LCS). Definitions for the QC samples described above are provided in Chapter 1, Update III to EPA SW-846. The matrix used for LCS analyses shall be reagent grade water for aqueous analyses and reagent sand for soil/sediment matrices.

Second column confirmation for all GC sample analyses involving identification of discrete peaks with detected concentrations will be required, as per the methods. Second column confirmation is not required for concentrations reported between the MDL and the QL.

6.5.2 Blanks

Two types of blanks routinely analyzed in the laboratory are method blanks and reagent blanks. Method blanks and reagent/solvent blanks are used to assess laboratory procedures as possible sources of sample contamination.

Method or preparation blanks for all samples consist of deionized water or reagent sand that is subjected to the entire analytical procedure, including extraction, distillation, digestion, etc., as appropriate for the analytical method being utilized. One method blank will be analyzed for each analytical batch (minimum of one per day; one every 12 hours for GC/MS analyses). If the blank does not meet acceptance criteria, the source of contamination will be investigated and appropriate corrective action will be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on the sample results. Corrective actions may include reanalysis of the blank and/or repreparation and reanalysis of the blank and all associated samples. No method blank may exhibit a detected concentration greater than the quantitation limit. However, exceptions may be made when the analyte is not detected in the related sample. Sample results are not corrected for blank contamination unless required by the analytical method.

Reagent/solvent blanks consist of individual reagents or solvents subjected to the entire analytical procedure as appropriate for the analytical method being utilized. The blanks are only used if contamination problems are indicated by the method blank or if a new lot of materials are being checked before use.

6.5.3 Laboratory Control Samples

Laboratory control samples (LCS) are used as a means of evaluating the efficiency of the analytical process. As discussed above, LCS is used to generate precision and accuracy data that are compared, on a daily basis, to control limits. Laboratory control samples are subjected to the entire sample procedure, including extraction, digestion, etc., as appropriate for the analytical method utilized. They are generally introduced into an analytical batch (20 samples) immediately before extraction or analysis. LCS samples will be performed for both inorganic and organic laboratory methods.

6.5.4 Matrix Spikes and Matrix Spike Duplicates

A Matrix Spike (MS) is an environmental sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

A Matrix Spike Duplicate (MSD) is a duplicate of the environmental sample described above, each of which is spiked with known concentrations of analytes. The two spiked samples are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as relative percent difference (RPD) and percent recovery (%R).

6.5.5 Surrogate Recoveries and Standard Additions

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. Surrogates are added to samples to monitor the effect of the matrix on the accuracy of the analysis. Results are reported in terms of percent recovery. Laboratories routinely add surrogates to samples requiring GC or GC/MS analysis and report these surrogate recoveries to the client. The laboratory does not modify its operations based on surrogate recoveries in environmental samples. The surrogate recoveries are primarily used by the laboratory to assess matrix effects. However, obvious problems with sample preparation and analysis (e.g. evaporation to dryness, leaking septum, etc.) which can lead to poor surrogate spike recoveries must be ruled out prior to attributing low surrogate recoveries to matrix effects.

Standard Additions is the practice of adding a series of known amounts of an analyte to an environmental sample. The fortified samples are then analyzed and the recovery of the analytes calculated. The practice of standard addition is generally used with metals analysis and wet chemistry to determine the effect of the sample matrix on the accuracy of the analyses.

6.5.6 Calibration Standard

A calibration standard is prepared in the laboratory by dissolving a known amount of a purchased pure compound or standard mix in an appropriate matrix. The final concentration calculated from the known quantities is the true value of the standard. The results obtained from these standards are used to generate a standard curve and thereby quantify the compound in the environmental sample.

6.5.7 Reference Standard

A reference standard is prepared in the same manner as a calibration standard or may be obtained from National Institute of Standards and Testing (NIST). A reference standard is obtained from a source independent of the source of the calibration standard. The concentration of the known quantity is the "true" value of the standard. A reference standard is not carried through the same process used for the environmental samples, but is analyzed without digestion or extraction. A reference standard result is used to validate an existing concentration calibration standard file or calibration curve. The reference standard can provide information on the accuracy of the instrumental analytical method independent of various sample matrices.

6.5.8 Laboratory Performance Evaluation Samples

At a minimum the contract laboratory will participate in at least one performance evaluation program.

The performance evaluation samples are single blind (prepared by the laboratory from ambulated standards) and are often associated with the regular laboratory audits performed by the agencies.

6.6 Corrective Action

The Project Leader is responsible for initiating corrective action and for implementation of all corrective actions with respect to the field sampling operations. The laboratory QA Director in consultation with the Project Chemist is responsible for implementing corrective actions in the laboratory. It is their combined responsibility to see that all analytical and sampling procedures are followed as specified and that the data generated meet the acceptance criteria. The acceptance criteria for many of the QC samples (LCS, MS, surrogate recoveries) will be those calculated by the laboratory as control limits. The number of samples used to develop the statistical control limits shall be all those analyzed within the previous six months or a minimum of 20 datapoints. The comparison control limits in Attachment A are to ensure that the laboratory can produce data with acceptable accuracy. If the laboratory statistical limits are consistently different from the comparison limits, a different laboratory shall be selected for that analytical method, or an alternate analytical or preparation method shall be selected that increases the accuracy of the laboratory. Corrective action procedures are summarized for each method in Attachment A.

Corrective actions for the laboratory may include, but are not limited to:

- Reanalyzing samples;
- Correcting laboratory procedures;
- Recalibrating instruments using freshly prepared standards;
- Replacing solvents or other reagents that give unacceptable blank values;
- Training laboratory personnel in correct sample preparation and analysis procedures; and
- Accepting data with an acknowledged and documented level of uncertainty.

Whenever corrective action is deemed necessary, the Laboratory Director will ensure that the following steps are taken:

- The problem is defined;
- The cause of the problem is investigated and determined;
- Appropriate corrective action is determined; and
- Corrective action is implemented and its effectiveness verified.

6.7 Documentation

All calibration information, instrument maintenance and repair are recorded by the laboratory on appropriate forms developed for SW-846 procedures. Out-of-control analyses are generally described on a QA/QC discrepancy form and submitted to the laboratory supervisor for corrective action. Copies are distributed to the laboratory QA coordinator and laboratory director for approval, and to the case file. The calibration information is filed with the raw data in the reports area.

7.0 DATA REDUCTION, VALIDATION AND REPORTING

7.1 Laboratory

7.1.1 Data Reduction and Validation

All analytical data generated within the laboratories shall be reviewed prior to report generation to assure the validity of the reported data. The data validation process consists of data generation, reduction, and three levels of documented review. In each stage, the review process will be documented by the signature of the reviewer and the date reviewed.

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

- The correct samples were analyzed and reported in appropriate units,
- Preservation and holding time requirements were met,
- Sample preparation information is correct and complete,
- Appropriate SOPs have been followed,
- Analytical results are correct and complete,
- QC samples are within established control limits,
- Blanks are within appropriate QC limits,
- Special sample preparation and analytical requirements have been met, and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, anomaly forms are complete; holding times are documented, etc.).

The data reduction and validation steps shall be documented, signed and dated by the analyst. The analyst will then pass the data package to an independent reviewer, who will perform an independent review of the data package. This review is also to be conducted according to an established set of guidelines and to be structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented,

- QC samples are within established guidelines,
- Qualitative identification of sample components is correct
- Quantitative results are correct,
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; anomaly forms are complete; holding times are documented, etc.), and
- The data are ready for incorporation into the final report; and the data package is complete and ready for data archive.

The review is to be structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples will be checked to the bench sheet. This process will continue until no errors are found or until the data package has been reviewed in its entirety.

Data reviews shall be documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared. Before the report is released to the client, the data are reviewed for completeness and to ensure that the data satisfy the overall objectives of the project. This review is typically done by the Laboratory Project Manager.

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

7.1.2 Data Reporting

At the conclusion of all analytical work for this project, the primary laboratory will submit a comprehensive certificate of analysis. The final certificates of analysis will be submitted no later than 21 days after the last sample has been submitted to the laboratory for the project. All samples shall be reported in a legally defensible package.

The data package for organics analyses will consist of a case narrative, chain-of-custody documentation, cooler receipt form, summary of results for environmental samples, summary of QA/QC results, and the data. Legible copies of all data will be organized systematically on numbered pages. The data for compound identification and quantitation must be sufficient to support all results presented in other sections of the data package. This section of the data package will include legible copies of the data for environmental samples (arranged in increasing order of field ID), and instrument calibration, QA/QC analyses, sample extraction and cleanup logs, instrument analysis logs for each instrument used. Instrument analysis logs are particularly important because they provide the basic link between all sample analyses and QC information (calibration, matrix spike, etc.). Instrument analysis logs for all instruments used for sample data for each analysis will include measurement printouts and quantitation reports for each instrument used.

Raw data will be available for further inspection, if required, and maintained in the central job file. All records related to the analytical effort are maintained at the primary laboratory in secured filing cabinets (i.e., cost information, scheduling, and custody). All records are maintained for five years after the final report is issued. Types of records to be maintained for the project include the following:

- Chain-of-custody records, including: information on the sampler's name, date of sampling, type of sampling, location of sampling, location of sampling station, number and type of containers used, signature of sampler relinquishing samples to non-contract personnel (e.g., Federal Express agent) with the date and time of transfer noted, signature of primary laboratory sample custodian receiving samples with date and time noted
- Cooler receipt form documenting sample conditions upon arrival at the laboratory.
- Any discrepancy/deficiency report forms due to problems encountered during sampling, transportation, or analysis
- Sample destruction authorization forms containing information on the manner of final disposal of samples upon completion of analysis
- All laboratory notebooks including raw data readings, calibration details, QC checks, etc
- Hard copies of data system printouts (chromatograms, mass spectra, ICP data files, etc.)

- Tabulation of analytical results with supporting quality control information

7.1.2.1 Case Narrative

The case narrative will be written and the release of data will be authorized by the laboratory director or his/her designee. Items to be included in the case narrative are the field sample ID with the corresponding laboratory ID, parameters analyzed in each sample and the methodology used (EPA method numbers or other citation), detailed description of all problems encountered and corrective actions taken, discussion of possible reasons for out-of-control QA/QC results, and observations regarding any occurrences which may affect sample integrity or data quality.

7.1.2.2 Chain-of-Custody Documentation

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

7.1.2.3 Summary of Environmental Results

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

7.1.2.4 Summary of QA/QC Results

The following QA/QC results will be presented in summary form. Details specified in Section 7.1.2.3 also will be included for the summary of QA/QC results. Acceptance limits for all categories of QC criteria will be provided with the data.

7.1.2.4.1 Organic Analyses (General)

The summary of QA/QC results for organic analyses will include:

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

- Daily Calibration and Mid-level Standard - The concentration of the calibration standard used for daily calibration and/or the mid-level calibration check will be reported. The response factor, percent difference, and retention time for each analyte will be reported (GC and GC/MS). Daily calibration information will be linked to sample analyses by summary.
- Method Blank Analyses - The concentrations of any analytes found in method blanks will be reported even if detected amounts are less than the QL. The environmental samples and QA/QC analyses associated with each method blank will be stated.
- Surrogate Standard Recovery - The name and concentration of each surrogate compound added will be detailed. The percent recovery of each surrogate compound in the samples, method blanks, matrix spike/matrix spike duplicates and other QA/QC analyses will be summarized with sample IDs such that the information can be linked to sample and QA/QC analyses.
- Precision and Accuracy - For matrix spike/matrix spike duplicate analyses, the sample results, spiked sample results, percent recovery, and RPD with the associated control limits will be detailed. For laboratory duplicate analyses, the RPD between duplicate analyses will be reported as applicable. For laboratory QC check and/or LCS analyses, the percent recovery and acceptable control limits for each analyte will be reported. All batch QC information will be linked to the corresponding sample groups.
- Compound Identification (GC, HPLC, GC/MS): The retention times and the concentrations of each analyte detected in environmental and QC/QC samples will be reported for both primary and confirmation analyses.
- Method Detection Limit (MDL): The MDL study result sheet will have laboratory heading, instrument identification, analysis date, spike level, average recovery, standard deviation and calculated MDL for each analyte.

In addition, the summary of QA/QC results for organic analyses will include the following information relating specifically to the method used.

7.1.2.4.2 GC and GC/MS Analyses

This section of the data package will include legible copies of the data for environmental samples (arranged in increasing order of field ID, primary and confirmation analyses). The raw data for each analysis will include chromatograms (with target compound, internal standard, and surrogate compounds labeled by name) with a quantitation report and/or area printout. GC/MS analyses will also include the mass spectra or ion chromatograms for each reported analyte.

7.1.2.4.3 Inorganic Analyses

The summary of QA/QC results for the inorganic analyses will include:

- Initial Calibration: The source of the calibration standards, true value concentrations, found concentrations, the percent recovery for each element analyzed, and the date and time of analysis will be reported.
- Continuing Calibration Verification: The source of the calibration standard, true value concentrations, found concentrations, the percent recovery for each element analyzed, and the date and time of analysis will be reported.
- Method Blank Analyses: The concentrations of any analytes found in initial calibration, continuing calibration blank, and in the preparation blank will be reported. The date and time of analysis also will be reported.
- Precision and Accuracy - Matrix Spikes and Sample Duplicates: For matrix spike analyses, the sample results, spiked sample results, percent recovery, spiking solution used, and the control range for each element will be detailed. For post digestion spikes, the concentrations of the spiked sample, the sample result, the spiking solution added, and recovery and control limits will be detailed. For laboratory duplicates, the original concentration, duplicate concentration, relative percent difference, and control limits will be detailed. Date and time for all analyses will be recorded.
- Precision and Accuracy - Laboratory Control Samples: The source of the laboratory control sample, true value concentrations, found concentrations, percent recovery for each element analyzed, and the date and time of analysis will be reported.
- Method of Standard Additions (MSA): This summary must be included when MSA analyses are required for analysis by Graphite Furnace AA. The absorbance values and the corresponding concentration values, the final analyte concentrations, and correlation coefficients will be reported for all analyses. Date and time of analysis will be recorded

for all analyses.

- Method Detection Limit (MDL): The MDL study result sheet will have laboratory heading, instrument identification, analysis date, spike level, average recovery, standard deviation and calculated MDL for each analyte.

7.1.3 Quality Assurance Reports

The laboratory data will be validated using guidelines in Attachment C. The validation guidelines are based on EPA SW-846 methods and the EPA National Functional Guidelines for Organic and Inorganic Data Review. The Project Chemist, or designee, will review the data and prepare a Quality Control Summary Report (QCSR). The QCSR presents all laboratory and field QC results and any qualifiers applied to the data. The Project Chemist will discuss the data usability and precision based upon all information that affects the quality of the data (not just laboratory QC results) in a Chemical Data Quality Assessment Report (CDQAR).

7.2 Field Activities

7.2.1 Data Reduction

Since no field screening equipment will be used during this sampling event, data reduction is not applicable.

7.2.2 Data Integrity

Integrity of information and data on field activities shall be maintained by the Project Leader. Integrity of the field sample custody is accomplished by the field staff, according to the sample custody procedures discussed in Section 5.0. This information is generated in the field and recorded in the project field logbook and on the sample chain-of-custody form, shall be verified before sample shipping, and confirmed at the laboratory upon their receipt of the samples.

7.2.3 Data Validation

Validation of information and data on field activities shall be conducted as a QC procedure by the Project Leader. The Project Manager shall review laboratory results and field data before use. Field logbooks and chain-of-custody forms shall be cross-checked to each other and to the laboratory results to assure conformity of sample identification numbers. This information is compared to results of duplicate and blank samples, and field conditions at the time of sample collection will be taken into account when qualifying the sample analytical results.

7.2.4 Data Storage

Field and laboratory data shall be stored in hard copy and floppy disk format (when applicable) as part of the project file. This information is retained in the project file until project completion and closeout. Upon project closeout, all records shall be archived for permanent storage.

8.0 PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. Each laboratory shall have detailed SOPs on file that describe preventive maintenance procedures and schedules. All service and maintenance will be conducted by qualified laboratory staff or under service agreement with the manufacturer or their approved agent. All repairs, adjustments, and calibrations will be documented in a maintenance notebook or data sheet that will be maintained in a permanent file. The instrument notebook will clearly document the date, the problem description, corrective action taken, results of actions, and the name of the person performing the work. Table 8-1 lists common laboratory preventative maintenance parameters for laboratory instrumentation.

Table 8-1. Routine Laboratory Instrument Maintenance

nstrument	peration	requency
Gas Chromatography	Change septum Change injection port liner Change column Bake detectors	Daily when used Daily when used As needed (when standard response decreases or sample carryover is noted, approximately monthly) As needed (when standard response decreases or sample carryover is noted , approximately monthly)
GC/MS	Clean source	As needed (show reduced sensitivity)
Atomic Absorption Spectrometer	Warm up instrument for 30 min. Digital readout values checked; check gas flows, cell alignment, wavelength, Photo multiplier voltage and lamp voltage Tygon tubing replaced Change contact rings Replace optical lens	Daily when used Daily when used Quarterly or as needed Daily, as needed or when used 6 months, or if deterioration is observed
Balances	Calibrate by manufacturer	Annually / verify monthly
Ovens/Refrigerators	Check temperature	Daily

9.0 LABORATORY PROCEDURES USED TO ASSESS DATA QUALITY AND DETERMINE SENSITIVITY

9.1 Data Quality Assessment

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its PARCC parameters as presented in Section 3.0. These terms are described as follows:

9.1.1 Precision

Precision is a measure of the reproducibility of analyses under a given set of conditions. Precision can be assessed by replicate measurements of duplicate control samples, reference materials, or environmental samples. The routine comparison of precision is measured by the relative percent different (RPD) between duplicate control sample measurements with control limits established at plus three standard deviations from the mean RPD of historical duplicate control sample data. The overall precision of a sampling event has a sampling and an analytical component. The following QC data will be collected to determine sampling and analytical precision:

- Laboratory Control Standards and duplicates (LCD/LCSD) as well as matrix spikes and matrix spike duplicates (MS/MSD) will be used as a measure the precision of the analytical process for organic analyses. LCS/LCSD and/or MS/MSD samples will be run on each batch of samples up to a maximum of 20.
- Field duplicate samples, submitted to the laboratory "blind", measure the precision of the entire measurement system including sampling and analytical procedures. Field duplicate samples will be collected at a rate of 1 per 10 primary samples.
- Laboratory duplicates will be performed for every inorganic analytical batch. The maximum size of each batch shall not exceed 20 samples.

The RPD between the two samples may be used to estimate precision where:

$$\text{RPD} = \frac{|D_1 - D_2|}{(D_1 + D_2)} \text{ x } 200$$

RPD = *absolute relative percent difference*

D_1 = *first sample value*

D_2 = *second sample value (duplicate)*

Note: If the laboratory determines that failure to meet QC criteria for accuracy or precision is a result of objectively verifiable matrix effects, no further re-extractions will be required. However, the narrative must contain an explicit description of the laboratory's rationale in this regard with reference to objectively verifiable features of raw data. The sufficiency of the laboratory's explanation will be determined by the Project Manager or an appointed representative.

9.1.2 Accuracy

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using laboratory control samples, standard reference materials, or spiked environmental samples. Unless specified otherwise in special contracts, the laboratory shall monitor accuracy by comparing laboratory control sample results with control limits established at plus or minus three standard deviation units from the mean of historical laboratory control sample results. The accuracy of the data submitted for this project will be assessed in the following manner:

- Accuracy for each sample will be checked by calculating surrogate percent recoveries, as applicable.
- The percent recovery of matrix spikes, matrix spike duplicates, and/or laboratory control samples will be calculated.
- The level of target compounds that are found (if any) in laboratory method blanks will be checked. If a target compound is found above the practical quantitation limit in the method blank corresponding to a batch of samples and the same target compound is found in a sample, the data will not be background subtracted but will be flagged to indicate the result in the blank.

Accuracy is presented as percent recovery. Since accuracy is often determined from spiked samples, laboratories commonly report accuracy as

$$\% \text{ Recovery } \frac{R}{S} \text{ x } 100$$

Where: S = spiked concentration

R = reported concentration

Note: If the laboratory determines that failure to meet QC criteria for accuracy or precision is a result of objectively verifiable matrix effects, no further re-extractions will be required. However, the narrative must contain an explicit description of the laboratory's rationale in this regard with reference to objectively verifiable features of raw data. The sufficiency of the laboratory's explanation will be determined by the Project Manager or an appointed representative.

9.1.3 Representativeness

Representativeness is a qualitative parameter that reflects the extent to which a given sample is characteristic of a given population at a specific location or under a given environmental condition. Representativeness is best satisfied by making certain that sampling locations are selected properly, a sufficient number of samples are collected, and an appropriate sampling technique is employed. Variations at a sampling point will be evaluated based on the results of field duplicates. Some samples may require analysis of multiple phases to obtain representative results. Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. Sample representativeness will also be evaluated on the basis of results from method blanks and trip blanks.

9.1.4 Completeness

Completeness will be evaluated qualitatively and quantitatively. The qualitative evaluation of completeness will be determined as a function of all events contributing to the sampling event including items such as correct handling of COC forms, incorporation of QC samples at the appropriate frequency, etc. The quantitative description of completeness will be defined as the percentage of contract laboratory controlled QC parameters that are acceptable. The goals for completeness are as follows: contract (95%), analytical (85%, technical (95%), and field sampling completeness (100%). Contract completeness is a measure of the results that meets contract requirements relative to the number of reported results expressed as a percentage. Analytical completeness is a measure of all unqualified results relative to the number of reported results expressed as a percentage. Technical completeness is a measure of the usable results relative to the number of reported results expressed as a percentage. Field sampling completeness is a measure of the number of samples collected relative to the number of samples planned expressed as a percentage.

9.1.5 Comparability

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. To ensure comparability, field procedures will be standardized and field operations will adhere to standard operating procedures. Laboratory data comparability will be assured by use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), and consistency in reporting units (µg/L, mg/kg, etc.). Analysis of standard reference materials will follow USEPA or other standard analytical methods, which utilize standard units of measurement, methods of analysis, and reporting format.

9.2 Sensitivity

9.2.1 Method Detection Limit (MDL)

The method detection limit (MDL) is the lowest concentration at which a specific analyte in a matrix can be measured and reported with 99-percent confidence that the analyte concentration is greater than zero. MDLs are experimentally determined for each target analyte of the method. Each individual instrument will maintain a current MDL study. MDLs are based on the results of seven spikes of clean matrix at the estimated MDL and are statistically calculated in accordance with the Title 40, Code of Federal Regulations Part 136 (40 CFR 136), Attachment B. The standard deviation of the seven replicates is determined and multiplied by 3.143 (i.e., the 99-percent confidence interval from the one-sided student t-test). The MDLs are updated annually and whenever significant instrument maintenance is performed (i.e., GC Column, AA lamp, etc.).

9.2.2 Quantitation Limit (QL)

The quantitation limit is defined by the lowest concentration in the multi-point initial calibration. The QL is the lowest level for quantitation decisions based on individual measurements for a given method and representative matrix. The QL for this project is based on a project-specific action level and the capability of the method and laboratory. Detected results above the MDL but below the QL, are qualified with a J flag due to the very low comparator values. The J flag will denote the sample results as below the QL and as qualitative, estimated concentrations. This increases the probability of false positive results at these low concentrations, especially for the sample matrix anticipated for this project. However, analyst judgment will be used to determine if an apparent detected value should be reported or appears to be a false positive due to the sample matrix (e.g., from baseline "noise").

If dilution to bring the reported concentration of a single compound of interest within the linear range of the calibration, results in non-detect values for all other analytes with detected concentrations in the initial sample analysis, the results of the original run and the dilution will be reported with appropriate notations in the narrative of the report. Matrix effects (i.e., highly contaminated samples requiring dilution for analysis, dilution to bring detected levels within the range of calibration, and matrix interference requiring elevation of detection limits) will be considered in assessing compliance with the requirements for sensitivity. Cleanup procedures will be used to minimize interferences and lower the QLs to those required. In addition, the sample aliquot will be increased from the standard mass to make up for the increased QLs when data is reported on a dry weight basis (these samples are expected to be at least 50% moisture). This increased aliquot size may also increase the matrix interferences, as they too will have increased in mass. The QLs required by this project are listed in the method-specific tables in Attachment B of this document.

10.0 CORRECTIVE ACTION FOR UNACCEPTABLE QUALITY CONTROL DATA

10.1 Field Activities

All technical staff will be responsible for reporting all suspected technical nonconformances by initiating a nonconformance report of any issued deliverable or document. All staff will be responsible for reporting all suspected QA nonconformance by initiating a nonconformance report.

The Project Leader will be responsible for ensuring that corrective actions for nonconformance are implemented by:

- Evaluating all reported nonconformance;
- Controlling additional work on nonconforming items;
- Determining disposition or action to be taken;
- Maintaining a log of nonconformance;
- Reviewing nonconformance reports;
- Evaluating disposition or action taken; and
- Ensuring nonconformance reports are included in the final site documentation in document control.

Any staff member who discovers or suspects a nonconformance, which is an identified or suspected deficiency in an approved document, is responsible for initiating a nonconformance report. The Project Leader will ensure that no additional work, which is dependent on the nonconforming activity, is performed until the nonconformance report is corrected. The Project Leader will also be responsible for carrying out corrective action as initiated by the program QA manager. Each nonconformance report will be evaluated and the disposition and action taken will be recorded.

10.2 Laboratory

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions", to resolve problems and restore proper functioning to the analytical system (see section 5.0).

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy;
- Blanks, duplicate control samples or single control samples contain contaminants above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the project records.

10.3 Non-routine Occurrence Reports

The laboratory will send written reports of all significant non-routine occurrence events to the project chemist within 48 hours of occurrence of non-routine events for laboratory work. These reports will identify:

- the problem,
- corrective actions taken,
- verbal / written instructions from the USACE project chemist regarding reextraction and reanalysis of project samples and/or other applicable corrective actions to be taken.

Significant events are occurrences impacting cost of work, schedule of work, and quality of environmental analytical data.

11.0 REFERENCES

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