Quality Assurance Project Plan

Nemo Work Center EDB Site
Remedial Investigation Feasibility Study (RI/FS)
Black Hills National Forest, South Dakota

Prepared for

USDA Forest Service

Black Hills National Forest

Contract No. GS-10F-0117J/AG-82X9-D-08-0179

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QUALITY ASSURANCE PROJECT PLAN (QAPP)

QAPP Version 709R0

FOR THE USFS NEMO WORK CENTER RI/FS

Nemo, South Dakota

Prepared for:

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LIST OF ACRONYMS AND ABBREVIATIONS

AA	atomic absorption
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
DEQPPM	Defense Environmental Quality Program Policy Memorandum
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
FS	feasibility study
FSP	field sampling plan
g	gram
G	glass
GC	gas chromatography
GC/MS	gas chromatography/mass spectroscopy
GCD	*Guidance for Contract Deliverables, Version 1.1, March 1998*
GFAA	graphite furnace atomic absorption
GRO	gasoline range organics
HCl	hydrochloric acid
HNO_3	nitric acid
H_2SO_4	sulfuric acid
IAW	in accordance with
ICPAES	inductively coupled plasma atomic 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
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
ORP	oxidation-reduction potential
OVA	organic vapor analyzer
PE	performance evaluation
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
REC	recovery
RCA	recommendations for corrective action
RCRA	Resource Conservation and Recovery Act
RF	response factor
RI	remedial investigation
RI/FS	remedial investigation/feasibility study
RPD	relative percent difference
RSD	relative standard deviation
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	semi volatile organic compound
TCLP	toxicity characteristic leaching procedure
TIC	tentatively identified compound
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

SECTION 1
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) which is provided under separate cover, shall constitute, by definition, the USFS Nemo Work Center 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 during the remedial investigation/feasibility study (RI/FS), 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); and Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (U.S. EPA SW-846, Third Edition and its first, second and third update).

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 Forest Service managers, regulatory agencies, remedial project managers, project managers, and QA coordinators. Whenever Forest Service 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.

SECTION 2
PROJECT DESCRIPTION

2.1 THE USFS NEMO WORK CENTER RI/FS

The objective of the USFS Nemo Work Center RI/FS is to assess past hazardous waste disposal and spill sites at the Nemo Work Center located in the Black Hills National Forest, South Dakota, and to develop remedial actions consistent with the applicable federal, state, and local regulations 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 Agriculture (DoA), 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.

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.

2.2 PURPOSE AND SCOPE

The purpose of this project is to conduct a Remedial Investigation/Feasibility Study (RI/FS) for the Nemo Work Center at Nemo, South Dakota to delineate the extent of existing contamination, to determine the presence or absence of contamination throughout the site, and define the nature and extent of such contamination, if present. The RI/FS will be conducted on public and privately-owned property located adjacent to portions of existing, abandoned, and removed work center facilities:

- Adjacent to the USFS Nemo work center;
- Adjacent privately owned lands northwest, north and east of the USFS Nemo work center; and
- On USFS lands south of the Nemo Work Center.

2.3 PROJECT BACKGROUND

A project background description, including: (1) the locations of sites at the work center facility; (2) a summary of the contamination history at each site; and (3) the findings from previous investigations are included in Section 2.1 through Section 2.3 of the FSP.

2.4 PROJECT SCOPE AND OBJECTIVES

A summary of the objectives and the proposed work for each site is 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 also described in Section 3.1 of the FSP.

SECTION 3
PROJECT ORGANIZATION AND RESPONSIBILITY

As the primary contractor for this RI/FS, TEC will be responsible for overall RI project management and ensuring that all RI tasks described in project scoping documents are completed in a satisfactory manner. Key project team members and points of contact (POCs) for these affiliations are listed in Table 1-2 of the site-specific WP (AWP). Responsibilities for key TEC project team members and USFS Black Hills National Forest POCs are described as follows.

Prime Contractor Project Manager. As the project manager for the prime contractor on this contract, Mr. John Birdenbaugh of Weston Solutions, Inc. will provide the following services and oversight of TEC's activities on this project:

- technical monitoring of TEC performance and act as the sole technical POC between TEC and the USFS personnel;
- technical reviews of TEC proposals for any changes to the contract;
- coordinate activities with TEC personnel and individuals at the USFS Nemo Work Center, South Dakota;
- procurement of subcontractors;
- review invoices/payment vouchers according to Special Contract Requirements provisions of the basic contract;
- review, comment, and accept the completed effort specified in the SOW for this RI/FS;
- attend meetings as necessary with Key TEC project managers; and
- maintain written records for POC review of all actions taken by technical personnel, TEC, and himself to ensure that costs, schedule, and technical performance are accurately documented.

Project Director. The Project Director is Mr. Joe Lockerd. Mr. Lockerd is located at the TEC Golden, Colorado Office. As the Project Director, Mr. Lockerds' primary responsibility will be to provide guidance to the RI/FS Project Manager (PM). He also will:

- oversee project QA;
- serve as acting Project Manager in the Project Manager's absence;
- contact the client on a monthly basis to check on project status and hear the client's perception of the work activity first hand;
- attend key meetings with the Project Manager and the client as needed;
- attend key meetings with regulatory officials or other groups involved in the project as needed; and
- ensure that appropriate corporate resources will be applied to the project.

Project Manager. Mr. Jeff Hart is the RI/FS PM. He is located at the TEC office in Golden, Colorado. As PM, Mr. Hart will have the primary responsibility for all project management matters affecting the TEC portion of the RI/FS. The PM will be responsible for all aspects of the day-to-day operation of the RI/FS. These responsibilities will include:

- contract administration;
- personnel scheduling;
- budget tracking and control;
- client relations;
- technical direction;
- overall project QC; and
- production scheduling.

To complete these efforts, the project manager will work directly with project task managers to:

- develop and implement work task responsibilities and strategies;
- select project team members;
- assign responsibilities to project team members;
- schedule and coordinate staff meetings;
- prepare and submit Monthly Status reports to the client; and
- attend progress meetings with USFS Black Hills National Forest officials.

The PM will be responsible for ensuring that tasks are completed on schedule and within budget; reviewing work progress at various stages of its completion; maintaining regular communication with the client; and responding promptly to client concerns.

QA Manager. The TEC QA Manager is Mr. James Yocum. Mr. Yocum is located in the TEC Golden, Colorado Office. As QA Manager, Mr. Yocum will ensure that all work is performed according to the procedures described in the SAP. He will review evaluation reports and corrective action procedures to ensure that the project meets project planning documents and federal government standards.

3.1 SUBCONTRACTORS

A significant portion of the RI/FS will be conducted by subcontractors. To ensure that all subcontractor activities are planned, undertaken, and completed according to the guidelines and standards described in the contract documents, and project scoping documents, the PM will maintain regular contact with project subcontractors. The PM or FTL will be responsible for managing the subcontractor performance.

The subcontractors required for completing the RI will include:

- drillers;
- geophysical surveyor;
- off-site analytical laboratory and;
- data validator

Responsibilities for each of the project team subcontractors are described below. Prior to initiating field activities, TEC will prepare technical scopes of work (SOWs) for project subcontractors so that RI/FS activities can be effectively coordinated.

3.1.1 Drilling Services

During the RI, Antler Enterprises of Rapid City, South Dakota has been selected to install monitoring points for the monitoring well installation task of the project using air percussion hammer technology.

Additionally American Engineering, Inc., of Rapid City, South Dakota has been selected to conduct sub-surface sampling using direct push technology.

Soil and bedrock borings will be properly abandoned according to USFS and State of South Dakota regulations. The drilling subcontractors will observe all health and safety protocols and equipment decontamination procedures that are described in the HSP and this FSP.

3.1.2 Surveying Services

Land surveying services have not been included in the USFS scope of work. TEC recommends that all new wells be surveyed to an accuracy of ± 0.1 foot in the horizontal plane and ± 0.1 foot in the vertical dimension by a survey professional licensed in the State of South Dakota.

Hydrogeophysics (HGI) of Tuscon, AZ has been selected to perform non-intrusive magnetic and electromagnetic mapping of subsurface anomalies and geology.

Weston will be present when geophysical surveys are being conducted in support of the Nemo RI fieldwork. A member of the Weston/TEC team or Forest Service personnel be present when surveying professionals are taking measurements off of the top of casing on monitoring points in order to ensure that no contaminants are inadvertently introduced into the monitoring system.

3.1.3 Off-Site Analytical Laboratory Services and data Validation

Mid-Continent Testing Laboratories (MCTL) has been selected to perform the primary analytical laboratory services. The MCTL laboratory located in Rapid City, South Dakota will perform laboratory analyses and QA/QC according to program protocols as outlined in this QAPP. MCTL has been audited by the State of South Dakota. TEC may include other off-site analytical laboratories to perform work outside the capabilities of MCTL during the project duration. Should the need of additional laboratory capability be determined, this document will be amended to reflect the addition of additional laboratories doing work under this project. Laboratory Data Consultants (LDC) has been selected to perform third party data validation of analytical data.

3.1.4 Borehole Geophysics

No borehole geophysical surveys are planned for this RI/FS.

3.1.5 Site Restoration

TEC will notify Weston if it will be necessary to retain a qualified subcontractor to restore the fieldwork sites after completion of the RI fieldwork. Conditions that may require site restoration include ground disturbance caused by heavy equipment usage or drilling activity. Site restoration, if required, will be coordinated with the USFS Black Hills National Forest POC to ensure that restoration is conducted according to facility requirements.

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SECTION 4
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 described below and are also summarized in Section 3.1 of the FSP which is provided under separate cover

4.1 DATA CATEGORIES

The two general categories of data used in RI/FS investigations 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 6.0).

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

Definitive data are generated using rigorous analytical methods (see Section 7.0), 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 7.0 and 8.0). 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 6.0 and 7.0.

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. USEPA 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. The required level of precision differs according to the method, and is listed in the accuracy and precision tables in Section 7.0.

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 semi-volatile 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 analytical batch, and the associated sample results are interpreted by considering these specific measurements. The formula for calculation of accuracy is included in Table 4.2.1-1 as percent recovery (%R) from pure and sample matrices. Accuracy requirements are listed for each method in Section 7.0.

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 (e.g. by site). 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 8.0 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 re-sampling and analysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of possible 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	Used to assess total and analytical precision of duplicate measurements

Statistic	Symbol	Formula	Definition	Uses
			observations	
Percent Recovery	%R	$\left(\frac{X_{meas}}{X_{true}}\right)$ x 100	Recovery of spiked compound in clean matrix	Used to assess accuracy
Percent Recovery	%R	x 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 TEC at the beginning of the project (i.e., before project samples are analyzed) and upon request in the format specified in Section 8.0. Results less than or equal to the MDL shall be reported as the MDL value and flagged with a "U" (see Section 8.0).

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).

(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 or Teflon chips of 1 mm diameter or smaller for metals) containing the analyte of interest at a concentration three to five times the estimated MDL.

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

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

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

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

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

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

(5) Determine the MDL for each analyte as follows:

$$MDL = 3.14(s)$$

note: 3.14 is the one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using 7 samples. MDLs calculated from study sets with greater than seven replicates must use the appropriate one sided t-statistic associated with the number of samples included in the MDL study.

(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.

(7) When designing the study, the laboratory may not arbitrarily drop analytical points in the MDL data set. This is done in order to skew the MDL result in a desired pre-determined result and constitutes fraud. All data points obtained in an MDL study must be utilized in the calculation of the method MDL.

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 7.0. The MDL may not be more than one-half the corresponding RL. The laboratories shall also verify RLs by including a standard at or below the RL as the lowest point on the calibration curve. All results shall be reported at or above the MDL values, however, for those results falling between the MDL and the RL, an "F" flag shall be applied to the results indicating the variability associated with the result (see Section 8.0). 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 7.0. All results reported shall be within the calibration range. Results outside the calibration range are unsuitable for quantitative work and will only give an estimate of the true concentration. For guidance on dilutions, see pages 8-2 and 8-10. For SW6010 and SW6020, results shall be within the working range determined by linear range studies. Records of standard preparation and instrument calibration shall be maintained. Records shall unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards shall be traceable to standard materials.

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

4.4 ELEMENTS OF QUALITY CONTROL

QC elements relevant to screening data are presented in Section 6.0. This section presents QC requirements relevant to analysis of environmental samples that shall be followed during 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 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 analytical batch also extends to cover samples that do not need separate extraction or digestion (e.g., volatile analyses by purge and trap). This 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 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 analytical batch.

The type of QC samples and the frequency of use of these samples are discussed below and in the method-specific subsections of Section 7.0. Variances are described in Table 13-1.

4.4.1 Laboratory Control Sample

The laboratory control sample (LCS) is ASTM type II laboratory reagent grade water for aqueous analyses or a choice of Ottawa sand, sodium sulfate, Teflon chips, or glass beads 1 mm or smaller in diameter for soil samples, spiked with all analytes listed in the QC acceptance criteria table in Section 7.0 for the method. The LCS spike must be manufactured from a standard source other than the standards used to calibrate the instrument. These “secondary source” standards may take the form of a standard made by a different manufacturer than the manufacture of the calibration standards or a standard made by the manufacture of the calibration standards of a different lot number. Each analyte in the LCS shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. (The midpoint is defined as the median point in the curve, not the middle of the range). The LCS shall be carried through the complete sample preparation and analysis procedure.

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

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

4.4.2 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 7.0 for the method. The standard used for the MS and MSD shall be from the standard stock used to calibrate the instrument. 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. Samples associated with the USFS Nemo project are required to be used as the MS and MSD. The sample(s) used for the MS/MSD shall be designated on the chain of custody.

The MS/MSD is used to document the bias of a method due to sample matrix. Thus, for soil samples, laboratories may use the same container for the parent sample, the MS sample, and the MSD sample (except for VOAs), if there is enough sample. The prime contractor should select the samples for MS/MSDs. The sample replicates will be generated in the field, to be used by the laboratory to prepare the appropriate MS/MSDs. They are used to document potential matrix effects associated with a site. The MS/MSD results and flags must be associated or related to samples that are collected from the same site from which the MS/MSD set were collected.

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

The performance of the MS and MSD is evaluated against the QC acceptance limits given in the tables in Section 7.0. If either the MS or the MSD is outside the QC acceptance limits, the analytes in all related samples shall be qualified according to the data flagging criteria in Sections 7.0 and 8.0.

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, re-prepare and re-analyze 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.

IS's 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) and inductively coupled plasma – mass spectroscopy (ICPMS) analyses only, contains both interfering and analyte elements of known concentrations.

The ICS is used to verify background and inter-element 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 a sample prepared in the laboratory consisting of ASTM type II laboratory reagent grade water 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 analytical batch.

The presence of analytes in a method blank at concentrations equal to or greater than the RL indicates a need for corrective action. Corrective action shall be performed to eliminate the source of contamination prior to proceeding with analysis. After the source of contamination has been eliminated, all samples containing the analyte(s) found in the method blank above the RL shall be re-prepared 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 Field 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. Field blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes.

Field 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 field blanks is specified in Section 3.2 of the FSP. Field 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 8.0, shall be applied to all sample results from samples collected with the affected equipment.

4.4.10 Trip Blank

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

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. Each cooler of samples sent to the laboratory for analysis of VOCs shall contain one trip blank. For methanol preserved soil samples being analyzed for GRO or VOC, a methanol blank shall be utilized.

When an analyte is detected in the trip blank the appropriate validation flag, as described in Section 8.0, shall be applied to all sample results from samples in the cooler with the affected trip blank.

4.4.11 Field Duplicates

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected by dispensing sample from an aliquot at least 2x the required sample volume into two separate containers, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned a unique identification number in the field. Specific locations are designated for collection of field duplicate samples prior to the beginning of sample collection.

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

The frequency of collection for field duplicates is specified in Section 3.2 of the 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 a unique identification number in the field. Specific locations are designated for collection of field replicate samples prior to the beginning of sample collection.

Replicate sample results are used to assess precision. The frequency of collection for field replicates is specified in Section 3.2 of the 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 re-analyses. 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 re-analyses.

If holding times are exceeded and the analyses are performed, the results shall be flagged according to the procedures as described in Section 8.0.

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 7.0, and shall be completed within the method-required holding times. For GC methods, a second column is used for confirmation. For HPLC methods, a second column or a different detector will be used. The result from the primary column/detector is the result that shall be reported. If holding times are exceeded and the analyses are performed, the results shall be flagged according to the procedures as described in Section 8.0.

4.5.3 Control Charts

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

4.5.4 Standard Materials

Standard materials, including second source materials, used in calibration and to prepare samples shall be traceable to National Institute Standards and Technology (NIST), EPA, American Association of Laboratory Accreditation (A2LA) or other equivalent source, if available. Standards should be certified by the manufacture as being compliant with the ISO 17025 standards if possible. If an NIST, EPA or A2LA standard material is not available, the standard material proposed for use shall be included in an addendum to the SAP and approved before use. The standard materials shall be current, and the

following expiration policy shall be followed: The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory shall label standard and QC materials with expiration dates.

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

4.5.5 Supplies and Consumables

The laboratory shall inspect supplies and consumables prior to their use in analysis. The materials description in the methods of analysis shall be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents shall be monitored by analysis of the method blank. 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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SECTION 5
SAMPLING PROCEDURES

5.1 FIELD SAMPLING

The field sampling procedures for collecting samples and sampling methods shall be included in Section 6.0 of the FSP which is provided under separate cover.

5.1.1 Sample Containers

Sample containers are purchased pre-cleaned 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 USFS samples are listed in Table 5.1.2-1. The required sample volumes, container types, and preservation requirements for analytical methods proposed for project work not listed in Table 5.1.2-1 shall be included in an addendum to the FSP and approved by the USFS POC and/or the TEC project manager before use.

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

Name	Analytical Methods	Container[a]	Preservation[b,c]	Min. Container Size	Maximum Holding Time
Volatile Organic Compounds (VOCs)	SW8260B	G, Teflon-lined septum, T	4°C, HCl to pH < 2	3 x 40 mL	14 days
VOCs	E524.2/ E504.1	G, Teflon-lined septum, T	4°C, HCl to pH < 2	3 x 40 mL	14 days
Alkalinity	SM2320B	P, G	4°C	50 mL	14 days
Hydrogen ion (pH) (W, S)	SW9040B/ SW9045C	P, G	None required	N/A	Analyze immediately[d]
Conductance	SW9050A	P, G	None required	N/A	Analyze immediately[d]

Name	Analytical Methods	Container[a]	Preservation[b,c]	Min. Container Size	Maximum Holding Time
Temperature	SM2550B	P, G	None required	N/A	Analyze immediately[d]
Oxidation-Reduction Potential	ASTM 1498	P, G	None required	N/A	Analyze immediately[d]
Ferrous Iron	Hach Kit	P, G	None required	N/A	Analyze immediately[d]
Carbon Dioxide	Probe	P, G	None required	N/A	Analyze immediately[d]
Nitrate	SM4500NO3-F	P	4°C, H_2SO_4 to a pH < 2	500 mL	28 Days
Methane, Ethane, Ethene	ERSK-175	G	4°C, HCl to pH < 2	40 mL	14 days
Sulfate	E375.2	P	4°C	500 mL	28 Days
Metals	SW6010B SW6020	P	HNO_3 to pH < 2, 4°C	500 mL or 8 ounces	180 days (water and soil)
Total petroleum hydrocarbons (TPH)-volatile	SW8015 (modified)	G, Teflon-lined septum, T	4°C, HCl to pH < 2	3 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,	4°C, H_2SO_4 to pH < 2	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 $Na_2S_2O_3$ is only required when residual chlorine is present.
d. Measurement should be performed on site.

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 chain of custody (COC) form (as illustrated in Section 8.0):

- unique sample identification for each container;
- date and time of sample collection;
- source of sample (including name, location, and sample type);
- designation of MS/MSD;
- preservative used;
- analyses required;
- name of collector(s);
- pertinent field data (pH, temperature, etc.);
- serial numbers of custody seals and transportation cases (if used);
- custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories; and
- 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 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 the USFS POC and/or the TEC project manager. The decision regarding the potentially affected samples shall also be documented.

Once the samples reach the laboratory, they shall be checked against information on the COC form for anomalies. For the safety of the personnel involved, coolers containing samples shall be opened in a hood in case there has been any breakage of container of potentially contaminated sample material. The condition, temperature, and appropriate preservation of samples shall be checked and documented on the COC form or a sample receipt condition form produced by the laboratory. Checking an aliquot of the sample using pH paper is an acceptable procedure except for VOCs where preservation should be checked post analysis and noted on the report. 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 such as a LIMS, 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 USFS work are specified in Table 5.1.2-1. **Samples not analyzed in accordance with these requirements shall be re-sampled and analyzed, at no additional cost to the USFS.** Subcontracted analyses shall be documented with a COC form from the subbing laboratory to the subcontract laboratory. It is required that this information be included in the laboratories final analytical report package. 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 at least annually. As indicated by the findings of the calibration, correction factors shall be applied to each thermometer. Records that include acceptance criteria shall be maintained. Samples for volatile organics determination shall be stored separately from other samples, standards, and sample extracts. Samples shall be stored after analysis until disposed of IAW applicable local, state, and federal regulations. Disposal records shall be maintained by the laboratory. Refrigerators storing USFS VOA samples shall contain a blank that shall be analyzed at a minimum of every two weeks. Detection of VOCs in the storage blank requires that the laboratory undertake corrective actions and this occurrence shall be noted in the laboratory case narrative in the final analytical data package.

Standard operating procedures (SOPs) describing sample control and custody shall be maintained by the laboratory. These SOPs are required to be signed and acknowledged by all laboratory personnel involved in the receipt and processing of the samples.

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SECTION 6
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.

Table 6.1. Screening Analytical Methods

Method	Parameter
SW846 (3550)	Percent Dry Weight (as % solids)
SW9040B	pH (water)
SW9050A	Conductance
SM2550B	Temperature
E180.1	Turbidity
Hach Kit	Alkalinity, Chloride, Sulfate, Nitrate, Ferrous Iron
SM4500NO3-F	Nitrate+Nitrite
E300.0	Bromide, Chloride, Fluoride, Sulfate
ERSK-175	Methane
SM4500O-G	Dissolved Oxygen, Carbon Dioxide
Organic Vapor (FID and PID)	Soil gas screening-halogenated, aromatic, and petroleum hydrocarbons
ASTM D422	Particle size
ASTM D1498	Oxidation-reduction potential
ASTM D3152	Capillary Moisture
SW8015B	TPH Gasoline
SW8021B	BTEX

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: and

- the RL (if applicable)

6.1.1 SW-846 (Described in Method SW3550)–Percent Solids

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

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

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

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

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

6.1.2 EPA Method SW9040B (Water)

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.

6.1.3 EPA Method SW9050A–Conductance

Standard conductivity meters are used. Temperature is also reported.

6.1.4 EPA Method 300.0

A small volume of sample, typically 2 to 3 mL, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector. Retention times and peak areas are used to identify the analyte type and calculate the concentration of each analyte.

6.1.5 Standard Methods 4500NO_3-F

A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that was originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically.

6.1.6 HACH Kit–Alkalinity, Chloride, Sulfate, Nitrate, Ferrous Iron

A well-mixed sample is tested using a portable chemical testing kit.

6.1.7 Standard Methods 2550B –Temperature

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

6.1.8 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.

6.1.9 Standard Methods 4500O-G –Dissolved Oxygen and Carbon Dioxide

An instrumental probe, usually dependent upon an electrochemical reaction, is used for determination of dissolved oxygen and carbon dioxide in water. Under steady-state conditions, the current or potential can be correlated with dissolved oxygen and carbon dioxide concentrations.

6.1.10 ASTM D422–Standard Method for Particle-Size Analysis of Soils

This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process using a hydrometer.

6.1.11 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.

6.1.12 ASTM D3152-Capillary Moisture

This test method covers the determination of capillary-moisture properties of fine-textured soils as indicated by the moisture content - moisture tension relationships determined by pressure-membrane apparatus using tensions between 1 and 15 atm (101 and 1520 kPa). Moisture tension (matrix suction) is defined as the equivalent negative gage pressure, or suction, in soil moisture. The test result is a moisture content which is a measure of the water retained in the soil subjected to a given soil - water tension (or at an approximately equivalent height above the water table).

6.1.13 Real-Time Portable Organic Vapor Analyzers

Two types of portable analyzers shall be used to perform real-time nonspecific analyses of hydrocarbon vapors. The instruments include an FID (e.g., Foxboro Century OVA) and a photoionization detector (PID) (e.g., HNu® Systems [HNu®] trace gas analyzer) organic vapor monitor. Instruments of equivalent type mad by other manufacturers may be substituted. One or more of these instruments may be used at a specific site, depending on the contaminant species of interest. When used together, the instruments provide complementary information because they are sensitive to different types of hydrocarbon vapors.

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

instrument. Comparability is greatest when the instruments are calibrated with the same standards and operated within similar concentration ranges.

The FID uses the principle of hydrogen flame ionization to detect and measure total hydrocarbon vapors. The FID has a dynamic operating range from 1 ppmv to 10 ppmv or 1 ppmv to 100,000 ppmv, depending on the instrument, and provides a nonspecific response to total hydrocarbons. If concentrations exceed the range of the instrument, a dilution probe shall be attached to the FID to allow elevated vapor concentrations to be measured. The instrument is highly sensitive to compounds such as methane, benzene, and acetone, but is less sensitive to alcohols and halogenated compounds.

During operation, a sample is drawn into the probe and transmitted to the detection chamber by an internal pumping system. Inside the chamber, the sample is exposed to a hydrogen flame that ionizes the organic vapors. As the organic vapors burn, the ions produced are collected on an electrode in the chamber, and a current proportional to the hydrocarbon concentration is generated. This current is measured and displayed on the meter.

The PID uses a photoionization detector to detect and measure total hydrocarbon vapors. The instrument has an operating range of 0.01 - 2,000 ppm. During operation, a gas sample is drawn into the probe and past an ultraviolet light source by an internal pumping system. Contaminants in the sample are ionized, producing an instrument response if their ionization potential is equal to or less than the ionizing energy supplied by the lamp. The radiation produces a free electron for each molecule of ionized contaminant, which generates a current directly proportional to the number of ions produced. This current is measured and displayed on the meter. The PID measures the *total* value for all species present with ionization potentials less than or equal to that of the lamp.

6.1.14 EPA Methods SW8015 and SW8021B

These methods may be used for soil or aqueous screening for gasoline or diesel range organic compounds. Details of these methods are provided in Appendix A.

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 8.0). The other data qualifiers that shall be used with screening data are also shown in Table 6.2-1 and Section 8.0. 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%
SW 9050A	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
SW 9040B	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
SM2550B	Temperature	Field duplicate	10% of field samples	± 1.0ºC	Correct problem, repeat measurement	J
E 180.1	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

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%
SW 9050A	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
SW 9040B	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
SM4500NO3-F	Nitrate+Nitrite	Calibration with three standards plus a blank	Once per day at beginning of testing	Correlation coefficient ≥ 0.995	If calibration is not acceptable, re-analyze calibration standards. If re-analysis of calibration standards still results in unacceptable results, investigate instrument problems, mix fresh standards and analyze fresh calibration standards	R

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%
SW 9050A	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
SW 9040B	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
		Initial Calibration Verification (ICV)	Analyze immediately after calibration.	±10% of true ICV value	If the ICV sample fails the criteria, the ICV sample may be re-analyzed once. If the second ICV analysis fails, the laboratory must recalibrate the instrument.	R

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%
SW 9050A	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
SW 9040B	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
		Continuing Calibration Verification (CCV)	Analyze at a frequency of every ten samples. A CCV sample must be run and pass prior to the analysis of any samples and a closing CCV must be run at the end of the analysis sequence.	±10% of true CCV value	If the CCV sample fails the criteria, the CCV sample may be re-analyzed once. If the second CCV analysis fails, the laboratory must reanalyze the samples between failed CCV samples.	R

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%
SW 9050A	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
SW 9040B	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
		Laboratory Control Sample (LCS)	Analyze at a frequency of once per sample batch	±10% of true LCS value	If the LCS sample fails the criteria, the LCS sample may be reanalyzed once. If the second LCS analysis fails, the laboratory must reject all analysis data in the batch associated with the failing LCS. All samples in the batch must be re-prepared and analyzed on a separate run.	R
		Method Blank (MB)	Analyze at a frequency of once per sample batch	< Method MDL	Clean analytical system. Reanalyze samples.	B

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%
SW 9050A	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
SW 9040B	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
		Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Analyze at a frequency of one MS/MSD pair per batch	±10% of true MS/MSD value for recovery. The RPD between the MS/MSD sample shall be no greater than 10%	Reanalyze a failing sample spike. If the failure repeats, the result shall be qualified as matrix affected. If the RPD between the MS/MSD fails, the samples shall be qualified as estimated	M J

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%
SW 9050A	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
SW 9040B	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
E300.0	Bromide, Chloride, Fluoride, Sulfate	Calibration with three standard and a blank.	Once per day at beginning of testing	Correlation coefficient ≥ 0.995	If calibration is not acceptable, re-analyze calibration standards. If re-analysis of calibration standards still results in unacceptable results, investigate instrument problems, mix fresh standards and analyze fresh calibration standards	R

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%
SW 9050A	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
SW 9040B	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
		Initial Calibration Verification (ICV)	Analyze immediately after calibration.	±10% of true ICV value	If the ICV sample fails the criteria, the ICV sample may be re-analyzed once. If the second ICV analysis fails, the laboratory must recalibrate the instrument.	R

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%
SW 9050A	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
SW 9040B	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
		Continuing Calibration Verification (CCV)	Analyze at a frequency of every twenty samples. A CCV sample must be run and pass prior to the analysis of any samples and a closing CCV must be run at the end of the analysis sequence.	±10% of true CCV value	If the CCV sample fails the criteria, the CCV sample may be re-analyzed once. If the second CCV analysis fails, the laboratory must reanalyze the samples between failed CCV samples.	R

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%
SW 9050A	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
SW 9040B	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
		Laboratory Control Sample (LCS)	Analyze at a frequency of once per sample batch	±10% of true LCS value	If the LCS sample fails the criteria, the LCS sample may be reanalyzed once. If the second LCS analysis fails, the laboratory must reject all analysis data in the batch associated with the failing LCS. All samples in the batch must be re-prepared and analyzed on a separate run.	R
		Method Blank (MB)	Analyze at a frequency of once per sample batch	< Method MDL	Clean analytical system. Reanalyze samples.	B

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%
SW 9050A	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
SW 9040B	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
		Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Analyze at a frequency of one MS/MSD pair per batch	±10% of true MS/MSD value for recovery. The RPD between the MS/MSD sample shall be no greater than 10%	Reanalyze a failing sample spike. If the failure repeats, the result shall be qualified as matrix affected. If the RPD between the MS/MSD fails, the samples shall be qualified as estimated	M J
None	Organic vapor concentration (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

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%
SW 9050A	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
SW 9040B	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
Hach Kit	Alkalinity, Chloride, Sulfate, Nitrate, Ferrous Iron	Field duplicate	10% of field samples	RPD < 30%	Correct problem, repeat measurement	J
ASTM D1498	Oxidation reduction potential (ORP)	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

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Data Flagging Criteria[b]
E360.1	Carbon Dioxide	Field duplicate	10% of field samples	RPD < 30%	Correct problem, repeat measurement	J
SM4500O-G	Dissolved Oxygen	Field duplicate	10% of field samples	RPD < 30%	Correct problem, repeat measurement	J
ASTM D422-63	Particle Size	Field duplicate	10% of field samples	RPD < 30%	Correct problem, repeat measurement	J
ASTM D3152	Capillary Moisture	Field duplicate	10% of field samples	RPD < 30%	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".

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SECTION 7
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; and
- 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); *Guidance for Contract Deliverables (GCD), Version 1.1, March 1998*. Definitions of terms are given in Section 4.0, and data validation procedures are presented in Section 8.0.

7.1 PREPARATION METHODS

Extraction and digestion procedures for liquid and solid matrices presented in this section are outlined in Table 7.1-1.

Table 7.1-1. Extraction and Digestion Procedures

Method	Parameter
EPA 300	Common Anions in Soil
SW3005A	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy
SW3010A	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy
SW3020A	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy
SW3050B	Acid Digestion of Sediments, Sludges, and Soils
SW3510C	Separatory Funnel Liquid-Liquid Extraction
SW3520C	Continuous Liquid-Liquid Extraction
SW3535	Solid-Phase Extraction
SW3540C/SW3541	Soxhlet Extraction

Method	Parameter
SW3545	Pressurized Fluid Extraction
SW3550B	Ultrasonic Extraction
SW5021	Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis
SW5030B	Purge-and-Trap for Aqueous Samples
SW5031	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation
SW5032	Volatile Organic Compounds by Vacuum Distillation
SW5035	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

7.1.1 Method 300.0-Common Anions in Soil

Section 11.7 of USEPA Method 300.0 describes an extraction procedure for common anions in a solid matrix. A 10 to 1 water to solid mixture is mixed and filtered prior to analysis.

7.1.2 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.

7.1.3 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.

7.1.4 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 SW3050B–Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis

Method SW3050B is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by ICP or, for some metals, by GFAA. A sample is digested then refluxed with acid. A separate aliquot of the sample is dried for a total solids and/or percent moisture determination.

7.1.6 Method SW3510C-Separatory Funnel Liquid-Liquid Extraction

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

7.1.7 Method SW3520C-Continuous Liquid-Liquid Extraction

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

7.1.8 Method SW3535A-Solid-Phase Extraction

Method SW3535A is a procedure for isolating organic compounds from aqueous samples using solid-phase extraction media.

7.1.9 Method SW3540C/SW3541-Soxhlet Extraction

Method SW3540C is a procedure for extracting nonvolatile and semi volatile organic compounds from solids such as soils and sludges. Method SW3541 is an automated Soxhlet extraction. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

7.1.10 Method SW3545-Pressurized Fluid Extraction

Method SW3545 is a procedure for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, sediments, sludges, and waste solids using elevated temperature and pressure.

7.1.11 Method SW3550B-Ultrasonic Extraction

Method SW3550B is a procedure for extracting nonvolatile and SVOCs from solids such as soils and sludges. The sonication process ensures intimate contact of the sample matrix with the extraction solvent.

7.1.12 Method SW5021-Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis

Method SW5021 is a general purpose method for the preparation of VOCs in soils, sediments and solid wastes by GC or GC/MS analysis.

7.1.13 Method SW5030B-Purge and Trap for Volatile Organic Compounds

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

An inert gas is then bubbled through the sample solution at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept through a sorbent column where the volatile

components are trapped. After purging is completed, the sorbent column is heated and back flushed with inert gas to desorb the components onto a GC column.

7.1.14 Method SW5031-Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation

Method SW5031 is a method for separating nonpurgeable water-soluble and VOCs in aqueous or leachates from solid matrices using azeotropic distillation.

7.1.15 Method SW5032-Volatile Organic Compounds by Vacuum Distillation

Method SW5032 is a method used to determine volatile organic compounds from a variety of matrices using vacuum distillation.

7.1.16 Method SW5035-Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

Method SW5035 describes sample preparation and extraction for the analysis of VOCs in solid matrices. The method involves a heated purge of volatile components followed by analysis on a GC or GC/MS. Several sample preservation options are given in the method. Analyzing the sample unpreserved within the prescribed 48 hour holding time is the preferred option. If this is not possible, an appropriate preservation option must be chosen. For low-level VOC analysis, the preferred preservation is freezing with a 14 day holding time.

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 analytes included in these tables are not all inclusive lists. The specific lists of analytes for each method should be determined by regulatory requirements and site specific information. 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	Extraction and Digestion Methods
E8015B (modified)	TPH volatile and extractable (water and soil)	(volatiles) 5030B, 5031, 5035 (extractables) 3510C, 3520C, 3545C, 3541, 3545, 3550B
E8260B	Volatile organics (water and soil)	3585, 5021, 5030B, 5031, 5032, 5035
E6010B	Trace metals by ICPAES (water and soil)	3005A, 3010A, 3015, 3050B, 3051
E6020	Trace metals by ICP-MS (water and soil)	3005A, 3010A, 3015, 3050B, 3051
E 504.1	1,2-Dibromoethane (EDB), 1,2-Dibromo-3-chloropropane (DBCP), AND 1,2,3-Trichloropropane (123TCP) In Water By Microextraction And Gas Chromatography	(see analytical method)
ERSK-175	Soil gasses in water	N/A

7.2.1 Method SW8015B (Modified)-Volatile and Extractable Total Petroleum Hydrocarbons

Volatile petroleum hydrocarbon components, such as gasoline, jet fuel, and other low molecular weight petroleum products, are analyzed by the direct purge and trap technique described in method SW5030B followed by a modified approach to method SW8015. Extractable TPH components are analyzed by extraction followed by GC/FID analysis.

For volatile TPH, the sample is placed in the purge and trap sparge vessel and analysis is conducted using a GC equipped with a FID.

Extractable TPH components, such as kerosene, diesel, motor oil, and other high molecular weight extractable petroleum products, are typically prepared by method SW3520C or SW3510C for water-based matrices or by method SW3550B for soil/sludge matrices. Other extraction options are listed in table 7.2.1. After the sample is extracted, analysis is accomplished on a GC equipped with a capillary or megabore column and a FID. RLs for volatile TPH and extractable TPH are provided in Table 7.2.1-1.

Identification and quantitation of TPH components require more analytical judgment than other GC methods. The TPH chromatograms consist of groups of peaks that fall within a noted carbon retention time range (i.e., number of carbon atoms in the molecule). Standard fuel components are used to calibrate the instruments. The total petroleum hydrocarbons results are reported in mg/kg or mg/L based on quantitation of the total area count for the gasoline range organics (i.e., C6-C10) or the diesel range organics (i.e., C10-C28). The retention time window shall be set such that the window encompasses only the C6 through C28

range of organics. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7.2.1-2 and 7.2.1-3. Second column confirmation is not required.

Table 7.2.1-1. RLs for Method SW8015B (Modified)

		Water		Soil	
Parameter/Method	**Analyte**	**RL**	**Unit**	**RL**	**Unit**
Petroleum Hydrocarbons	Gasoline	0.1	mg/L	1.0	mg/kg
SW8015 (Mod)	Diesel	1.0	mg/L	10.0	mg/kg
	Jet Fuel	1.0	mg/L	10.0	mg/kg

Table 7.2.1-2. QC Acceptance Criteria for Method SW8015B (Modified)

Method	**Analyte**	**Accuracy Water (% R)**	**Precision Water (% RPD)**	**Accuracy Soil (% R)**	**Precision Soil (% RPD)**
SW8015 (Modified) GRO	TPH–Gasoline	67–136	≤ 30	57–146	≤ 50
	Surrogate:				
	Chlorobenzene	74–138		64–148	
SW8015 (Modified) DRO	TPH–Diesel	61–143	≤ 30	51–153	≤ 50
	TPH–Jet Fuel	61–143	≤ 30	51–153	≤ 50
	Surrogates (choose 2):				
	Octacosane	26–152		25–162	
	Ortho-Terphenyl	57–132		47–142	
	Fluorobenzene	75-125		65-135	

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
	Tricontane	40-140		30-150	

Table 7.2.1-3. Summary of Calibration and QC Procedures for Method SW8015B (Modified)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8015 (mod)	Volatile and Extractable Total Petroleum Hydrocarbons	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes ≤20% with no individual analyte RSD >30%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				linear – least squares regression r ≥ 0.995		
				non-linear – COD ≥ 0.990 (6 points shall be used for second order, 7 points for third order)		
		Continuing calibration verification	Daily, before sample analysis	All concentration levels of GRO within ±15% 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
			After every 10 samples and at the end of the	All concentration levels within ±15% of initial calibration	Correct problem then repeat initial cal. verification and reanalyze all	Apply R to all results for the specific analyte(s) in all

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
			analysis sequence		samples since last successful verification	samples since the last acceptable verification
SW8015 (mod)	Volatile and Extractable Total Petroleum Hydrocarbons	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Annually 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
		Method blank	One per analytical batch	No TPH detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.1-2	Correct problem then reanalyze a single time If still out, re-prep and reanalyze the LCS and all samples in the affected batch	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

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8015 (mod)	Volatile and Extractable Total Petroleum Hydrocarbons	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7.2.1-2	Correct problem then re-extract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 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
SW8015 (mod)	Volatile and Extractable Total	Retention time window calculated	Each initial calibration	GRO – calculate retention time based on 2-methylpentane	Correct problem then reanalyze all samples analyzed since the last	Apply R to the results from the sample

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
	Petroleum Hydrocarbons			and 1,2,4-trimethylbenzene (see 7.4.2 in method)	valid retention time check	
				DRO - calculate retention time based on C10 and C28 alkanes (see 7.4.3 in method)		
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 7.2.1-1	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 project work shall be documented, and all records shall be maintained by the laboratory.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

7.2.2 SW-846 Method SW8260B/ EPA Method 524.2 -Volatile Organics

Volatile (or purgeable) organics in water and soil samples are analyzed using SW-846 Method SW8260B or EPA Method 524.2. These methods use a capillary column GC/mass spectrometry technique. Volatile compounds are introduced into the GC by purge and trap (SW5030B or SW5035) or other approved method (see table 7.1.1). An inert gas is bubbled through the water samples (or a soil-water slurry for soil samples) to transfer the purgeable organic compounds from the liquid to vapor phase. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is back flushed 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 7.2.2-1. Soil samples with higher contaminant levels can be extracted using methanol before purging. However, the RLs arising from the use of this preparatory method will be higher than

those listed in Table 7.2.2-1 and the accuracy and precision requirements listed in Table 7.2.2-2 will not be met as well. Project specific DQOs and analytical protocols will need to be established if this preparatory method is used. Variances are provided in Table 13-1.

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 7.2.2-2 and 7.2.2-3.

Table 7.2.2-1. RLs for Method SW8260B or E524.2

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs	1,1,1,2-Tetrachloroethane	0.5	µg/L	0.003	mg/kg
SW8260B/E524.2	1,1,1-TCA	1.0	µg/L	0.005	mg/kg
	1,1,2,2-Tetrachloroethane	0.5	µg/L	0.003	mg/kg
	1,1,2-TCA	1.0	µg/L	0.005	mg/kg
	1,1-DCA	1.0	µg/L	0.005	mg/kg
	1,1-DCE	1.0	µg/L	0.006	mg/kg
	1,1-Dichloropropene	1.0	µg/L	0.005	mg/kg
	1,2,3-Trichlorobenzene	1.0	µg/L	0.005	mg/kg
	1,2,3-Trichloropropane	1.0	µg/L	0.005	mg/kg
	1,2,4-Trichlorobenzene	1.0	µg/L	0.005	mg/kg
	1,2,4-Trimethylbenzene	1.0	µg/L	0.006	mg/kg

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
	1,2-DCA	0.5	µg/L	0.003	mg/kg
	1,2-DCB	1.0	µg/L	0.005	mg/kg
	1,2-Dibromo-3-chloropropane	2.0	µg/L	0.01	mg/kg
	1,2-Dichloropropane	1.0	µg/L	0.005	mg/kg
	1,2-Dibromoethane (EDB)	0.01	µg/L	0.0005	mg/kg
	1,3,5-Trimethylbenzene	1.0	µg/L	0.005	mg/kg
	1,3-DCB	1.0	µg/L	0.006	mg/kg
	1,3-Dichloropropane	0.4	µg/L	0.002	mg/kg
	1,4-DCB	0.5	µg/L	0.002	mg/kg
	1-Chlorohexane	1.0	µg/L	0.005	mg/kg
	2,2-Dichloropropane	1.0	µg/L	0.005	mg/kg
	2-Chlorotoluene	1.0	µg/L	0.005	mg/kg
	4-Chlorotoluene	1.0	µg/L	0.005	mg/kg
	Acetone	10	µg/L	0.05	mg/kg
	Benzene	0.4	µg/L	0.002	mg/kg
	Bromobenzene	1.0	µg/L	0.005	mg/kg
	Bromochloromethane	1.0	µg/L	0.005	mg/kg
	Bromodichloromethane	0.5	µg/L	0.002	mg/kg
	Bromoform	1.0	µg/L	0.006	mg/kg
	Bromomethane	3.0	µg/L	0.01	mg/kg
	Carbon tetrachloride	1.0	µg/L	0.005	mg/kg
	Chlorobenzene	0.5	µg/L	0.002	mg/kg
	Chloroethane	1.0	µg/L	0.005	mg/kg
	Chloroform	0.3	µg/L	0.002	mg/kg
	Chloromethane	1.0	µg/L	0.005	mg/kg
	Cis-1,2-DCE	1.0	µg/L	0.005	mg/kg
	Cis-1,3-Dichloropropene	0.5	µg/L	0.003	mg/kg
	Dibromochloromethane	0.5	µg/L	0.003	mg/kg
	Dibromomethane	1.0	µg/L	0.005	mg/kg
	Dichlorodifluoromethane	1.0	µg/L	0.005	mg/kg

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
	Ethylbenzene	1.0	µg/L	0.005	mg/kg
	Hexachlorobutadiene	0.6	µg/L	0.003	mg/kg
	Isopropylbenzene	1.0	µg/L	0.005	mg/kg
	Methylene chloride	1.0	µg/L	0.005	mg/kg
	Methyl t-butyl ether (MTBE)	5.0	µg/L	0.02	mg/kg
	MEK (2-Butanone)	10	µg/L	0.02	mg/kg
	MIBK (methyl isobutyl ketone)	10	µg/L	0.02	mg/kg
	n-Butylbenzene	1.0	µg/L	0.005	mg/kg
	n-Propylbenzene	1.0	µg/L	0.005	mg/kg
	m,p-Xylene	2.0	µg/L	0.005	mg/kg
	Naphthalene	1.0	µg/L	0.005	mg/kg
	o-Xylene	1.0	µg/L	0.005	mg/kg
VOCs	p-Isopropyltoluene	1.0	µg/L	0.006	mg/kg
SW8260B/E524.2	Sec-Butylbenzene	1.0	µg/L	0.005	mg/kg
(concluded)	Styrene	1.0	µg/L	0.005	mg/kg
	TCE	1.0	µg/L	0.005	mg/kg
	Tert-Butylbenzene	1.0	µg/L	0.005	mg/kg
	Tetrachloroethene	1.0	µg/L	0.005	mg/kg
	Toluene	1.0	µg/L	0.005	mg/kg
	Trans-1,2-DCE	1.0	µg/L	0.005	mg/kg
	Trans-1,3-Dichloropropene	1.0	µg/L	0.005	mg/kg
	Trichlorofluoromethane	1.0	µg/L	0.005	mg/kg
	Vinyl chloride	1.0	µg/L	0.005	mg/kg

Table 7.2.2-2. QC Acceptance Criteria for Method SW8260B/E524.2

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS
SW8260B/	1,1,1,2-Tetrachloroethane	70-130	≤ 20	74-125	≤ 30	2

E524.2	1,1,1-TCA	70-130	≤ 20	68-130	≤ 30	1
	1,1,2,2-Tetrachloroethane	70-130	≤ 20	59-140	≤ 30	3
	1,1,2-TCA	70-130	≤ 20	62-127	≤ 30	1
	1,1-DCA	70-130	≤ 20	73-125	≤ 30	1
	1,1-DCE	70-130	≤ 20	65-136	≤ 30	1
	1,1-Dichloropropene	70-130	≤ 20	70-135	≤ 30	1
	1,2,3-Trichlorobenzene	70-130	≤ 20	62-133	≤ 30	3
	1,2,3-Trichloropropane	70-130	≤ 20	63-130	≤ 30	3
	1,2,4-Trichlorobenzene	70-130	≤ 20	65-131	≤ 30	3
	1,2,4-Trimethylbenzene	70-130	≤ 20	65-135	≤ 30	3
	1,2-DCA	70-130	≤ 20	72-137	≤ 30	1
	1,2-DCB	70-130	≤ 20	74-120	≤ 30	3
	1,2-Dibromo-3-chloropropane	70-130	≤ 20	49–135	≤ 30	3
	1,2-Dichloropropane	70-130	≤ 20	71-120	≤ 30	1
	1,2-EDB	70-130	≤ 20	70-124	≤ 30	2
	1,3,5-Trimethylbenzene	70-130	≤ 20	65-133	≤ 30	3
	1,3-DCB	70-130	≤ 20	72-124	≤ 30	3
	1,3-Dichloropropane	70-130	≤ 20	76-123	≤ 30	2
	1,4-DCB	70-130	≤ 20	72-125	≤ 30	3
	1-Chlorohexane	70-130	≤ 20	60–135	≤ 30	2
	2,2-Dichloropropane	70-130	≤ 20	67-134	≤ 30	1
	2-Chlorotoluene	70-130	≤ 20	69-128	≤ 30	3
	4-Chlorotoluene	70-130	≤ 20	73-126	≤ 30	3
SW8260B/	Acetone	70-130	≤ 20	40-141	≤ 30	1
E524.2	Benzene	70-130	≤ 20	73-126	≤ 30	1
	Bromobenzene	70-130	≤ 20	66-121	≤ 30	3
	Bromochloromethane	70-130	≤ 20	71-127	≤ 30	1
	Bromodichloromethane	70-130	≤ 20	72-128	≤ 30	1
	Bromoform	70-130	≤ 20	66-137	≤ 30	2
	Bromomethane	70-130	≤ 20	45-141	≤ 30	1
	Carbon Tetrachloride	70-130	≤ 20	67-133	≤ 30	1

	Chlorobenzene	70-130	≤ 20	75-123	≤ 30	2
	Chloroethane	70-130	≤ 20	41-141	≤ 30	1
	Chloroform	70-130	≤ 20	72-124	≤ 30	1
	Chloromethane	70-130	≤ 20	51-129	≤ 30	1
	Cis-1,2-DCE	70-130	≤ 20	67-125	≤ 30	1
	Cis-1,3-Dichloropropene	70-130	≤ 20	72-126	≤ 30	1
	Dibromochloromethane	70-130	≤ 20	66-130	≤ 30	2
	Dibromomethane	70-130	≤ 20	73-128	≤ 30	1
	Dichlorodifluoromethane	70-130	≤ 20	34-136	≤ 30	1
	Ethylbenzene	70-130	≤ 20	74-127	≤ 30	2
	Hexachlorobutadiene	70-130	≤ 20	53-142	≤ 30	3
	Isopropylbenzene	70-130	≤ 20	77-129	≤ 30	3
	m,p-Xylene	70-130	≤ 20	79-126	≤ 30	2
	Methylene chloride	70-130	≤ 20	63-137	≤ 30	1
	Methyl t-butyl ether (MtBE)	70-130	≤ 20	50-135	≤ 30	1
	MEK (2-Butanone)	70-130	≤ 20	40-135	≤ 30	1
	MIBK (methyl isobutyl ketone)	70-130	≤ 20	47-147	≤ 30	3
	n-Butylbenzene	70-130	≤ 20	65-138	≤ 30	3
SW8260B/	n-Propylbenzene	70-130	≤ 20	63-135	≤ 30	3
E524.2	Naphthalene	70-130	≤ 20	51-135	≤ 30	3
	o-Xylene	70-130	≤ 20	77-125	≤ 30	2
	p-Isopropyltoluene	73-130	≤ 20	75-133	≤ 30	3
	Sec-Butylbenzene	70-130	≤ 20	63-132	≤ 30	3
	Styrene	70-130	≤ 20	74-128	≤ 30	2
	TCE	70-130	≤ 20	77-124	≤ 30	1
	Tert-butylbenzene	70-130	≤ 20	65-132	≤ 30	3
	Tetrachloroethene	70-130	≤ 20	67-139	≤ 30	2
	Toluene	70-130	≤ 20	71-127	≤ 30	1
	Trans-1,2-DCE	70-130	≤ 20	66-134	≤ 30	1
	Trans-1,3-Dichloropropene	70-130	≤ 20	65-127	≤ 30	1
	Trichlorofluoromethane	70-130	≤ 20	49-139	≤ 30	1

	Vinyl Chloride	70-130	≤ 20	58-126	≤ 30	1
	Surrogates:					
	Dibromofluoromethane	85-115		65–135		
	Toluene-D8	81-120		84-116		
	4-Bromofluorobenzene	76-119		84-118		
	1,2-DCA-D4	72-119		52–149		
	Internal Standards:					
	Fluorobenzene					1
	Chlorobenzene-D5					2
	1,4-Dichlorobenzend-D					3

Table 7.2.2-3. Summary of Calibration and QC Procedures for Method SW8260B/E524.2

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8260 B/E524.2	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 – linear least squares regression r ≥ 0.995 for each analyte		
				option 3 non-linear – COD ≥ 0.990		

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
				(6 points shall be used for second order, 7 points 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
SW8260 B/E524.2	Volatile Organics	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
		Continuing Calibration verification	Daily, before sample analysis and after 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
		Demonstrate ability to generate acceptable accuracy	Once per analyst	QC acceptance criteria, Table 7.2.2-2	Recalculate results; locate and fix problem with system and then rerun	Apply R to all results for all samples analyzed by the analyst

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		and precision using four replicate analyzes of a QC check sample			demonstration for those analytes that did not meet criteria	
		Internal Standards (ISs)	Each sample	Retention time ±30 seconds from retention time of the IS in the ICAL mid-point std. EICP area within -50% to +100% of area from IS in ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; if system was malfunctioning, mandatory reanalysis of associated samples	Apply R to all results for analytes associated with a failed IS unless a matrix effect can be verified, then apply M.
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.2-2	Correct problem then reanalyze If still out, re-prep and reanalyze the LCS and all samples in the affected batch	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

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
						to all non-detects
SW8260 B/E524.2	Volatile Organics	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
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (section 7.2.9)	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 7.2.2-2	Correct problem then re-extract 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	Once per 12	Detection limits	none	Apply R to all

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		study	month period	established shall be ≤ ½ the RLs in Table 7.2.2-1		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 USFS Nemo Work Center 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, chloromethane and 1,1-dichloroethane

7.2.3 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 (ICPAES). The elements and corresponding RLs for this method are listed in Table 7.2.3-1. Variances to the RLs for this method are provided in Table 13-2. 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 SW6010B

		Water		Soil	
Parameter/Method	Analyte	RL	Unit	RL	Unit
ICP Screen for Metals	Aluminum	0.2	mg/L	20.0	mg/kg
SW6010B	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg

		Water		Soil	
Parameter/Method	**Analyte**	**RL**	**Unit**	**RL**	**Unit**
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.0	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.06	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

Table 7.2.3-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤ 20	80-120	≤ 30
	Antimony	80-120	≤ 20	80-120	≤ 30
	Arsenic	80-120	≤ 20	80-120	≤ 30
	Barium	80-120	≤ 20	80-120	≤ 30
	Beryllium	80-120	≤ 20	80-120	≤ 30
	Cadmium	80-120	≤ 20	80-120	≤ 30

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
	Calcium	80-120	≤ 20	80-120	≤ 30
	Chromium	80-120	≤ 20	80-120	≤ 30
	Cobalt	80-120	≤ 20	80-120	≤ 30
	Copper	80-120	≤ 20	80-120	≤ 30
	Iron	80-120	≤ 20	80-120	≤ 30
	Lead	80-120	≤ 20	80-120	≤ 30
	Magnesium	80-120	≤ 20	80-120	≤ 30
	Manganese	80-120	≤ 20	80-120	≤ 30
	Molybdenum	80-120	≤ 20	80-120	≤ 30
	Nickel	80-120	≤ 20	80-120	≤ 30
	Potassium	80-120	≤ 20	80-120	≤ 30
	Selenium	80-120	≤ 20	80-120	≤ 30
	Silver	80-120	≤ 20	75-120	≤ 30
	Sodium	80-120	≤ 20	80-120	≤ 30
	Thallium	80-120	≤ 20	80-120	≤ 30
	Vanadium	80-120	≤ 20	80-120	≤ 30
	Zinc	80-120	≤ 20	80-120	≤ 30

Table 7.2.3-3. Summary of Calibration and QC Procedures for Method SW6010B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6010 B	ICP Metals	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	If more than one standard is used, correlation coefficient must be ≥ 0.995	If applicable, correct problem and repeat initial calibration	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 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
		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
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Annually per analyst for each matrix tested.	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
SW6010 B	ICP Metals	Low level calibration check standard (at or below RL)	Once per analytical batch prior to sample analysis unless multi-point (3+) calibration with low std at or below RL is performed	All analyte(s) with ± 50% of expected value	Correct problem then reanalyze	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Linear range calibration (high) check standard	Every three months	Analyte within ± 10% of expected value	Correct problem then reanalyze or re-set linear range	Apply J to specific analyte(s) for all results not within linear range
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with contaminated blank	Apply B to all results for the specific analyte(s) above the RL 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 7.2.3-2	Correct problem then reanalyze If still out, re-prep and reanalyze the LCS and all samples in the affected batch	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
SW6010 B	ICP Metals	Dilution test	Each new sample matrix, at least once per analytical batch (only applicable for analytes with concentrations ≥50X MDL)	Fivefold (1+4) dilution must agree within ±10% of the original determination	Perform post digestion spike addition	Apply J to all sample results for specific analyte from the same matrix in the batch if either of following exist: (1) dilution test not run and batch had analyte concentrations ≥50X MDL (2) %D ≥ 10 and post digestion spike not performed
		Post digestion spike addition	When dilution test fails or if an analyte's concentration for all samples in a batch is less than 50X MDL	Recovery within 75-125% of expected results	Check for instrumental problem then reanalyze post digestion spike addition if appropriate	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 project samples per matrix	QC acceptance criteria, Table 7.2.3-2	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
SW6010 B	ICP Metals	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(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 USFS Nemo Work Center 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 SW6020-Trace Elements (Metals) by Inductively Coupled Plasma Mass Spectrometry for Water and Soil

Samples are analyzed for trace elements or metals using method SW6020 for water and soils. Analysis for total (i.e., acid leachable) metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The elements and Reporting Limits (RLs) for this method are listed in Table 7.2.4-1. The calibration, Quality Control (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 SW6020

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
ICP Screen for Metals	Aluminum	0.02	mg/L	2.0	mg/kg
SW6020	Antimony	0.001	mg/L	0.10	mg/kg
	Arsenic	0.02	mg/L	2.0	mg/kg
	Barium	0.003	mg/L	0.30	mg/kg
	Beryllium	0.003	mg/L	0.30	mg/kg
	Cadmium	0.002	mg/L	0.20	mg/kg
	Chromium	0.004	mg/L	0.40	mg/kg
	Cobalt	0.008	mg/L	0.80	mg/kg
	Copper	0.006	mg/L	0.60	mg/kg
	Lead	0.002	mg/L	0.20	mg/kg
	Manganese	0.002	mg/L	0.20	mg/kg
	Nickel	0.002	mg/L	0.20	mg/kg
	Silver	0.002	mg/L	0.20	mg/kg
	Thallium	0.0002	mg/L	0.02	mg/kg
	Zinc	0.025	mg/L	2.5	mg/kg

Table 7.2.4-2. QC Acceptance Criteria for Method SW6020

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6020	Aluminum	80–120	≤ 15	80–120	≤ 25
	Antimony	80–120	≤ 15	80–120	≤ 25

	Arsenic	80–120	≤ 15	80–120	≤ 25
	Barium	80–120	≤ 15	80–120	≤ 25
	Beryllium	80–120	≤ 15	80–120	≤ 25
	Cadmium	80–120	≤ 15	80–120	≤ 25
	Chromium	80–120	≤ 15	80–120	≤ 25
	Cobalt	80–120	≤ 15	80–120	≤ 25
	Copper	80–120	≤ 15	80–120	≤ 25
	Lead	80–120	≤ 15	80–120	≤ 25
	Manganese	80–120	≤ 15	80–120	≤ 25
	Nickel	80–120	≤ 15	80–120	≤ 25
	Silver	80–120	≤ 15	80–120	≤ 25
	Thallium	80–120	≤ 15	80–120	≤ 25
	Zinc	80–120	≤ 15	80–120	≤ 25

Table 7.2.4-3. Summary of Calibration and QC Procedures for Method SW6020

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6020	ICP/MS Metals	MS tuning sample	Prior to initial calibration and calibration verification	SW6020 paragraph 5.8	Retune instrument then reanalyze tuning solution	Apply R to all results for all analytes for all samples associated with the MS tuning
		Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	If more than one standard is used, correlation coefficient must be ≥ 0.995	If applicable, correct problem and repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration if calibration not done
		Calibration blank	Before beginning a sample run, after every 10 samples and at end of the analysis sequence	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) above the RL in all samples associated with the blank

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		Initial Calibration verification (Second source standard)	After initial calibration before beginning a sample run – at a concentration other than used for calibration	All analytes within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for the specific analyte(s) in all samples
		Continuing Calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within ±10% 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(s) in all samples since the last acceptable calibration
		Low level calibration check standard (at or below RL)	Once per analytical batch prior to sample analysis unless multi-point (3+) calibration with low std at or below RL is performed	All analyte(s) with ± 50% of expected value	Correct problem then reanalyze	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Linear range calibration (high) check standard	Every three months	Analyte within ± 10% of expected value	Correct problem then reanalyze or re-set linear range	Apply J to specific analyte(s) for all results not within linear range
SW6020	ICP/MS Metals	Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem re-prep and analyze method blank and all associated samples	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		Interference check solutions (ICS-A and ICS-AB)	At the beginning and end of an analytical run or once during an 12 hour period, whichever is more frequent	ICS-A All non-spiked analytes < RL unless they are a verified trace impurity from one of the spiked analytes ICS-AB Within ±20% of true value	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples	Apply R to all results for specific analyte(s) in all samples associated with the ICS

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 7.2.4-2	Correct problem then reanalyze If still out, re-prep and reanalyze the LCS and all samples in the affected batch	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 matrix in a analytical batch (only applicable for analytes with concentrations ≥100X MDL)	Fivefold (1+4) dilution must agree within ±10% of the original determination	Perform post digestion spike addition	Apply J to all sample results where analyte concentrations were ≥100X MDL and either dilution test not performed or %D > 10 but post digestion spike test was not performed
		Post digestion spike addition	When dilution test fails Or if an analyte's concentration for all samples in a batch is less than 100X MDL	Recovery within 75-125% of expected results	Dilute the sample; reanalyze post digestion spike addition	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the failed post digestion spike addition
SW6020	ICP/MS Metals	MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 7.2.4-2	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

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		Internal Standards (ISs)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in method SW6020, section 8.3	Apply R to all results for specific analyte(s) in all samples associated with the IS.
		IDL study	Every three months	Detection limits established shall be ≤ ½ the RLs in Table 7.2.4-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		MDL study	Every 12 months			
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Annually per analyst for each matrix tested	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 all results for all samples analyzed by the analyst
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

a. All corrective actions associated with USFS Nemo Work Center 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 EPA Method 504.1 – EDB and DBCP by GC/ECD Analysis

1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-chloropropane (DBCP) in drinking water samples are analyzed using method E504.1. This method uses capillary column gas chromatograph (GC)/electron capture detector (ECD) instrumentation. Ethylene dibromide (EDB) and 1,2-Dibromo-3-chloropropane (DCBP) are prepared by micro-extraction using hexane. The extract is injected into the GC. Positive results must be confirmed using a dissimilar analytical column.

Calibration—The GC is initially calibrated using a five-point calibration curve. 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 E504.1

Method	Analyte	Water	
		RL	Unit
E504.1	1,2-Dibromo-3-chloropropane	0.2	µg/L
	Ethylene dibromide	0.02	µg/L

Table 7.2.5-2 QC Acceptance Criteria for Method E504.1

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
E504.1	1,2-Dibromo-3-chloropropane	63-150	≤ 37
	Ethylene dibromide	65–141	≤ 20
	Surrogate:		
	1,1,12-Tetrachloroethane	67-137	

Table 7.2.5-3. Summary of Calibration and QC Procedures for Method E504.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
E504.1	EDB & DBCP	Five-point initial cal. for all analytes	Initial calibration prior to sample analysis	*option 1 linear*- mean RSD for all analytes ≤20% with no individual analyte RSD >30%	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 2 linear – least squares regression r > 0.995		Apply R to specific analyte(s) for all samples associated with the calibration
				option 3 non-linear – COD ≥ 0.990 (6 points shall be used for second order, 7 points for third order)		
		Second-source cal. verify	Once per five-point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to specific analyte(s) for all samples associated with the calibration
		Retention time window	Each sample	+ 3 times std. dev. For each analyte retention time from 72-hour study	Correct problem: reanalyze all samples since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Cal. verify	Daily, before sample analysis, every 10 samples and at the end of the run	All calibration analytes within ±20% of expected value	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
E504.1	EDB & DBCP	Demonstrate ability to generate acceptable	Annually per analyst	QC acceptance criteria, Table 7.2.5-2	Recalculate results; locate and fix problem with system and then	Apply R to all results for all samples analyzed by the analyst

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		accuracy and precision using four replicate analyses of a QC check sample			rerun demonstration for those analytes that did not meet criteria	
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with 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 re-prep and analyze the LCS and all samples in the affected analytical batch	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
		Surrogate spike	Every sample, spiked sample, and method blank	QC acceptance criteria, Table 7.2.5-2	none	For the samples; if; the %R > UCL for any surrogate, apply J to all positive results If the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects If any surrogate recovery is <10% then apply R to all results.
E504.1	EDB & DBCP	Second column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to all results for the specific analyte not confirmed. Apply J if RPD is>40% from first column result.
		MDL Study	One per every 12	Detection limits established shall be ≤ the RLs in	none	Apply R to all results for the specific analyte(s) in all samples in the

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
			months	the Table 7.2.5-1.		associated analytical batch
		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

a. All corrective actions associated with 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 RSK-175 –Soil Gases (Volatile Organics) in water

Soil gases in water are sampled and analyzed using method RSK-175. This method uses a high resolution GC coupled to one or more appropriate detectors (TEC recommends the use of a mass-selective detector). The analytes detected and RLs for this method are listed in Table 7.2.6-1.

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; and
- 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 7.2.6-2 and 7.2.6-3.

Table 7.2.6-1. RLs for Method RSK-175

Parameter/Method	Analyte	Water	
		RL	Unit
VOCs	Methane	5	µg/L
RSK-175	Ethane	5	µg/L
	Ethene	5	µg/L

Table 7.2.6-2. QC Acceptance Criteria for Method RSK-175

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
RSK-175	Methane	60-120	≤ 20
	Ethane	65-115	≤ 20
	Ethene	65-115	≤ 20

Table 7.2.6-3. Summary of Calibration and QC Procedures for Method RSK-175

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
RSK-175	Volatile Organics	Initial multipoint calibration minimum 3 standards	Initial calibration prior to sample analysis	%RSD for all calibration analytes ≤ 30% or linear regression correlation coefficient r≥ 0.995	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 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
		Calibration verification (one point)	Daily, before sample analysis and every 12 hours of analysis time	All calibration 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
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Annually per analyst	QC acceptance criteria, Table 7.2.6-2	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
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify	Apply R to all results for all samples associated with the tune

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
RSK-175	Volatile Organics	ISs	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 re-prep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.6-2	Correct problem then reanalyze If still out, re-prep and reanalyze the LCS and all samples in the affected analytical batch	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
		MDL study	Once per 12 month period			

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
RSK-175	Volatile Organics	Results reported between MDL and RL	Detection limits established shall be ≤ ½ the RLs in Table 7.2.6-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed	
			none	none	Apply F to all results between MDL and RL	

a. All corrective actions associated with 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.

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SECTION 8
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. Parts of this section are applicable to mainly the project management team and the individuals involved in the data validation of the analytical results. Some aspects, however are valid for the laboratory selected for the analysis of samples.

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. Definitions of these data qualifiers are shown in Table 8.2-1. 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.*

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 appropriate CLP type reporting forms 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 sample results shall be reported to one decimal place more than the corresponding RL, unless the appropriate number of significant figures for the measurement dictates otherwise. Soil samples shall have results reported on a dry weight basis. A wet weight aliquot of sample equivalent to the method specified dry weight aliquot of sample should be taken for analysis. Alternately, the lab may choose to use a consistent wet weight aliquot that is expected to be large enough to compensate for the moisture in the sample (e.g. 50% more) and use this as a consistent weight. RLs are NOT adjusted for sample moisture. If possible, samples

should be analyzed undiluted and non-detects reported to the project specified RLs. RLs for minority constituents in highly contaminated samples may be 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 precedence, are *R, M, J, F, 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 all completed data packages, and additionally 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, fiel

d 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. Contractual requirements for payment for laboratory services are beyond the scope of this document and may be different than the data validation requirements.

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 rejected 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 %R < LCL	J for the positive results J for the positive results, R for the non-detects	The specific analyte(s) in all samples in the associated AAB
Method Blank	Analyte(s) detected ≥ RL	B	The specific analyte(s) in all samples in the associated AAB with results above the RL
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 non-detects	The specific analyte(s) in all samples collected on the same sampling date
MS/MSD	MS or MSD % R > UCL OR MS or MSD % R < LCL OR MS/MSD RPD > CL	M for all results	The specific analyte(s) in all samples collected from the same site as the parent sample
Sample Preservation/ Collection	Preservation/collection requirements not met	R for all results	All analytes in the sample
Sample Storage	< 2°C or > 6°C or as required	J for the positive results R for the non-detects	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
	≥ high std / linear range	J

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

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

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

Table 8.2-3. 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	J for the positive	

QC Requirement	Criteria	Flag	Flag Applied To
	OR surrogate % R < LCL OR surrogate recovery < 10%	results J for the positive results R for the non-detects 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
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

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

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

QC Requirement	Criteria	Flag	Flag Applied To
			calibration verification
Calibration blank	Analyte detected ≥ RL	B	All results for specific analyte(s) above the RL 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 % R < 10%	J R	All sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
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 ELECTRONIC DATA DELIVERABLE REPORTS

The prime contractor shall provide an electronic deliverable report in the electronic data deliverable format as specified by the SOW for the project.

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), (3) method blank results, (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 items identified in this section.

Each hardcopy analytical report will contain the following items:

- A cover letter from the laboratory;
- An analytical summary report which will detail:
    - The Client responsible for submitting samples to the laboratory;
    - Laboratory assigned ID or Work Order;
    - Project ID;

  - Laboratory Sample ID;
  - Client Sample ID;
  - Sample collection date and time;
  - Receipt date and time;
  - Matrix;
  - Tests authorized to be run by the submitting client; and
  - Signature of authorized laboratory representative certifying the results.
- The laboratory analytical report containing the following information:
  - Client ID;
  - Project ID;
  - Lab ID;
  - Client sample ID;
  - Report date;
  - Collection date and time;
  - Date of submittal;
  - Analyte name;
  - Quantitated result;
  - Units;
  - Laboratory data qualifiers;
  - Reporting Limit;
  - Dilution factor used if any;
  - Method ID;
  - Analysis date and time; and
  - Identification of the analyst responsible for the generation of the data.
- A QA/QC report detailing performance of the QC samples run in conjunction with the associated client samples. The report shall have the following information:
  - Results of the Initial Calibration Standards used in the calibration procedure;
  - Results of the Initial Calibration Verification (ICV) sample run to verify proper calibration;
  - Results of the ICSA and ICSAB samples;
  - Results of the Continuing Calibration Verification (CCV) samples bracketing the client samples submitted for analysis;

- Results of the Continuing Calibration Blank (CCB) samples bracketing the client samples submitted for analysis;
- Results of any Laboratory Fortified Blank (LFB) associated with the preparation of the sample batch;
- Results of any Laboratory Reagent Blank (LRB) associated with the preparation of the sample batch;
    - LRBs should be reported down to the MDL of the instrument for the method used;
- Results of the Laboratory Control Sample (LCS) run after being subjected to all the preparatory steps as the field samples;
- Results of the Matrix Spike and Matrix Spike Duplicate (MS/MSD) samples.
    - The report shall report the reference value of the spiked sample in addition to the spike concentration added.
    - The report shall provide the recoveries observed for the MS MSD sample and provide the QC limits set for each analyte.
    - The report shall also provide the calculated RPD (Relative Percent Difference) between the MS and the MSD along with the QC RPD limit set for each analyte.
- Results of the method blank (MB or MBLK) analyzed after being subjected to all preparatory steps as the field samples;
    - MB data should be reported down to the MDL of the instrument for the method used;

- A detailed case narrative, which will detail all notations observed by the laboratory from the point of SDG receipt to report preparation. Any QC exceedences and subsequent corrective action reports shall be detailed or referenced in the case narrative.

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SECTION 9
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 *International Standards Organization 17025 Standard*. 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 8.0.

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 the USFS. The prime contractor shall review the audit findings and provide a written report to USFS. 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 TEC, Inc., 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 the USFS 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 may 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 7.0. 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, USFS, and other appropriate agencies of the situation at the earliest possible time and the prime contractor shall keep the USFS 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. The USFS or their representative 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 non-project-specific PE programs also demonstrate proficiency in methods used to analyze USFS 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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SECTION 10
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.

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SECTION 11
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, re-sampling 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.

11.3 MANUAL INTEGRATION

Manual integration is not to be a routine procedure for the purpose of meeting QA/QC acceptance criteria. It is to be done only rarely as a corrective action measure when chromatography software inappropriately integrates chromatographic peaks. An example would be when there are co-eluting compounds and those compounds cannot be separated by the instrument. Manual integration would be appropriate to identify the peaks. When manual integration is used the following procedures are to be implemented for documenting the event and to conduct training for consistency in performing the manual integration.

- The USFS requires the laboratory or section SOP to include instructions for the analyst to document any required manual integrations associated with the initial/continuing calibration. The SOP is to require the following hard copy documentation:

    - a “before” and “after” hard copy for the manual integration, with the reason, date and signature of the analyst; and

- review and approval for the manual integration by the Section supervisor and the QAO.

- The USFS requires that technical guidelines be developed for manual integration. Topics covered must include under what conditions manual integrations are to be initiated and what constitutes technically acceptable manual integration events, and improperly integrated examples (e.g. – peak shaving, baseline extension). This will ensure consistency when manual integrations are performed.

SECTION 12
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 USFS auditors upon request.

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SECTION 13
LABORATORY VARIANCES

The following table is a complete list of variances to the USFS Nemo Work Center QAPP, that are requested by the subcontracted analytical laboratories for this project (*To Be Filled In Later*).

Table 13-1. Laboratory Analytical Variances for USFS Nemo Work Center QAPP

Lab	QAPP Section	Comment	Supporting Information/Reason