CP2-ES-5107/FR1

Inorganic Analyses
Data Verification and Validation
Paducah Gaseous Diffusion Plant,
Paducah, Kentucky

FOUR RIVERS
NUCLEAR PARTNERSHIP, LLC

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Inorganic Analyses
Data Verification and Validation
Paducah Gaseous Diffusion Plant,
Paducah, Kentucky

Date Issued—December 2017

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Office of Environmental Management

Prepared by
FOUR RIVERS NUCLEAR PARTNERSHIP, LLC,
managing the
Deactivation and Remediation Project at the
Paducah Gaseous Diffusion Plant
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APPROVALS

Inorganic Analyses
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Paducah Gaseous Diffusion Plant,
Paducah, Kentucky

CP2-ES-5107/FR1

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ACRONYMS

AA	atomic absorption
CCB	continuing calibration blank
CCV	continuing calibration verification
CLP	contract laboratory program
COC	chain of custody
DQO	data quality objective
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
EB	equipment blank
ICB	initial calibration blank
ICP	inductively coupled plasma
ICP-MS	inductively coupled plasma/mass spectrometry
ICS	interference check sample
ICV	initial calibration verification
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MDL	method detection limit
MS/MSD	matrix spike/matrix spike duplicate
PDS	post-digestion spike
PB	preparation blank
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RI	relative intensity
RB	rinseate blank
RL	reporting limit
RPD	relative percent difference
SD	serial dilution
SDG	Sample Delivery Group
SMO	Sample Management Office
SOW	statement of work
TDS	total dissolved solids
TOC	total organic carbon
TSS	total suspended solids
%R	percent recovery

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REVISION LOG

REVISION NUMBER	DATE	DESCRIPTION OF CHANGES	PAGES AFFECTED
FR0	11/14/17	Bluesheet	ALL
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SMO	Sample Management Office
SOW	statement of work
TDS	total dissolved solids
TOC	total organic carbon
TSS	total suspended solids
%R	percent recovery

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DEFINITIONS

NOTE 1: Qualifier definitions are listed in Appendix A.

NOTE 2: In this plan, the words "shall" and "must" are used to denote a requirement; the word "should" is used to denote a recommendation; and the word "may" is used to denote permission (neither a requirement nor a recommendation). In conformance to this plan, all steps shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification must be documented for deviations from recommendations.

AFFECTED SAMPLE RESULT—A sample result is considered to be affected when it is significantly influenced by a quality deficiency and is qualified accordingly through analytical data validation.

ANALYTICAL DATA VALIDATION—Analytical data validation is a systematic process, performed independently from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

ANALYTICAL DATA VERIFICATION—Analytical data verification is a systematic process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract that is performed either by the data generator or by an entity independent to the data generator.

BATCH—A batch is a group of samples prepared at the same time in the same location using the same method, not to exceed 20 samples of similar matrix.

CASE—A finite, usually predetermined number of samples, that have been collected over a given time period from a particular site. A case consists of one or more sample delivery groups.

CHAIN OF CUSTODY (COC)—The history of the transfer of samples from the time of sample acquisition through archival and disposal of samples. COC documentation is required as evidence of sample integrity.

CONTINUING CALIBRATION VERIFICATION (CCV)—A standard solution analyzed at a specified frequency during an analytical run to assure continued validity of the calibration curve.

CORRECTABLE PROBLEM—Correctable problems are deficiencies within data packages that may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation. Correctable problems revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory. Correctable problems revealed during validation are those deficiencies with analyses that can be solved either by a second preparation and/or by analysis of a sample.

DATA QUALITY OBJECTIVES (DQO)—DQOs are qualitative and quantitative statements derived from the outputs of each step of the DQO Process that specify the study objectives, domain, limitations, the most appropriate type of data to collect, and specify the levels of decision error that will be acceptable for the decision.

DATA QUALITY OBJECTIVES PROCESS—The DQO Process is a quality management tool based on the scientific method and developed by EPA to facilitate the planning of environmental data collection activities. The DQO Process enables planners to focus their planning efforts by specifying the use of the

data (the decision), the decision criteria (action level), and the decision maker's acceptable decision error rates.

HOLDING TIME—Holding time, as described in this plan, is defined as the period of time between sample collection and sample activity determination.

LABORATORY CONTROL SAMPLE (LCS)—The LCS is a control sample of known composition. Aqueous and solid LCSs are analyzed using the same preparation, reagents, and method employed for field samples.

LABORATORY DUPLICATE—The laboratory duplicate is a randomly chosen split of an analytical sample into two aliquots prior to sample preparation. The purpose of a laboratory duplicate is to monitor the precision of the analytical method.

MATRIX SPIKE (MS)—The matrix spike is a split of a field-originating analytical sample in which one half of the split is spiked with a known amount of radionuclide of interest prior to sample preparation. The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation.

NONCORRECTABLE PROBLEM—Noncorrectable problems are deficiencies within data package that preclude the evaluation of data quality by predefined criteria. Noncorrectable problems may be revealed during both data verification and data validation.

PREPARATION BATCH—A preparation batch is a group of sample aliquots prepared together at the same time using the same method and related to the same quality control samples.

QUALITY-INDICATOR SAMPLE—Quality-indicator samples are those samples made ready in the laboratory that provide direct or indirect evaluation of the status of the analytical system and resulting data quality. Collectively, quality indicator samples are the laboratory control sample, laboratory duplicate, matrix spike, and method blank.

REPORTING LIMIT (RL)—The RL is a contractually specified detection limit that, under typical analytical circumstances, should be achievable.

SAMPLE DELIVERY GROUP (SDG)—An SDG is defined by one of the following, whichever occurs first: (1) Case of field samples; (2) Each 20 field samples within a case; (3) Each 14-day calendar period during which field samples in a case are received, beginning with receipt of the first sample in the SDG.

SAMPLE RESULT—A sample result, as described in this plan, is a numeric denotation of the concentration, amount, or activity of a specific analytical parameter uniquely associated with an aliquot of environmental media.

STATEMENT OF WORK (SOW)—The validation SOW is a document prepared to function as the mechanism by which validation requirements are communicated from the project to the validation organization.

TURN-AROUND TIME—Turn-around time is contractually specified as the amount of time that elapses between laboratory receipt of the raw samples and subsequent data receipt by the client.

VALIDATION QUALIFIER—A qualifier is an alphabetic character physically or electronically associated with a discrete sample result during validation due to a data quality deficiency, which provides guidance in data usability.

VALIDATION STATEMENT OF WORK—The validation SOW is a document prepared to function as the mechanism by which validation implementation requirements are communicated from the project to the validation organization.

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1. INTRODUCTION

1.1 PURPOSE, SCOPE, AND APPLICATION

1.1.1 Purpose

This plan defines the minimum requirements, responsibilities, and methodology for inorganic data verification and validation generated using instrumental methods.

This plan provides requirements for developing and implementing a validation methodology for inorganic Contract Laboratory Program (CLP) and SW-846 (6010, 6020, 7470, and 7471) analytical methods primarily for analytes in aqueous and soil/sediment matrices. It also covers the analysis of cyanide. It is flexible enough to allow evaluation of data usability for project-specific Data Quality Objectives (DQOs). Data produced by analytical methods for which this plan provides limited guidance [i.e., U.S. Environmental Protection Agency (EPA) 200 series methods] may necessitate development of modified criteria from this plan; however, the general validation strategy outlined in this plan should be applicable. In the absence of specific guidance contained herein, data validators are advised to seek guidance in the specific method employed and/or from other industry standards. Examples include EPA CLP, National Functional Guidelines for Inorganic Data Review, EPA Regional Data Validation Guidance, and subject matter experts within the industry.

Specifications in this plan should be incorporated into project documentation such as the Quality Assurance Project Plan (QAPP), into contractual SOWs between the project and the analytical laboratories and into contractual validation SOWs between the project and the organization chosen to validate the data. If data validation is performed by individuals within the project, the SOW is not required, but a mechanism to specify data validation requirements is recommended. This plan shall be used as a baseline to create project-specific reports needed to perform inorganic data verification and validation.

1.1.2 Scope and Application

This plan applies to inorganic data verification and validation activities performed by the Sample Management Office (SMO) or its subcontractors.

2. RESOURCES

- Analytical Method
- Laboratory Statement of Work (SOW)
- Data Validation SOW
- Project-Specific QAPP

3. PREPERFORMANCE ACTIVITIES

Project manager shall ensure that individuals who perform inorganic data verification and validation are knowledgeable of the latest version of this plan before beginning any activities.

4. GENERAL INFORMATION

4.1 REQUIRED ELEMENTS OF REVIEW AND VALIDATION

To the extent possible, all laboratory data packages will be produced by the laboratory performing the analysis as Level IV (i.e., U.S. EPA Stage 4) laboratory data deliverables. One-hundred percent of the data deliverables will undergo a data quality review and data validation comparable to a Level I validation (depending on analyte and method). As required by project-specific requirements, the data review and validation effort may be increased to cover a Level II, Level III, or a full Level IV validation of the data package. The activities included in the review and validation effort for each level are provided in Table 1.

Table 1. Required Elements of Review and Validation

Report Elements to be Reviewed[1]	**Level I**	**Level II**	**Level III**	**Level IV**
Cover/Signature Page	x	x	x	x
Table of Contents			x	x
Report Narrative	x	x	x	x
Executive Summary (if included)			x	x
Method Summary/Analyst Summary			x	x
Sample Summary/Sample Data Sheets	x	x	x	x
Shipping and Receiving Documents	x	x	x	x
Client Chain of Custody (COC)	x	x	x	x
Sample Receipt Checklist	x	x	x	x
Interlab COC (where applicable)		x	x	x
Internal COC (if required)			x	x
Glossary of Abbreviations	x	x	x	x
QC RESULTS				
Quality Control (QC) Association Summary		x	x	x
Laboratory Chronicle			x	x
Surrogate and/or Tracer and Carrier Recovery Report		x	x	x
Blank Reports		x	x	x
Laboratory Control Sample (LCS) Reports		x	x	x
Matrix Spike(MS)/Matric Spike Duplicate (MSD) and LCS Duplicate (LCSD) Reports		x	x	x
Hold Times and Preservation Requirements	x	x	x	x
[Extended Data Deliverables/Forms]				
CLP-Like Organics				
SUMMARY FORMS			x	x
Summary Forms (Org I-X)			x	x
QC SUMMARY			x	x
QC Forms (Org I-IV,VIII)			x	x
SAMPLE DATA			x	x
Quant Rpt + Chro + spectra				x

Table 1. Required Elements of Review and Validation (Continued)

Report Elements to be Reviewed[1]	Level I	Level II	Level III	Level IV
STANDARDS DATA			x	x
Calibration Forms (VI-VII; for GC- VIII-X)			x	x
(Quant + chro follows each form set)				x
QC DATA			x	x
Tune			x	x
Blank Form I			x	x
Blank Quant Rpt + Chro + spectra				x
LCS/LCSD Form I			x	x
LCS/LCSD Quant Rpt + Chro + spectra				x
MS/MSD Form I			x	x
MS/MSD Quant Rpt + Chro + spectra				x
GEL Permeation Data				x
Florisil Data				x
Logs—Instrument, Prep, Standard			x	x
CLP-Like Inorganics				
Cover Page			x	x
Sample Forms (I) (CLP-like)			x	x
Calibration + QC Forms (ex:II-XIV)			x	x
Instrument Data				x
Preparation Data				x
SHIPPING/RECEIVING DOCUMENTS				
Internal COC (if required)			x	x
Interlab COC (where applicable)			x	x
Client COC	x	x	x	x
Sample Receipt Checklist	x	x	x	x

[1] Report elements listed represent common elements. The laboratory may provide more or less information as required by the method being analyzed. For example, those wet chemistry methods with no true calibration information will not have calibration forms included in the data package.

The requirements of the Level I and II review and validation effort will be referred to as "Data Verification" and will be performed by a member of the SMO. The requirements of the Level III and IV review and validation effort will be referred to as "Data Validation" and typically is performed by an entity external to the project. This can be an internal staff member that is not associated with the project, or it may be an independent third party external to Paducah Site. The following sections summarize the requirements of each type of review and validation efforts.

4.2 DATA VERIFICATION REQUIREMENTS

Data verification is defined as a systematic process, performed either by the data generator (on-site or fixed-base laboratory) or by an entity external to the data generator, which results in evaluation of the completeness, correctness, consistency, and compliance of a data set against a standard or contract.

If data verification is performed by the data generator, a project-level surveillance must be established by which the performance of the verification process is evaluated.

Data verification, at the project level, is conducted by a SMO representative to expedite the review process. If data verification is conducted independently of the data validator, it includes two activities. The first activity entails inventory of the data package to ensure compliance with the contract and SOW in terms of the required deliverables. The second activity entails various checks of data quality to determine the need for qualification. This process is referred to commonly as the "contractual screen" and is the

beginning of the data validation process in that it encompasses the review of the Level I and some Level II validation elements identified in Table 1. The data verifier will qualify data based on the review and validation elements in accordance with Section 5 of this plan. If the data set is being reviewed and validated at the Level III or IV requirements, then the data verifier will provide a copy of the data verification checklist to the data validator to expedite the validation process, or the data validator will perform both the data verification and data validation processes.

Data verification should provide a mechanism for problem resolution with the laboratory; it should not be exclusively an "after-the-fact" identification of noncorrectable deficiencies.

A data verification checklist is completed by the data verifier and takes, as input, the steps in this plan that that are listed as "Data Verification." The data verifier shall complete Form CP3-ES-5003-F03, "Data Verification Checklist," in accordance with CP3-ES-5003, *Quality Assured Data,* for all Level II, III, and IV validations.

4.3 ANALYTICAL DATA VALIDATION REQUIREMENTS

Analytical data validation, including laboratory data review, is defined as a systematic process, performed externally from the data generator, which applies a defined set of performance-based criteria to a body of data to determine the quality of reported results. Data validation is not performed by the analytical laboratory. Data validation provides a level of assurance, based on a technical evaluation, that an analyte is present or absent and, if present, the level of uncertainty associated with the measurement. Analytical data validation for inorganic methods includes a technical review of the laboratory data package specified in the laboratory SOW. Data validation incorporates an evaluation of sample custody, sample handling and preparation, holding times, instrument calibration, instrument performance, batch QC samples (e.g., LCS), the identification and quantitation of target analytes, performance standards (e.g., surrogates, internal standards), and the effect QC performance and/or deficiencies have on the quality of analytical sample data.

A data validation report that includes the results of data validation activities must be completed by the data validator for Level III and Level IV data validation requests and takes, as input, the data verification checklist (or equivalent) and the steps in this plan that are listed as "Data Validation."

Data validation requires that personnel performing it have the appropriate level of training and experience to ensure data review and qualification is completed in a reasonable manner and in accordance with industry practices. Professional judgment may be required when performing data validation. Where professional judgment is used, resulting in either qualification of data or data left unqualified, the rationale for the selection of this path will be documented fully in the data validation report. Documentation will include the following components: citations from this plan, other industry standards, and/or the literature demonstrating the reasonableness of the evaluation.

The actions described in this plan must serve as the baseline for incorporation into project verification/validation activities. Project-specific procedures applying to analytical methods not covered in this document must be reviewed and approved prior to use.

Implementation of this plan is expedited through the agreement of work to be performed by an analytical laboratory in the form of a project-specific laboratory SOW. Deliverable requirements specified in the analytical SOW must be consistent with the requirements of this plan and with the Basic Ordering Agreement contract with the laboratory.

The validation SOW must be written consistent with the requirements and specifications of this plan. The validation SOW is prepared by a SMO representative and communicated to the validation organization (for Level III and Level IV validation requests only).

The validation SOW will include as attachments full copies of the analytical data package, as well as an electronic data deliverable (EDD) in the form of a Microsoft Excel file. Placement of the qualifier may be assigned by hand writing on the laboratory report form, initialed and dated, or electronically on provided EDDs in the Validation Code field. If data are not qualified during data validation, an equals sign ("=") shall be entered on the sample result or placed in the Validation Code field of the provided EDD.

Form CP3-ES-5003-F03, "Data Verification Checklist," (in accordance with CP3-ES-5003 *Quality Assured Data)* must be completed for every sample delivery group (SDG) that undergoes Level II, III, or IV data validation. In addition to the data verification checklist, a data validation report must be completed for every SDG that undergoes Level III or Level IV data validation.

5. PROCEDURE

NOTE 1: Refer to Appendix A for qualifier descriptions. Refer to Appendix B for Qualification guidance due to multiple quality deficiencies. Refer to Appendix C for a listing of relevant equations to use with this plan.

The following is a step-by-step approach to implement analytical data verification and validation activities. This approach is based on current industry accepted standards. Because changes to methodology and the referenced guidance documents are not within the verifier's or the validator's control, the data verifier and the data validator should always follow the most current methodology and associated guidance documents referenced throughout this text to perform the review and validation of associated data.

5.1 VALIDATION STRATEGY AND SOW DEVELOPMENT

The project team, with input as needed from a QA specialist and/or a representative of the SMO, shall develop a data validation strategy based on inputs identified through the data quality objective (DQO) process. The project-specific sampling and analysis plan will define the DQOs and the framework for performing data validation. A SMO representative shall prepare a validation SOW to communicate data verification and validation requirements to the organization performing the work (for Level III and Level IV validation only).

5.2 CUSTODY OF SAMPLES AND SAMPLE DOCUMENTATION

The COC form provides the basis for the traceability of project samples by documenting the sample from its origin through all steps of the sampling, sample handling, and analysis process. The COC serves as documentation of sample possession from collection through disposal to ensure that sample representativeness is maintained prior to analysis. By documenting personal accountability for samples, the COC is used to ensure that proper custody has been maintained from the time a sample is generated through its final disposition (cradle to grave). Any break in custody, as demonstrated by the series of signatures denoting sample holders, could jeopardize the legal and/or technical defensibility of associated sample data.

While data verification/validation cannot replicate the custody history of a sample (i.e., fully assure the sample truly has been in custody from the field to the final result), an evaluation of field notes, laboratory records, and the COCs provides the best available indicator of sample traceability. A sample is defined as being under proper custody if any one of the following conditions is met:

- The sample is within the possession of an authorized person (e.g., field personnel, laboratory personnel, etc.);
- The sample is within view of an authorized person;
- The sample was in an authorized person's possession and then was secured to prevent tampering; or
- The sample is placed in a designated secure area.

NOTE: Data verification of sample documentation includes result report header checks for accuracy from the COC. If sample identity is in question, every attempt should be made to verify the true identity of each sample. When custody problems cannot be resolved, they will affect the defensibility of the sample.

5.2.1 Data Verification

The data verifier shall trace custody of all samples in the reporting batch from field sampling through receipt at the laboratory by reviewing the COCs. If the information is missing, the data verifier will seek to obtain field documentation from the sampler or laboratory to determine if the omission affects sample integrity. If there is a break in the signature chain on the COC or other omissions in the custody record (e.g., date of sample collection, date of transfer to the laboratory), indicate the problem on the data verification checklist and provide this information to the data validator.

5.2.2 Data Validation

If sample data are not traceable through signature records on COCs or other sample record information demonstrating custody (e.g., laboratory logbooks and/or sample data forms) cannot establish custody history, then the data validator shall qualify associated results rejected "R."

Custody of Samples	Yes	No	N/A
1. Does the data verification checklist or associated attachments in the data report indicate that samples are traceable?			

5.3 HOLDING TIME, TEMPERATURE, AND SAMPLE PRESERVATION

Holding times have been established by EPA to define the maximum period of time during which a sample remains representative of its sampling location. Holding times begin when a sample is collected in the field and are measured by determining the elapsed time from collection through extraction (when applicable) and/or analysis. If the reported data is the result of a dilution, reinjection, or re-extraction and analysis, the result must have been generated within the prescribed holding time in order for the result to be considered definitive.

5.3.1 Deliverables

- Field Sampling Notes
- Field COCs
- Laboratory COCs
- Laboratory Reports and/or raw data containing the following information: dates of collection, preparation, and analysis for all samples, dilutions, and re-extractions.

5.3.2 Criteria

Table 2 provides current industry-accepted standards for sample preservation and holding times for inorganic parameters. In all cases, the data verifier or validator shall always follow the most current methodology guidance for sample holding time, temperature, and preservation requirements.

Table 2. Inorganic Preservation and Holding Time Criteria

Matrix	Parameters	Preservatives	Holding Times
Aqueous	Atomic Absorption (AA) and Inductively Coupled Plasma (ICP) Metals	HNO_3 to pH < 2, 0–6°C	180 days
	Mercury	HNO_3 to pH < 2, 0–6°C	28 days
	Cyanide	NaOH to pH > 12, 0–6°C	14 days
Soil/Sediment	AA and ICP Metals	0–6°C	180 days
	Mercury	0–6°C	28 days
	Cyanide	0–6°C	14 days

5.3.3 Data Verification

The data verifier shall verify the presence of the pertinent COC forms in laboratory deliverables. If information is missing, the data verifier will seek to obtain field documentation from the sampler and/or the laboratory to determine if the omission affects sample integrity. Upon receipt, this information will be placed in the data package for delivery to the data validator. If missing information cannot be obtained or reconstructed from field notes, COCs, etc., the data verifier will note omitted information on the data verification checklist as noncorrectable.

5.3.4 Data Validation

5.3.4.1 Holding Times

Review the data verification checklist for holding times to confirm all holding times have been met. Holding times that are listed in hours from collection to analysis always will be calculated using the time collected to ensure the holding time in hours has not lapsed. Holding times that are listed in days will be calculated using dates only. The data validator shall review field and/or laboratory COC forms, field notes, laboratory report forms, and laboratory raw data, as necessary, to determine the elapsed time from sample collection to sample analysis for deviations identified on the data verification checklist.

If the elapsed time falls within the prescribed holding time, no actions will be taken and no qualification assigned.

If holding time is exceeded, qualify as follows:

- If the holding time has been exceeded by a factor < 2, qualify detected results "J" and nondetected results "UJ."
- If the holding time has been exceeded by a factor > 2, qualify detected results "J" and nondetected results "R."

5.3.4.2 Temperature/Preservation

Review laboratory receiving records to determine if samples were received at the appropriate temperature and that proper preservative addition (if required) has resulted in the appropriate pH adjustment(s). If records demonstrate samples were received at the proper temperature and with the appropriate pH adjustment, no action is warranted.

If the pH of aqueous samples is ≥ 2 for metals or < 12 for cyanide at the time of sample receipt, determine if the laboratory adjusted the pH of the sample to < 2 for metals or > 12 for cyanide at the time of sample receipt. If not, use the following guidance:

- If samples are received without the proper pH adjustment and not adjusted by the laboratory on receipt, qualify positive results "J" and nondetects "R" in the affected samples.
- If samples are received at elevated temperature (6°C < sample temperature < 10°C) but have received the proper pH adjustment, qualify detected analytes "J" and nondetects "UJ." If sample temperatures upon receipt are > 10°C, the data validator must evaluate the integrity of the reported concentrations, and the data may require qualification of "R."
- If samples are received at elevated temperature and proper preservation has not been followed (pH adjustment), qualify all affected sample results "R" rejected.

Holding Times and Sample Preservation				**Qualification Guidance**	
Validation Step	Yes	No	N/A	Detects	Nondetects
1. Does the data verification checklist indicate that all samples were analyzed within the appropriate holding time?				J	UJ/R
2. Were all samples preserved properly?*				R	UJ/R

*If samples are received without the proper pH adjustment and not corrected by the laboratory or if sample temperatures upon receipt are > 10° C, the data validator must evaluate the integrity of the reported concentrations, and the data may require qualification of "R."

5.4 CALIBRATION

Calibration is performed to ensure that the instrument used for analysis is capable of producing quantitative data. Initial calibration demonstrates the instrument is capable of acceptable performance at the beginning of the analytical run, and of producing a linear calibration curve (if applicable for the instrumentation used). Initial calibration verifications (ICV) and continuing calibration verifications (CCV) demonstrate the instrument remains in control throughout sample analysis.

5.4.1 Deliverables

- CLP Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN (or equivalent for SW-846 methods) for each initial calibration
- ICV/CCV Forms
- Analysis Results
- Standard Preparation Log
- Analytical Run Log
- Raw Data (required for confirmation)

5.4.2 Frequency

Initial calibration is method specific and must be performed daily (or every 24 hours), after CCV failure, or each time the instrument is set up.

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). If the ICV percent recovery (%R) falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

CCV samples shall be analyzed following each group of 10 samples or every two hours, whichever is more frequent, and following the last sample in the SDG. As required by a specific method, a low level CCV may also be analyzed during the analysis of samples.

5.4.3 Criteria

5.4.3.1 Initial calibration

- For ICP metals, at least one and up to five standards and a blank must be analyzed to develop the calibration curve.
- For ICP-MS metals, the mass spectrometer must be tuned properly, calibrated for, and checked for resolution in the mass regions of interest. Once proper performance has been demonstrated, at least one standard and a blank must be analyzed to develop the calibration curve.
- For mercury, four standards and a blank must be analyzed. The correlation coefficient must be ≥ 0.995.
- For cyanide, six standards and a blank must be analyzed. The correlation coefficient must be ≥ 0.995.

5.4.3.2 Calibration verification

Table 3 provides recovery criteria for calibration verification.

Table 3. Recovery Criteria for Calibration Verification

ICP and ICP-MS Metals	ICV: 90%–110% CCV: 90%–110%
Mercury	ICV: 85%–115% CCV: 85%–115%
Cyanide	ICV: 85%–115% CCV: 85%–115%

If a single calibration standard and blank are used to establish the initial calibration curve, then a low-level CCV should be included in the analytical sequence to verify the calibration curve is effective at the low end of the curve. Low-level CCVs are method specific and may not be included with all analytical results. When a low-level CCV is included, it should be within the laboratory's standard acceptable limits.

5.4.4 Data Verification

Data verifier shall verify that appropriate documentation of the initial calibration and the ICV/CCVs have been provided in the data package. If any one of the following occurs, the data verifier shall contact the laboratory immediately to obtain the missing information:

- Evidence of initial calibration is not included in the laboratory deliverable;
- Frequency of calibration has not been satisfied; and/or
- Required numbers of calibration standards or required standard concentrations were not used.

Upon receipt, this information will be placed in the data package for delivery to the data validator.

If these occurrences cannot be resolved with the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist. As they are contract compliance related, all such occurrences shall be communicated to the SMO and to the data validator on the data verification checklist.

5.4.5 Data Validation

If the initial calibration, the ICV, or the CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. Table 4 provides guidance for evaluating the calibration and the initial and continuing calibration verifications. See Appendix C for %R calculation.

When reviewing low-level CCVs, qualification for exceedances will be applied only to associated sample results that are within 20% of the low-level standard. Qualification of sample results based on the low-level CCV will follow the guidance for CCVs in Table 4.

Table 4. Calibration Actions for Data Validation

Method/Analyte	Calibration Result	Qualification Guidance
All	Calibration not performed	Qualify all results "R"
	Calibration incomplete	Use professional judgment Qualify results ≥ RL as "J" or "R" Qualify nondetects as "UJ" or "R"
	Correlation Coefficient < 0.995	Use professional judgment Qualify results ≥ RL as "J" or "R" Qualify nondetects as "UJ" or "R"
ICP/ICP-MS	ICV/CCV %R = 90-110%	No action
	ICV/CCV %R = 75-89%	Qualify results ≥ RL as "J"
	ICV/CCV %R < 75%	Qualify results ≥ RL as "J" or "R" Qualify nondetects as "R"
	ICV/CCV %R = 111-125%	Qualify results ≥ RL as "J" Results < RL = No Action
	ICV/CCV %R > 125%	Qualify results > RL as "J" or "R" Results < RL = No Action
Mercury	ICV/CCV %R = 85-115%	No action
	ICV/CCV %R = 70-84%	Qualify results ≥ RL as "J" Qualify nondetects as "UJ"
	ICV/CCV %R < 70%	Qualify results ≥ RL as "J" or "R" Qualify nondetects as "R"
	ICV/CCV %R = 116-130%	Qualify results ≥ RL as "J" Results < RL = No Action
	ICV/CCV %R > 130%	Qualify results ≥ RL as "J" or "R" Results < RL = No Action
Cyanide	ICV/CCV %R = 85-115%	No action
	ICV/CCV %R = 70-84%	Qualify results ≥ RL as "J" Qualify results < RL as "UJ"
	ICV/CCV %R < 70%	Qualify results > RL as "J" or "R" Qualify nondetects as "R"
	ICV/CCV %R = 116-130%	Qualify results ≥ RL as "J" Results < RL = No Action
	ICV/CCV %R > 130%	Qualify results ≥ RL as "J" or "R" Results < RL = No Action

5.5 BLANKS

Blank analyses serve to determine the existence and magnitude of contamination resulting from laboratory or field activities. Initial calibration blanks (ICB) and continuing calibration blanks (CCB) are used to ensure a stable instrument baseline before analysis of analytical samples. The preparation blank (PB) or method blank is used to assess the level of contamination introduced to the analytical samples throughout the sample preparation process. If contamination is found in <u>any</u> blank, all associated data must be evaluated carefully to determine whether a systematic problem affecting greater than one sample exists or whether the contamination is an isolated occurrence.

Additionally, the project team may elect to collect and analyze field and equipment rinseate blanks to evaluate the existence and magnitude of contamination that may arise as a result of field level activities. The field blank provides an indication of ambient conditions during the sampling activities, as well as providing an indication that the source of decontamination water is free of targeted analytes. The equipment rinseate blank provides an indication as to whether nondedicated sampling equipment has been

decontaminated properly and what, if any, carryover may arise between sampled locations. It has been EPA Region 4 data validation policy to evaluate the field blanks and equipment rinseate blanks as part of the validation process, but not to qualify the data based on these field samples.

5.5.1 Deliverables

- CLP Form III or equivalent for SW-846 methods
- Raw data for each blank (required for confirmation)

5.5.2 Frequency

Table 5 provides frequency of blank analyses.

Table 5. Blank Frequency

Parameter	Frequency
ICB	Immediately following the ICV
CCB	Immediately following each CCV
PB/MB	One for each sample batch and each sample matrix. The PB or MB will accompany no more than 20 samples for an individual matrix type.

5.5.3 Criteria

- No contaminants should be found in the blank.
- The absolute value of the analyte concentration in a blank analysis must be < method detection limit (MDL).
- All blanks in a SDG must be evaluated against sample results. All samples prepared together shall be evaluated against the associated PB.
- When evaluating blank results for solid matrices, the units of the PB will have solid reporting units (e.g., mg/kg).
- Dilution factors must be applied to blanks when evaluating sample results versus blank values.

NOTE: It is never permissible for the analytical laboratory or the data verifier/data validator to correct sample results by subtracting a blank value.

5.5.4 Data Verification

The data verifier shall verify the presence of the pertinent deliverables for blank analyses. If the required information is not present in the laboratory report, or if the frequency of analysis is not satisfied, the data verifier will contact the laboratory to obtain the omitted information. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If the information cannot be obtained, these occurrences are considered noncorrectable problems and will be identified as such on the data verification checklist. As this is a contract compliance issue, the occurrence should be communicated to the SMO and the data validator on the data verification checklist.

5.5.5 Data Validation

Review the laboratory deliverable to determine if any one of these occurs:

- Sample results have been corrected for blank values;
- Blank concentrations for any analyte > MDL;
- Any negative blank value for any analyte > MDL; or
- Each sample matrix being evaluated has an associated preparation blank.

Qualification is considered when the absolute value of any blank associated with project samples is > the MDL. Table 6 describes the actions to be taken in these cases. The data validator will use the highest absolute value for all associated blanks to determine qualification requirements for sample data.

Table 6. Blank Qualification

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	> MDL but < RL	Nondetect	No action
		> MDL but < RL	Qualify data "U"
		> RL	Use professional judgment
ICB/CCB	> RL	>MDL but < RL	Qualify data "U"
		> RL but < Blank Result	Report with "U" or qualify data as unusable "R"
		> Blank Result	Use professional judgment
ICB/CCB	< (-MDL) but > (-RL)	> MDL or nondetect	Use professional judgment
ICB/CCB	< (-RL)	< 10× RL	Qualify data "U"
PB/MB	> RL	> MDL but < RL	Qualify data "U"
		> RL but < 10× Blank Result	Qualify results as unusable "R" or estimated high "J"
		> 10× the Blank Result	No action
PB/MB	> MDL but < RL	Nondetect	No action
		> MDL but < RL	Qualify data "U"
		> RL	Use professional judgment
PB/MB	< (-RL)	< 10× RL	Qualify results > RL as estimated low "J" and nondetects as estimated "UJ"

Blanks				**Qualification Guidance**	
Validation Step	Yes	No	N/A	Detects	Nondetects
1. Were blanks (PB, ICB, CCB) prepared and/or analyzed at the appropriate frequency?				*	*
2. Were sample results verified as uncorrected for blank concentrations?				J	N/A
3. Were all blanks evaluated for contamination?				See plan text for guidance	
4. Were negative concentrations in blanks evaluated?				N/A	U
5. Was the presence of blank contaminants confirmed from raw data? (Applies to Level IV data only)				**	**

*Qualify only if the deviation indicates an adverse effect on data quality.
**Use professional judgment in qualifying data.

5.6 INTERFERENCE CHECK SAMPLE

The interference check sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples. It is required for ICP methods only. The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for those reported elements.

5.6.1 Deliverables

- CLP Form IV or equivalent for SW-846 methods
- Raw data (required for confirmation)

5.6.2 Frequency

The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A, for all wavelengths used for each analyte reported. An ICS must be run at the beginning of each sample analysis run. The ICS is not to be run prior to the ICV and is to be followed immediately by a CCV, which will be followed by a CCB.

5.6.3 Criteria

Results for the analysis of ICS solution A must fall within the control limits of ± RL, or ± 20% of the true value (whichever is greater) for the analytes and interferents.

Results for the analysis of ICS Solution AB must fall within the control limits of ± RL or ± 20% of the true value (whichever is greater) for the analytes and interferents included in the solution.

If the value of an ICS result exceeds ± RL, or ± 20% of true value (whichever is greater), the analysis should be terminated, the problem corrected, the instrument recalibrated, the new calibration then reverified, and the affected samples reanalyzed

5.6.4 Data Verification

The data verifier shall verify the presence of ICS results. If the results are not provided or if the required frequency of analysis is not demonstrated in the laboratory deliverable, the data verifier will seek to obtain the missing information from the laboratory. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If this missing information cannot be obtained with the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist. As they are contract compliance related, all such occurrences shall be communicated to the SMO and to the data validator on the verification checklist.

5.6.5 Data Validation

The data validator shall review the raw data and recalculate 5% of reported values to ensure results are correct. The data validator will determine if %Rs are within 80-120% recovery criteria and if nonanalyte results are ± RL. Inter-element corrections provided by the laboratory will be verified to determine which elements of interest are interfered with by ICS Solution A.

NOTE: For an ICS for ICP-MS that does not meet the technical criteria, apply the action to all samples reported from the analytical run.

The raw data may not contain results for interferents. In this case, the data validator shall use professional judgment to qualify the data. If the data does contain results for interferents, the data validator should apply the following actions to samples with concentrations of interferents that are comparable to, or greater than, their respective levels in the ICS:

- The ICS %R for an analyte is > 120% (or > the true value + the RL [for ICP-AES] or > the true value + 2× the RL [for ICP-MS] as applicable) and the sample results are nondetects, the data should not be qualified.

- If the ICS %R for an analyte is > 120% (or > the true value + the RL [for ICP-AES] or > the true value + 2× the RL [for ICP-MS] as applicable, qualify sample results that are ≥ MDL as estimated high "J." If the ICS %R (or true value) grossly exceeds the limits, use professional judgment to qualify the data.

- If the ICS %R for an analyte falls within the range of 50-79% (or < the true value -RL [for ICP-AES] or < the true value – 2× the RL [for ICP-MS] as applicable, qualify sample results that are ≥ MDL as estimated low "J."

- If the ICS recovery for an analyte falls within the range of 50-79% (or < the true value -RL [for ICP-AES] or < the true value -2× the RL [for ICP-MS] as applicable, the possibility of false negatives exists. Qualify sample nondetects as estimated "UJ."

- If the ICS Solution AB %R for an analyte or interferent is < 50%, qualify all sample results that are ≥ MDL and all sample nondetects as unusable "R."

If results that are ≥ MDL are observed for analytes which are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify sample results that are ≥ MDL as estimated high "J." Nondetects should not be qualified.

If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is ≥ MDL, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents, qualify nondetects for the affected analytes as estimated "UJ," and results that are ≥ MDL, but < 10× the absolute value of the negative result as estimated low "J."

Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be extremely complex. Use professional judgment to determine the need for the associated sample data to be qualified. The data validator may need to obtain additional information from the laboratory. All interpretive situations then should be recorded in the data validation report.

Interference Check Sample				Qualification Guidance	
Validation Step	Yes	No	N/A	Detects	Nondetects
1. Was the ICS analyzed at the appropriate frequency?				*	*
2. Were all ICS %R within acceptance criteria?				See plan text for guidance	
3. Were samples evaluated for results for elements not present in the ICS solution?				N/A	J
4. Were negative results for elements not present in the ICS solution evaluated?				UJ	N/A

*Qualify only if the deviation indicates an adverse effect on data quality.

5.7 LABORATORY CONTROL SAMPLE

The LCS serves to monitor the overall performance of all steps in the analysis, including sample preparation and instrumental analysis.

5.7.1 Deliverables

- CLP Form VII or equivalent for SW-846 methods
- Raw data (required for confirmation)

5.7.2 Frequency

Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance (QA/QC) procedures as employed for the samples. One LCS shall be prepared and analyzed for each matrix type being analyzed (e.g., aqueous or solid) or for each batch of samples digested, whichever is more frequent. The LCS will accompany no more than 20 samples for an individual matrix type.

5.7.3 Criteria

The LCS recovery should be within the laboratory's acceptable limits. In the absence of laboratory-specific limits, the recovery limit of 70-130% can be used.

The aqueous LCS solution may be provided to the laboratory by EPA. If unavailable, other industry recognized sources of standards will be utilized to obtain known standards for LCS preparation. The LCS solution can come from the same source as the ICV. It may not come from the same source as calibration or continuing calibration standards.

In rare cases, a matrix-specific LCS may not be available. In such cases, an LCS of similar matrix will be selected and analyzed. In absence of a similar matrix, an aqueous LCS may be used by the laboratory. The data validator should make a note if an aqueous LCS was used with solid field samples. If an aqueous LCS used for soil samples is out of %R criteria, careful inspection must be made to determine the effect(s) on sample data. In comparing an aqueous LCS to soil sample data, ensure that units are comparable. An LCS is required for mercury and cyanide analysis in aqueous matrices.

5.7.4 Data Verification

The data verifier shall verify the presence of LCS results. If results are omitted from the laboratory report, the data verifier will contact the laboratory to obtain the omitted information. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If LCS analysis was required but not performed, this is considered a noncorrectable problem and shall be indicated on the data verification checklist. As this is a contract compliance issue, the occurrence should be communicated to the SMO and the data validator on the data verification checklist.

5.7.5 Data Validation

If the LCS criteria are not met, the laboratory performance and method accuracy are in question. Professional judgment should be used to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data associated with an LCS that does not meet the required criteria.

- For an LCS that does not meet the technical criteria, apply the action to all samples in the sample preparation batch.
- Review reported LCS results versus raw data (if provided) to ensure accuracy in the values. Recalculate 5% of reported LCS results to verify laboratory calculations.
- Review LCS types to ensure a matrix-specific LCS has been prepared for each matrix type being quantified in the SDG. If special circumstances are present such that another LCS matrix has been used, ensure laboratory documentation reflects this deviation.
- Determine if LCS performance is acceptable. If recovery criteria have not been met, qualify samples in accordance with Table 7.

NOTE: In the event poor LCS recoveries are observed for antimony and silver, data validators are advised to evaluate results for both elements knowing that both antimony and silver traditionally are very difficult to recover from solid matrices. In most cases, it is prudent to qualify antimony and silver results "J" estimated based on poor LCS recoveries, unless other QC difficulties are observed in conjunction with poor LCS performance.

Table 7. LCS Qualification

LCS %R	Sample Result	Qualification Guidance
40%—lower control limit or 40%–69%*	> MDL	J
	< MDL	UJ
> upper control limit or > 130%*	> MDL	J
	< MDL	No qualification
< 40%	> MDL	J
	< MDL	R

*These limits are used when laboratory defined limits are not available.

Laboratory Control Sample				Qualification Guidance	
Validation Step	Yes	No	N/A	Detects	Nondetects
1. Was the LCS prepared and analyzed at the appropriate frequency?				*	*
2. Was the LCS matrix the same as the analytical samples?				UJ	J
3. Were percent recoveries within acceptable limits?				See plan text for guidance	

*Qualify only if the deviation indicates an adverse effect on data quality.

5.8 MATRIX SPIKE

MS data are generated to determine the accuracy of the analytical method in the specific sample matrices. They provide a sample/project-specific measure of the method's ability to recover target analytes under real sample conditions. See Appendix C for %R calculation.

NOTE: For a MS that does not meet the technical criteria, apply the action to all samples of the same matrix, if the data validator considers the samples sufficiently similar. The data validator will need to exercise professional judgment in determining sample similarity. The data validator should make use of all available data, including site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., total suspended solids (TSS), total dissolved solids (TDS), total organic carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The data validator should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. The data validator may determine that only some of the samples in the data package are similar to the MS sample, and that only these samples should be qualified. The data validator also may determine that no samples are sufficiently similar to the sample used for the MS; thus, may determine only the field sample used to prepare the MS sample should be qualified.

5.8.1 Deliverables

- CLP Form V, Form XII, or equivalent for SW-846 methods
- Instrument printouts
- Raw data (required for confirmation)

5.8.2 Frequency

One MS sample shall be prepared and analyzed for each sample matrix and each analytical method used for analysis of an SDG. The MS will accompany no more than 20 samples for an individual matrix type.

5.8.3 Criteria

- Samples identified as field blanks shall not be used for the preparation and analysis of the MS.

- MS recoveries must be within the control limits defined in Table 8; however, if sample concentration is $\geq 4\times$ the added spike concentration, recovery criteria are not applicable and the data are acceptable for use without qualification.

- A post-digestion spike (PDS) shall be performed for any analyte (except silver) that does not meet the specific criteria. PDS %R must be within 75-125%. PDS are not required for silver. For cyanide, there should be a post-distillation spike instead of post-digestion spike.

- Qualifications will not be applied to data based on the recovery of a "batch" MS/MSD analysis (i.e., when a parent sample is not from the sample set being analyzed).

5.8.4 Data Verification

The data verifier shall verify that field blanks were not used for the MS. If a field blank has been used, the SMO will be notified immediately to ensure timely corrective action. If reanalysis cannot be completed, this issue will be identified as noncorrectable on the data verification checklist.

The data verifier shall verify the presence of MS results. If they are not provided or if the required frequency of analysis is not demonstrated in the laboratory deliverable, the data verifier will seek to obtain the missing information from the laboratory. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If the missing information cannot be obtained from the analytical laboratory they are considered noncorrectable problems and shall be identified in this way on the data verification checklist. As they are contract compliance related, all such occurrences shall be communicated to the SMO and to the data validator on the data verification checklist.

NOTE: If the same sample that was used for duplicate analysis is used for predigestion spike analysis, spike calculations must be performed using the results of the "original sample."

5.8.5 Data Validation

- Review reported MS results versus raw data to ensure accuracy in the values. Recalculate 5% of reported MS results to verify laboratory calculations.

- Review MS types to ensure a matrix-specific MS has been prepared for each matrix type being quantified in the SDG. If special circumstances are present such that an MS has not been used from the associated sample set (e.g., insufficient sample volume), ensure laboratory documentation reflects this deviation.

- Determine if MS performance is acceptable. If recovery criteria have not been met, qualify sample results in accordance with Table 8.

Table 8. Matrix Spike Qualification

Spike Sample Results	Sample Qualification
ICP Methods	
MS %R < 30% PDS %R < 75%	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "R"
MS %R < 30% PDS %R ≥ 75%	Qualify affected results that are ≥ MDL "J" and affected nondetects "UJ"
MS %R = 30-74% PDS %R < 75%	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "UJ"
MS %R = 30-74% PDS %R ≥ 75%	Qualify affected results that are ≥ MDL "J" and affected nondetects "UJ"

Table 8. Matrix Spike Qualification (Continued)

Spike Sample Results	Sample Qualification
ICP Methods	
MS %R > 125% PDS %R > 125%	Qualify affected results that are ≥ MDL "J" (estimated high)
MS %R > 125% PDS %R ≤ 125%	Qualify affected results that are ≥ MDL "J"
MS %R < 30% No PDS performed (not for silver)	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "R"
MS %R = 30-74% No PDS performed (not for silver)	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "UJ"
MS %R > 125% No PDS performed (not for silver)	Qualify affected results that are ≥ MDL "J" (estimated high) and nondetects are not qualified
Mercury Analysis	
MS %R < 30%	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "R"
MS %R = 30-74%	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "UJ"
MS %R > 125%	Qualify affected results that are ≥ MDL "J" (estimated high) and nondetects are not qualified
Cyanide Analysis	
MS %R < 30% Post-distillation spike %R < 75%	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "R"
MS %R < 30% Post-distillation spike %R ≥ 75%	Qualify affected results that are ≥ MDL "J" and affected nondetects "UJ"
MS %R = 30-74% Post-distillation spike %R < 75%	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "UJ"
MS %R = 30-74% Post-distillation spike %R ≥ 75%	Qualify affected results that are ≥ MDL "J" and affected nondetects "UJ"
MS %R > 125% Post-distillation spike %R > 125%	Qualify affected results that are ≥ MDL "J" (estimated high)
MS %R > 125% Post-distillation spike %R ≤ 125%	Qualify affected results that are ≥ MDL "J"
MS %R < 30% No post-distillation spike performed	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "R"
MS %R = 30-74% No post-distillation spike performed	Qualify affected results that are ≥ MDL "J" (estimated low) and affected nondetects "UJ"
MS %R > 125% No post-distillation spike performed	Qualify affected results that are ≥ MDL as estimated high "J" (nondetects are not qualified)

Matrix Spike/Matrix Spike Duplicate (MS/MSD)				**Qualification Guidance**	
Validation Step	Yes	No	N/A	Detects	Nondetects
1. Was the MS/pre-digestion spike analyzed at the appropriate frequency?				--	--
2. Were MS/pre-digestion spike %R within acceptance criteria?				See plan text for guidance	
3. Was the post-digestion spike (or post distillation spike for cyanide) analyzed at the appropriate frequency?				--	--
4. Are post-digestion spike %R within acceptance criteria?				See plan text for guidance	

5.9 DUPLICATES

A laboratory duplicate sample is analyzed for each matrix to evaluate the precision of the laboratory at the time of analysis. A field duplicate sample is collected and analyzed to evaluate the precision of both the sampling techniques as well as the laboratory methodology. A field duplicate also may provide information on the homogeneity of the sample. Nonhomogenous samples can impact the apparent method precision; however, aqueous/water samples generally are homogenous, and most soil/sediment samples are homogenous within a factor of two or three.

5.9.1 Deliverables

- CLP Form VI or equivalent for SW-846 methods
- Raw data (required for confirmation)

5.9.2 Frequency

One laboratory duplicate shall be analyzed in accordance with the methodology being used. Typically, a laboratory duplicated is analyzed per each sample batch or once per 20 samples, whichever is more frequent. Field duplicates are collected at a frequency identified in associated project planning documents (QAPPs, etc.).

5.9.3 Criteria

Samples identified as field blanks must not be analyzed as laboratory duplicates.

For sample concentrations > 5× the RL, the laboratory duplicate precision as measured by relative percent difference (RPD) must be within ± 20% for aqueous and solid samples (lab duplicate). For field duplicates, the RPD must be within ± 25% for aqueous samples and ± 35% for solid samples. If the sample values are < 5× the RL, RPD does not apply. Instead the absolute difference between sample and duplicate must be < 5× the RL.

5.9.4 Data Verification

The data verifier shall verify that field blanks were not analyzed as laboratory duplicates. If a field blank has been used, the SMO will be notified immediately to ensure timely corrective action. If reanalysis cannot be completed, this issue will be identified as noncorrectable on the data verification checklist.

The data verifier shall verify the presence of laboratory and/or field duplicate results. If they are not provided or if the required frequency of analysis is not demonstrated in the laboratory deliverable, the data verifier will seek to obtain the missing information from the laboratory. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If the missing information cannot be obtained from the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist. As they are contract compliance related, all such occurrences shall be communicated to the SMO and to the data validator on the data verification checklist.

5.9.5 Data Validation

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to samples of the same matrix if the data validator considers the samples to be sufficiently similar. The data

validator will need to exercise professional judgment in determining sample similarity. The data validator should make use of all available data, when determining similarity, including the following: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions). The data validator should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The data validator may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or the data validator may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus only the field sample used to prepare the duplicate sample should be qualified.

- Examine the raw data (if provided) for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility).
- Verify that appropriate methods and amounts were used in preparing the samples for analysis.
- Verify that there are no transcriptions or reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.
- Verify that results fall within the linear range(s) of the ICP instruments.

Table 9. Lab and Field Duplicate Qualification

Duplicate Type	Matrix	RPD	Sample Results	Qualification Instructions
Laboratory Duplicate	Aqueous	> 20%	Sample and duplicate > 5× RL	Qualify results > RL "J" Qualify nondetects "UJ"
	Solid	> 20%		
	Aqueous	> 20%	Sample and duplicate < 5× RL	Absolute difference > RL "J" Absolute difference < RL no action
	Solid	> 20%		
Field Duplicate	Aqueous	> 25%	Sample and duplicate > 5× RL	Qualify results > RL "J" Qualify nondetects "UJ"
	Solid	> 35%		
	Aqueous	> 25%	Sample and duplicate < 5× RL	Absolute difference > RL "J" Absolute difference < RL no action
	Solid	> 35%		

The above control limits are method requirements for matrix-specific duplicate samples. It should be noted that laboratory variability arising from the subsampling of nonhomogenous matrices is a common occurrence; therefore, for technical review purposes only, regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 5× RL) to be used in assessing nonhomogeneous matrices. When project-specific DQOs mandate broader precision requirements, this information will be provided to the data validators as part of the validation SOW.

Duplicate				**Qualification Guidance**	
Validation Step	Yes	No	N/A	Detects	Nondetects
1. Was the laboratory duplicate prepared and analyzed at the appropriate frequency?				*	*
2. Were reported precision estimates for the laboratory and/or field duplicate(s) within acceptance criteria?				See plan text for guidance	

*Qualify only if the deviation indicates an adverse effect on data quality.

5.10 SERIAL DILUTION ANALYSIS

Serial dilution (SD) analysis determines whether significant physical or chemical interferences from the matrix spike are present and are affecting the analysis of samples. This dilution is prepared from a selected digested sample. SD is only applicable for ICP methods.

5.10.1 Deliverables

- CLP Form VIII or equivalent for SW-846 methods
- Raw data (required for confirmation)

5.10.2 Frequency

An ICP serial dilution analysis shall be performed on a sample from each group of samples with a similar matrix type (e.g., water or soil) or for each SDG, whichever is more frequent.

5.10.3 Criteria

- Field Blanks and Preparation Blanks must not be used for the serial dilution analysis.

- For ICP analysis, if analyte concentration is > 50× MDL, the SD analysis (a five-fold dilution) must agree within 10% difference of the original.

NOTE: The above criteria are method requirements for SD samples, regardless of the sample matrix type; however, for technical review purposes only, project DQOs may allow the use of less restrictive criteria (e.g., %D < 15) to be assessed against serial dilution soil samples.

5.10.4 Data Verification

The data verifier shall verify that field blanks and preparation blanks were not used for the SD analysis.

The data verifier shall verify the presence of SD results. If results are not provided or if the required frequency of analysis is not demonstrated in the laboratory deliverable, the data verifier will seek to obtain the missing information from the laboratory. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If the missing information cannot be obtained from the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist. As they are contract compliance related, all such occurrences shall be communicated to the SMO and the data validator on the data verification checklist.

5.10.5 Data Validation

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to all samples of the same matrix if the data validator considers the samples sufficiently similar. The data validator will need to exercise professional judgment in determining sample similarity. The data validator should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions), in determining similarity. The data validator should also use the sample data (e.g., similar concentrations

of analytes) in determining similarity between samples in the SDG. The data validator may determine that only some of the samples in the SDG are similar to the serial dilution sample, and that only these samples should be qualified. Or the data validator may determine that no samples are sufficiently similar to the sample used for serial dilution, and thus only the field sample used to prepare the serial dilution sample should be qualified.

- Review reported SD results versus raw data to ensure accuracy in the values. Recalculate 5% of reported SD results to verify laboratory calculations. See Appendix C for %D calculation.

- Review SD types to ensure a matrix-specific SD has been prepared for each matrix type being quantified in the SDG.

- Determine if SD performance is acceptable. If SD %D > 10%, verify if undiluted sample result is > 50× the MDL. Qualify using the following guidance:

  — If undiluted sample result < 50× the MDL, no qualification of results is warranted.

  — If undiluted sample result > 50× the MDL, qualify associated sample results ≥ MDL "J" and nondetects "UJ."

If negative interference is found (i.e., results of diluted samples are higher than the original sample), use professional judgment in qualifying data.

Serial Dilution Analysis				**Qualification Guidance**	
Validation Step	Yes	No	N/A	Detects	Nondetects
1. Was the serial dilution analyzed at the appropriate frequency?				--	--
2. Was the serial dilution %D criterion satisfied?				See plan text for guidance	

5.11 INTERNAL STANDARDS

The analysis of internal standards determines the existence and magnitude of instrument drift and physical interferences and is applicable for ICP-MS analyses only. The criteria for evaluation of internal standard results apply to all analytical and QC samples analyzed during the run, beginning with the calibration.

5.11.1 Deliverables

Form XIII-IN, Form XV-IN, Form XVII-IN, instrument printouts, and raw data.

5.11.2 Frequency

All samples analyzed during a run, with the exception of the ICP-MS tune, shall contain internal standards. A minimum of five internal standards from the following list shall be added to each sample: Li (the ^{6}Li isotope); Sc; Y; Rh; Tb; Ho; Lu; and Bi. If the laboratory uses lithium as an internal standard, the laboratory shall use an ^{6}Li-enriched standard. The laboratory shall monitor the same internal standards throughout the entire analytical run and shall assign each analyte to at least one internal standard.

5.11.3 Criteria

The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The percent relative intensity (%RI) in the sample shall fall within 60-125% of the response in the calibration blank.

If the %RI of the response in the sample falls outside of these limits, the laboratory shall reanalyze the original sample at a two-fold dilution with internal standard added.

5.11.4 Data Verification

The data verifier shall verify that an internal standard has been analyzed and reported for ICP-MS analyses.

The data verifier shall verify the presence of ICP-MS internal standards results. If they are not provided or if the required frequency of analysis is not demonstrated in the laboratory deliverable, the data verifier will seek to obtain the missing information from the laboratory. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If the missing information cannot be obtained from the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist. As they are contract compliance related, all such occurrences shall be communicated to the Sample & Data Management manager and the data validator on the data verification checklist.

5.11.5 Data Validation

NOTE: Apply the action to the affected analytes for each sample that does not meet the internal standard criteria.

If no internal standards were analyzed with the run, the sample data should be qualified as unusable "R."

If fewer than five of the required internal standards were analyzed with the run or a target analyte(s) is (are) not associated to an internal standard, the sample data, or analyte data not associated to an internal standard, should be qualified as unusable "R."

If the %RIs for all internal standards in a sample is within the 60-125%, the sample data should not be qualified.

If the %R for an internal standard in a sample is not within the 60-125%, qualify the data for those analytes associated with the internal standard(s) outside the limit as follows:

- If the sample was reanalyzed at a two-fold dilution with internal standard %RI within the limits, report the result of the diluted analysis without qualification. If the %RI of the diluted analysis was not within 60–125%, report the results of the original undiluted analyses and qualify the data for all analytes that are ≥ MDL in the sample associated with the internal standard as estimated "J," and nondetected analytes associated with the internal standard as estimated "UJ."

- If the sample was not reanalyzed at a two-fold dilution, the data validator should use professional judgment to determine the reliability of the data. The data validator may determine that the results are estimated "J" or unusable "R."

5.12 SAMPLE RESULT CONFIRMATION

Raw data should be requested based on the level of review by the data validator and based on records requirements of the project.

If the laboratory has a high rate of manual transcription in generation of sample results, the project team may choose to recalculate manually the sample results at a determined frequency. If sample results cannot be reproduced through manual calculation, contacting the laboratory may be necessary to resolve the problem. Data may be qualified "R" as a last resort, if no actions can reproduce reported values.

If results are to be recalculated manually from raw data, the following strategy is recommended:

- Examine raw data for anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).
- Verify from raw data two detected and two nondetected results for ICP analysis and two detected and two nondetected results for cyanide analyses in each SDG. For aqueous sample results, use the concentration reported in raw data; for soils, use equation C.7 in Appendix C to convert concentrations in per-volume in raw data to per-weight.
- Confirm from raw digestion logs that initial sample volumes are equivalent to final digestate sample volumes for ICP digestions. If volumes differ, confirm that sample results have been corrected for the difference in final vs. initial volumes.
- Confirm that results fall within linear range of the ICP and within calibration range for other non-ICP parameters.
- All analyses must fall within the calibration range. If outside, confirm that dilution results are corrected for dilution factor(s).
- Verify that appropriate methods and amounts were used in preparing the samples for analysis.

Sample Result Verification			
Validation Step	Yes	No	N/A
For the following evaluation, some qualification of sample data may be possible. For contractual noncompliance, a validation code is placed if the occurrence is noncorrectable.			
1. For each SDG, recalculate 2 detected and 2 nondetected results for each inorganic chemistry method from the raw data (applies to Level IV validation only).			
2. Did recalculation confirm reported results? If not, increase the frequency of recalculations until adequate confidence is gained in the reported results (applies to Level IV validation only)?			
3. Were reported results within the calibration range of the instrument?			
4. Were results from diluted samples corrected for the dilution factor?			

Action: Indicate instances of manual calculations not confirming reported results; where samples have been reanalyzed and both analyses are included in the data package, indicate on the laboratory reporting forms which results are the most reliable.

6. RECORDS

Generate and maintain all records in accordance with CP3-RD-0010, *Records Management Process.*

- Data Verification Checklist (for Level II, III, and IV validation only)
- Data Validation Report (for Level III and Level IV validation only)
- Copies of qualified or unqualified results reports (if applicable)

7. REFERENCES

NOTE: The most current versions of the references listed below should be accessed when using this plan for the data review, verification, and validation process.

EPA (U.S. Environmental Protection Agency) 2010. *Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review,* EPA-540/R 10-011, U.S. Environmental Protection Agency, Washington, DC, January.

EPA 2001. *EPA Requirements for Quality Assurance Project Plans*, EPA QA/R-5, U.S. Environmental Protection Agency, Washington, DC, March.

EPA 2006. *Guidance on Systematic Planning Using the Data Quality Objective Process* EPA QA/G-4, U.S. Environmental Protection Agency, Washington, DC, February.

EPA 1983. *Methods for Chemical Analysis of Water and Wastes,* EPA-600/4-79-020, U.S. Environmental Protection Agency, Washington, DC, March.

CP3-ES-5003, *Quality Assured Data.*

EPA 2009. *USEPA Test Methods for Evaluating Solid Waste*, Revisions through Update III, SW-846 Final Update IV of the Third Edition, U.S. Environmental Protection Agency, Washington, DC, March.

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APPENDIX A

DATA VALIDATION QUALIFIERS AND QUALIFICATION CODES

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DATA VALIDATION QUALIFIERS AND QUALIFICATION CODES

Data Validation Qualifiers

U Analyte compound or nuclide considered not detected above the reported detection limit.
J Analyte compound or nuclide identified; the associated numerical value is approximated.
NJ Analyte compound or nuclide presumptively present at an estimated quantity.
UJ Analyte compound or nuclide not detected above the reported detection limit, and the reported detection limit is approximated due to quality deficiency.
R Result is not usable for its intended purpose.
= "Equals" sign, indicates that no qualifier is necessary.

Data Validation Qualification Codes

Blanks
B01 Sample concentration was < the RL, and < 5× the blank concentration (10× for common contaminants).
B02 Sample concentration was > the RL, and < 5× the blank concentration (10× for common contaminants).
B03 Gross contamination exists; blank result impacted associated analyte data quality.
B04 Negative blank result impacted associated analyte data quality.
B05 Blanks were not analyzed at appropriate frequency.
B06 Sample not significantly different than radiochemical method blank.
B07 Blank data not reported.
B08 Instrument blank not analyzed after high level sample.
B09 Other (describe in comments)
B10 Method blanks not extracted at appropriate frequency.
B11 Sample results were corrected for blank contamination.
B12 Blank was not the same matrix as the analytical samples.
B13 Concentration of target compound detected in sample affected by carryover.

Calibration
C01 Initial calibration average RRF was < 0.05
C02 Initial calibration %RSD was exceeded
C03 Initial calibration sequence was not follows as appropriate
C04 Continuing calibration RRF was < 0.05
C05 Continuing calibration %D was exceeded
C06 Calibration or performance check was not performed at the appropriate frequency
C07 Calibration data not reported
C08 Calibration not performed
C09 Chemical resolution criteria were not satisfied
C10 Calibration standard matrix not the same as sample matrix
C11 Compounds quantitated against inappropriate standard or standard concentration level
C12 Compound quantitated against inappropriate ion
C13 Calibration factor RSD criteria were not satisfied
C14 Retention time of compound outside window
C15 Initial calibration % R was below lower acceptance limit
C16 Initial calibration % R was above upper acceptance limit
C17 Initial calibration curve fit was < 0.995

C18 Inappropriate standard concentrations
C19 Continuing calibration R was below the lower acceptance limit
C20 Continuing calibration %R was above the upper acceptance limit
C21 CRI %R was below the lower acceptance limit
C22 CRI %R was above the upper acceptance limit
C24 Standard curve was established with fewer than the appropriate number of standards
C27 Calibration verification efficiency outside control criteria
C28 Calibration verification background outside control criteria
C29 Calibration verification energy outside control criteria
C30 Calibration verification peak resolution outside control criteria
C31 Chromatogram does not show adequate gain setting
C32 Other (describe in comments)

Laboratory Duplicate/Dual Column Sample Confirmation
D01 Significant difference between sample and duplicate
D02 Laboratory duplicate was not analyzed at the appropriate frequency
D03 Laboratory duplicate exceeds RPD criteria
D04 Laboratory duplicate data not reported
D05 Other (describe in comments)
D06 %D between primary and secondary column confirmation exceeds acceptance criteria

Evidentiary Concerns
E01 Custody of sample in question
E02 Standard not traceable
E03 Other (describe in comments)

Interference Check Samples (ICS)
F01 ICS recovery below lower control limit or advisory limit
F02 ICS recovery above upper control limit or advisory limit

General
G01 Professional judgment was used to qualify the data
G02 Other (describe in comments)

Holding Times/Preservation
H01 Extraction holding times were exceeded
H02 Extraction holding times were grossly exceeded
H03 Analysis holding times were exceeded
H04 Analysis holding times were grossly exceeded
H05 Samples were not preserved properly
H06 Sample preservation cannot be confirmed
H07 Sample temperature exceeded criteria prior to preparation
H08 Other (describe in comments)

Internal Standards
I01 Area count was above upper control limits
I02 Area count was below lower control limits
I03 Extremely low area counts or performance was exhibited by a major drop off
I04 Internal standard retention time varied by more than 30 seconds
I05 Inappropriate internal standard used
I06 Inappropriate internal standard concentration(s) used

I07 Internal standard data not reported
I08 Other (describe in comments)

Laboratory Control Sample
L01 LCS recovery above upper control limit
L02 LCS recovery below lower control limit
L03 LCS was not analyzed at appropriate frequency
L04 LCS not the same matrix as the analytical samples
L05 LCS data not reported
L06 Other (describe in comments)

Matrix Spike and MS/MSD
M01 MS and/or MSD recovery above upper control limit
M02 MS and/or MSD recovery below lower control limit
M03 MS/MSD pair exceeds the RPD limit
M04 MS and/or MS/MSD not analyzed at the appropriate frequency
M05 MS and/or MS/MSD data not reported
M06 Other (describe in comments)

Instrument Performance
P01 High background levels or a shift in the energy calibration were observed
P02 Extraneous peaks were observed
P03 Loss of resolution was observed
P04 Peak tailing or peak splitting that may result in inaccurate quantitation were observed
P05 Instrument performance data not reported
P06 Instrument performance not analyzed at the appropriate frequency
P07 Other (describe in comments)
P08 Resolution Check Mixture (RCM) not analyzed at the beginning of the initial calibration sequence
P09 RCM criteria were not met
P10 RPD criteria in Performance Evaluation Mixture (PEM) was not met

Quantitation
Q01 Peak misidentified
Q02 Target analyte affected by interfering peak
Q03 Qualitative criteria were not satisfied
Q04 Cross contamination occurred
Q07 Analysis occurred outside 12 hour GC/MS window
Q09 TIC result was not above 10× the level found in the blank
Q10 TIC reported as detect in another fraction
Q11 Common artifact reported as a TIC
Q12 No raw data were provided to confirm quantitation
Q13 MDA > RL
Q14 Inappropriate aliquot sizes were used
Q15 Sample result < MDA
Q16 Sample result < 2σ uncertainty
Q17 Negative result
Q18 Compounds were not adequately resolved
Q19 Sample geometry different from calibration geometry
Q20 Sample weight greater than greatest weight on mass attenuation curve
Q21 Isotopes of same radionuclide do not show equilibrium

Q22 Peak not within appropriate energy range
Q23 Counting uncertainty ≥ 80% of sample result
Q24 Raw data anomaly
Q25 Other (describe in comments)
Q26 RT outside calculated RT window
Q28 Neither RL or the SQL are reported for a nondetect result
Q29 SQL > RL
Q30 Compound detected at < SQL and not qualified "J"
Q31 Presence of high molecular weight contaminants impacted sample quantitation

Surrogates
S01 Surrogate recovery was above the upper control limit
S02 Surrogate recovery was below the lower control limit
S03 Surrogate recovery was < 10%
S04 inappropriate surrogate standard used
S05 Inappropriate surrogate standard concentration(s) used
S06 Surrogate data not reported
S07 Surrogate outside retention window
S08 Other (describe in comments)

Instrument Tuning
T01 Mass calibration ion misassignment
T02 Mass calibration was not performed every 12 hours
T03 Mass calibration did not meet ion abundance criteria
T04 Mass calibration data was not reported
T05 Scans were not properly averaged
T06 Other (describe in comments)

Pesticide Sample Cleanup
U01 Florisil performance requirements not met
U02 GPC calibration not checked at required frequency
U03 GPC calibration criteria not met
U04 GPC blank not analyzed after GPC calibration
U05 GPC blank greater than half the RL for target compound

Cleanup
V01 10% recovery or less was obtained during either check
V02 Recoveries during either check were > 120%
V04 Cleanup data not reported
V05 Cleanup check not performed at the appropriate frequency
V06 Other (describe in comments)

Dilutions
X01 Serial dilution not analyzed at the appropriate frequency
X02 %D between the original sample and the diluted result (or serial dilution) exceeded acceptance criteria
X03 Reported results not corrected for dilution factor
X04 Other (describe in comments)

Radiochemical Yield

Y01 Radiochemical tracer yield was above the upper control limit
Y02 Radiochemical tracer yield was below the lower control limit
Y03 Radiochemical tracer yield was zero
Y04 Radiochemical yield data was not present
Y05 Other (describe in comments)

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APPENDIX B

QUALIFICATION TABLES FOR MULTIPLE QUALITY DEFICIENCIES

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QUALIFICATION TABLES FOR MULTIPLE QUALITY DEFICIENCIES

GUIDANCE FOR DATA QUALIFICATION DUE TO MULTIPLE QUALITY DEFICIENCIES

This appendix provides guidance in the qualification of data due to instances of multiple quality deficiencies. Quality deficiencies can be categorized based on potential effect on sample data. The effect of quality deficiencies may be applicable to only a single sample or to all samples within the reporting batch. A validation qualifier should not be placed on sample data until all quality deficiencies have been identified within the reporting batch.

The following is a listing of data quality indicators and the probable effects on sample data.

Data Quality Indicator	Effect on Sample Data
Standard curve correlation coefficient	Quantitative uncertainty
Continuing calibration verification	Positive or negative bias
Method blank	Positive bias
Laboratory control sample	Positive or negative bias and precision
MS/MSD	Positive or negative bias and precision

In the instance of multiple quality deficiencies, the validation qualifier should be placed consistent with the acceptable level of uncertainty associated with the intended use of the data. The validation SOW should provide a summary of the intended use(s) of the data. (e.g., risk assessment, fate and transport modeling, waste management) to facilitate appropriate placement of validation qualifiers.

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APPENDIX C

RULES, CALCULATIONS AND EQUATIONS

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RULES, CALCULATIONS, AND EQUATIONS

Rounding Rules

1. In a series of calculations, carry the extra digits through to the final result, and then round off.
2. If the digit to be removed is less than 5, the preceding digit stays the same.
3. If the digit to be removed is equal to or greater than 5, the preceding digit is increased by 1.

Calculations/Equations

C.1 Initial/Continuing Calibration Verification %R

$$\%R = \frac{ICV_{Found}}{ICV_{True}} \times 100$$

Where,
Found = concentration (µg/L) of each analyte measured in the ICV or CCV solution
True = concentration (in µg/L) of each analyte in the ICV or CCV source

C.2 Interference Check Sample %R

$$\%R_{ICSAB} = \frac{AB_{Found}}{AB_{True}} \times 100$$

Where,
Found = concentration (µg/L) of each analyte measured in the ICS solution
True = concentration (in µg/L) of each analyte in the ICS

C.3 Laboratory Control Sample %R

$$\%R_{LCS} = \frac{LCS_{Found}}{LCS_{True}} \times 100$$

Where,
Found = concentration (µg/L for aqueous; mg/kg for solid) of each analyte measured in the LCS solution
True = concentration (in µg/L for aqueous; mg/kg for solid) of each analyte in the LCS source

C.4 Laboratory Duplicate RPD

$$RPD = \frac{|R1 - R2|}{\bar{x}_{R1,R2}} \times 100$$

Where,
R1= first sample value (original)
R2 = second sample value (duplicate)

C.5 MS/Pre-digestion Spike %R

$$\%R_{PdS} = \frac{\text{Spiked Sample Result - Sample Result}}{\text{Spike Added}} \times 100$$

C.6 Serial Dilution %D

$$\%D = \frac{\text{Initial Result - Dilution Result}}{\text{Initial Result}} \times 100$$

C.7 Conversion of µg/L to mg/kg

$$\frac{mg}{kg} = \frac{ug}{L} \times \frac{vol(mL)}{wt(g)} \times \frac{1L}{1000mL} \times \frac{1000g}{Kg} \times \frac{1mg}{1000ug}$$

Where,
µg/L = concentration from raw data
vol = digestate volume in liters
wt = sample weight (1 g)

C.8 Conversion of soil/sediment wet weight to dry weight

$$\frac{mg}{kg} = \frac{mg}{kg}(wet) \times \frac{100}{\%solids}$$