U.S. Environmental Protection Agency, Region 2
Field Operations Quality Procedures

ADMINISTRATIVE STANDARD OPERATING PROCEDURE

Standard Operating Procedure for the Validation of ICP-AES Data

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The table below identifies information about the reviews conducted of this SOP.

REVIEW HISTORY		
Date	Reviewer Name	Changes Required (Y/N)

The table below identifies changes to this controlled document and the respective effective date(s) over time.

REVISION HISTORY		
Revision Number	Revision Description	Effective Date
0	Original Issue (Note: Replaces SOP HW-3a, Rev. 1 ISM02.2 – ICP-AES Data Validation, September 2016)	

NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (USEPA) and other governmental employees. They do not constitute rule-making by the USEPA and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at a variance with the policies and procedures in this Standard Operating Procedure (SOP).

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1.0 PURPOSE AND APPLICABILITY

This document is designed to promote uniformity of data review of analytical data generated through the US EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Superfund Analytical Methods SFAM01.1 and any future editorial revisions of SFAM01.1. It is applicable to the review of Contract Laboratory Program (CLP) data of various matrices (water, soil, sediment, waste, wipes, etc.) generated using Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) for metal analyses.

The guidelines presented in this document will aid in establishing (a) if data meets the specific technical and quality control (QC) criteria established in the SOW, and (b) the validity and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the user that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to, site-specific technical requirements, the need to facilitate the progress of specific projects, and the availability for re-sampling. The user should note that while this document is to be used as an aid in the formal data review process, the site-specific quality assurance project plan (QAPP), as well as professional judgement, should also be used to determine the ultimate validity of data, especially in those cases where all data do not meet specific technical criteria.

2.0 SUMMARY OF PROCESS OR METHODOLOGY

This document provides the criteria for performing technical quality assurance reviews of metal data generated by the CLP. Criteria are based on the quality assurance/quality control and technical requirements specified in Exhibit D of SOW SFAM01.1. This SOP incorporates much of the content of the National Functional Guidelines (NFG) and provides additional guidance specific to EPA Region 2.

Upon receipt by EPA Region 2, CLP data in the Sample Delivery Group (SDG) undergoes a technical quality assurance review based upon the criteria in this document. A report of this review is prepared by the data validator, reviewed by the EPA Task Order Contracting Officer Representative (TOCOR), and provided to the data user.

3.0 DEFINITIONS

3.1. **See Appendix C** – Definitions/Glossary of Terms

3.2. Acronyms and Abbreviations

The following acronyms and abbreviations are applicable to this document.

%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%RSD	Percent Relative Standard Deviation
%Solids	Percent Solids, (also **%S**)
ASB	Analytical Services Branch
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CLPSS	Contract Laboratory Program Support System
DAR	Data Assessment Report
DF	Dilution Factor
DL	Detection Limit
DV	Data Validation
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
EDS	Environmental Data Services
EICC	Electronic Internal Chain of Custody
EPA	Environmental Protection Agency (see also **USEPA**)
ESAT	Environmental Services Assistance Team
EXES	Electronic Data Exchange and Evaluation System
HWSS	Hazardous Waste Support Section
ICB	Initial Calibration Blank
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ICS	Interference Check Sample
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LEB	Leachate Extraction Blank
MDL	Method Detection Limit
MS	Matrix Spike
NFG	National Functional Guidelines
OSRTI	Office of Superfund Remediation and Technology Innovation
PDF	Portable Document Format
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center Coordinator
SAP	Sampling and Analysis Plan

SDG	Sample Delivery Group
SEDD	Staged Electronic Data Deliverable
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SP	SharePoint
SPLP	Synthetic Precipitation Leaching Procedure
TCLP	Toxicity Characteristic Leaching Procedure
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TOCOR	Task Order Contracting Officer Representative
TR/COC	Traffic Report/Chain of Custody
TSS	Total Suspended Solids
USEPA	United States Environmental Protection Agency

3.3. Data Qualifier Definitions

Data qualifier definitions are provided in the beginning of Appendix A.

4.0 RESPONSIBILITIES/QUALIFICATIONS

4.1. Qualifications

Data Validators must be familiar with the current CLP SOW, EDM and the documents referenced in Section 5.0 below.

4.2. Responsibilities

4.2.1. EPA TOCOR (when applicable) – will review data assessments reports and other deliverables prepared by contract data validators. They will update the MS Planner DV Flowboard indicating the progress of SDGs, post final deliverables to the EDS SharePoint site and send notification to clients via the established workflow.

4.2.2. Data Validator – will follow the criteria and actions provided in this document and prepare Data Assessment Reports (DAR) and Summary Reports, as necessary. If the validator is an ESAT contractor employee, they will consult the EPA TOCOR when questions arise. They will update the DV Flowboard indicating progress of SDGs.

5.0 REFERENCES

National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA 540-R-20-006, November 2020.

Contract Laboratory Program (CLP) Statement of Work (SOW) Superfund Analytical Method (SFAM) SFAM01.1

FA-0010.1, Standard Operating Procedure for Development and Use of Field SOPs, December 2015.

U.S. EPA, 2007. Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents. EPA QA/G-6, EPA/600/B-07/001. April 2007.

QA-HWSS-A-001, Document Control Room, Data Dissemination and Archive Operations. Revision 0, January 2021.

6.0 PROCEDURAL STEPS

6.1. EXES Processing

At the Sample Management Office (SMO) the data package and electronic data deliverables (EDD) are checked for compliance with the CLP SOW. A Contract Compliance Screening Report (CCS) is issued and posted on the SMO portal web site. The EDD is processed electronically to evaluate QC performance against the NFG and Region 2 criteria by EXES. An electronic report of the EXES review is also posted on the SMO portal website.

6.2. Initial Notification

The EICC SharePoint web application is setup to send an e-mail alert notification to EPA and ESAT data validators when a new data package is received and available for review and validation. Entry of data into the EICC SharePoint site will automatically trigger an e-proxy card to populate on the DV Flowboard in MS Planner.

Alternate electronic systems may be applied in the future.

6.3. DV Flowboard Updates

Updates to the DV Flowboard will be performed as per SOP QA-HWSS-A-001, Document Control Room, Data Dissemination and Archive Operations (or most current version).

6.4. Data Package Inspection

The EXES Data Manager (EDM) is a useful tool in the data review process. EDM will identify any missing and/or incorrect information in the data package. When available, the EDM should be reviewed as part of the initial data package inspection. The CLP laboratory may submit a reconciliation package for any missing items or to correct the data. If there are any concerns regarding the data package, contact the TOCOR.

An initial review of the data package is to be performed, taking into consideration all information specific to the sample data package, (e.g., modified analysis requests, trip report/chain-of-custody documentation, SDG narratives, etc.). The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The criteria for data validation outlined in the QAPP will supersede that in this SOP. The reviewer should access the HWSS SP Documents Dashboard to obtain a copy of the relevant documents.

The SDGs or cases routinely have unique samples that require special attention from the reviewer. These include field blank, equipment blanks, trip blanks, and field duplicates which must be identified in the sample records. The sampling records (i.e., trip reports or COC records) should identify:

1) The Region where the samples were taken,
2) The case number,
3) The complete list of samples with the following information, as applicable:
    a. Sample matrix,
    b. Field blanks (i.e., equipment, rinsate and trip),
    c. Field duplicates,
    d. Field spikes,
    e. Shipping dates,
    f. Preservatives, and
    g. Laboratories involved

6.5. Data Review/Validation

The EXES electronic validation will apply most of the criteria and actions provided in Appendix A. The data validator will examine the EXES report to identify any issues that warrant further investigation. All EXES rejected data will be manually evaluated. The data validator will use the criteria and actions in Appendix A, as well as their own professional judgement to manually assess the data.

To use this SOP effectively, the reviewer should understand the analytical method. The exact number of samples, their assigned numbers, type of matrix, and the number of laboratories involved in the analysis are essential information for the validator.

The TR/COC documentation includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation and unusual events should be documented in the SDG narrative. The reviewer should also inspect any email, or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office and USEPA Region 2.

All data are initially marked as “Reportable” (YES) in EDM before validation is begun. Sometimes, due to dilutions and/or re-analyses being performed, there may be multiple results for a single analyte from a sample. The following criteria and professional judgement are used to determine which result should be reported:

1) the analysis with the lower QL,
2) the analysis with the better QC results, and/or
3) the analysis with the higher result

Data validator will reconcile results from the multiple runs to provide results in one run and report. The analyte values and their respective QLs are then transferred into a single sample run. The runs and results that are not to be used are marked “not reportable” or entered “NO” in the “Reportable” fields of the EDM.

6.6. Data Assessment Report

The data validator will prepare a Data Assessment Report documenting the results of their data review. This report will be formatted in accordance with the template provided in Appendix B. Modifications to the template are allowed at the discretion of the user.

6.7. Summary Report

If requested by the client on the Analytical Request Form, the data validator will prepare a Summary Report using the HWSS Summary Report application.

7.0 DATA AND RECORDS MANAGEMENT

7.1. DATA MANAGEMENT

Posting data to the SP EDS site is done in accordance with QA-HWSS-A-001, “Document Control Room, Data Dissemination and Archive Operations”.

7.2. RECORDS MANAGEMENT

The data files uploaded to the EDS SharePoint site include:

1) Data Assessment Report (Adobe PDF),
2) Edited/Validated Sample Summary Report from SMO portal (Adobe PDF),
3) Edited/Validated EQuIS EDD report from SMO portal (MS Excel),
4) Generated Summary Report (MS Excel), if applicable, and
5) Generated Summary Report with Hits Only (MS Excel), if applicable.

In addition to the above stated documents, data validators also forward the following EXES files, which are not uploaded to the EDS SharePoint:

6) The CCS Report from the SMO Portal (Adobe PDF), .
7) Edit History Report from the SMO Portal (Adobe PDF)

All files stated above are saved to the Local Area Network (LAN) G: drive at DESADIV/HWSS/DATA VALIDATION/Site Name/Case #/SDG #. Files are renamed using the following naming convention, Case#_SDG#_Filetype.*, e.g., 12345_MBAB12_S3VEM.xlsx or 12345_BAB12-M_S3VEM.xlsx.

Note: The letter "M" in the beginning of the SDG name or appended as "-M" signifies that the analyses are inorganic. "M" in the file type signifies that the data has been manually validated by ESAT and/or EPA Staff.

Additional records management procedures are discussed in QA-HWSS-A-001, "Document Control Room, Data Dissemination and Archive Operations".

8.0 QUALITY ASSURANCE AND QUALITY CONTROL

8.1. This SOP will be reviewed annually. Reviews will be documented on the Review History Table on page 2 of the SOP. The SOP shall be updated every 5 years, or more frequently, when necessary, due to significant changes.

8.2. The "Request for SOP Change Form", Appendix D is used to document changes and is appended to the final SOP until such time as the changes are incorporated into the body of the text of the SOP.

9.0 APPENDICES

Appendix A - Data Validation Criteria and Actions

Appendix B - Data Assessment Report Template

Appendix C - Definitions/Glossary of Terms

Appendix D - SOP Change Request Form (CRF)

Appendix A

Data Validation Criteria and Actions

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I. Data Qualifier Definitions

The following table provides brief explanations of the qualifiers assigned to results during the data review process. The reviewer should use these qualifiers as applicable.

ICP-AES Table 1. Data Qualifier Definitions

Data Qualifier	Definition
U	The analyte was analyzed for but was not detected above the level of the adjusted detection limit or quantitation limit, as appropriate.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

NOTES:

1. Comments for sample results with data qualifiers other than "U" or no qualification based on professional judgement must be included in the DAR.
2. With familiarity of project data objectives and/or consultation with project staff, the reviewer should be able to refine the use of data qualifiers to avoid ambiguity. For example, if critical site decisions are to be made based on the data, the reviewer may decide to apply an "R" qualifier rather than a "UJ".
3. Although a "J+" or a "J-" may be seen as less ambiguous than a "J", the reviewer should reserve the application of directional bias indicators to those situations when there is an overwhelming influence in one direction. The exercise of professional judgment is critical, especially in situations where ambiguity exists due to opposing factors, to objectively interpret the effects of all factors.
4. Criteria, evaluation, quantitation limits (QLs), calculations, acceptable ranges and related parameters and definitions are detailed in the applicable Statement of Work (SOW) and/or National Functional Guidelines (NFG) documents referenced.

II. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, raw data, and narrative in the data package, checking for: pH, shipping container temperature, holding time, and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on the sample shipping and storage conditions and the holding time of the sample.

C. Action:

Refer to ICP-AES Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

1. If a discrepancy is found between the sample analysis dates on the Laboratory Results Reports and the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.
2. When holding times are grossly exceeded, note it for Contract Laboratory Program Contracting Officer Representative (CLP COR) action.

ICP-AES Table 2. Preservation and Holding Times Actions

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with pH ≥ 2 and pH adjusted by laboratory	No qualification	No qualification
Aqueous/water samples received with pH ≥ 2 and pH not adjusted	J-	R
TCLP/SPLP leachates with pH ≥ 2 and pH not adjusted	J-	R
Technical Holding Time: Aqueous/water and TCLP/SPLP leachates > 180 days	J-	R
Technical Holding Time: Soil/sediment/waste/wipe samples > 180 days	J-	R
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification

III. Calibration

A. Review Items

Laboratory initial calibration and calibration verification reports (if available), preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data in the data package.

B. Objective

The objective is to determine the validity of the analytical results based on initial calibration and calibration verification.

C. Action:

Refer to ICP-AES Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient initial calibrations or calibration verification standard.

1. For initial calibrations or ICV standard analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCV standards analyses that do not meet the technical criteria, apply the actions to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical sequence.
3. If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and at least one at or below the QL but above the MDL), qualify detects as estimated (J) and non-detects as estimated (UJ).

NOTE: For critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

ICP-AES Table 3. Calibration Actions

Criteria	Action	
	Detect	Non-detect
Calibration not performed or not performed at specified frequency	R	R
Calibration incomplete (insufficient number of standards or required concentrations missing)	J	UJ
For linear fits, the correlation coefficient < 0.995	J	UJ
%D outside ± 30%; or other specified statistical test values outside limits	J	UJ

ICV/CCV not performed at specified frequency	J	UJ
ICV/CCV %R < 75%	J-	UJ
ICV/CCV %R 75 - 89%	J	UJ
ICV/CCV %R 90 - 100%	No qualification	No qualification
ICV/CCV %R 111 - 125%	J	No qualification
ICV/CCV %R > 125%	J+	No qualification
ICV/CCV %RSD > 5%	J	UJ
Instrument blank analyzed prior to CCV	Use professional judgment	Use professional judgment

IV. Blanks

A. Review Items

Laboratory blanks reports (if available), preparation logs, calibration standard logs, instrument logs, and raw data in the data package, and sampling trip reports.

B. Objective

The objective is to determine the validity of the analytical results based on the blank responses by determining the existence and magnitude of contamination resulting from laboratory (or field) activities or baseline drift during analysis.

C. Action:

Refer to ICP-AES Table 4 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient blanks.

1. For ICB analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCB analyses that do not meet the technical criteria, apply the actions to all associated samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical sequence.
3. For Preparation Blank analyses that do not meet the technical criteria, apply the actions to all associated samples prepared in the same preparation batch. For LEBs that do not meet the technical criteria, apply the actions to all associated samples extracted in the same extraction batch.

4. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
5. If the absolute value of an ICB or a CCB result is ≥ QL, the analysis should have been terminated and the affected samples re-analyzed. If samples were not re-analyzed, qualify as described in Table 4 below.
6. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and ≥ QL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, qualify as described in Table 4 below.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute analysis and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. For blank results ≤ (-MDL) but > (-QL), the possibility of false negative exists.

NOTE: *Do not qualify blanks with blank results.* The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment or waste sample results reported in the Laboratory Results Reports will not be on the same basis (units, dilution) as the calibration blank data. It may be easier to work with the raw data and/or convert the ICB or CCB results to the same units as the soil/sediment or waste samples for comparison purposes.

ICP-AES Table 4. Blanks Actions

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	Not analyzed at the specified frequency	Non-detect	UJ
		Detect	J
ICB/CCB	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	No qualification
ICB/CCB	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	No qualification
ICB/CCB	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U

		≥ QL but < ICB/CCB Result	Report at ICB/CCB Result and qualify U
		≥ ICB/CCB Result but < 10x ICB/CCB Result	J+
		> 10x ICB/CCB Result	No qualification
ICB/CCB	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-
		≥ QL but < 10x QL	J-
		≥ 10x QL	No qualification
Preparation Blank/LEB	Not analyzed at specified frequency	Non-detect	UJ
		Detect	J
Preparation Blank/LEB/ Field Blank/ Rinse Blank	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	No qualification
Preparation Blank/LEB/ Field Blank/ Rinse Blank	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	No qualification
Preparation Blank/LEB/ Field Blank/ Rinse Blank	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but ≤ PB/LEB/FB/RB Result	Report at Blank Result and qualify U
		> PB/LEB/FB/RB Result but < 10x the PB/LEB/FB/RB Result	Report at Sample Result and qualify J+
		≥ 10x the Preparation Blank/LEB/Field Blank/Rinse Blank Result	No qualification
Preparation Blank/LEB/ Field Blank/ Rinse Blank	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-
		≥ QL but < 10x QL	J-
		≥ 10x QL	No qualification

V. Interference Check Sample

A. Review Items

Laboratory interference checks reports (if available), instrument printouts and raw data in the data package.

B. Objective

The objective is to determine the validity of the analytical results based on the instrument's ability to overcome interferences typical of those found in samples.

C. Action:

Refer to ICP-AES Table 5 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient ICSs.

1. For an ICS analysis that does not meet the technical criteria, apply the actions to all samples reported from the analytical sequence.

NOTE: The same result units should be used when comparing analyte results in samples to those in the ICS. Unit conversion may be necessary when soil/sediment/waste or wipe samples are evaluated.

Apply action only if the concentration of interferents Aluminum (Al), Calcium (Ca), Iron (Fe), and Magnesium (Mg) in the sample are found to be greater than their respective concentrations in the ICS.

2. In general, ICP-AES sample data can be accepted if the concentrations of Aluminum (Al), Calcium (Ca), Iron (Fe), and Magnesium (Mg) in the sample are found to be less than or equal to their respective concentrations in the ICS. If these elements are present at concentrations greater than the level in the ICS, or other elements are present in the sample at > 10 mg/L, investigate the possibility of other interference effects as given in the ICP-AES method or as indicated by the laboratory's interelement correction factors for that particular instrument. The analyte concentration equivalents presented in the method should be considered only as estimated values since the exact value of any analytical system is instrument-specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is > 2x the QL and > 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).
3. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be complex. Use professional judgment to determine the need for the associated sample data to be qualified. Obtain additional information from the laboratory, if necessary.

ICP-AES Table 5. Interference Check Actions

Criteria	Action	
	Detect	Non-detect
ICS not analyzed	R	R
ICS not analyzed in specified sequence	J	UJ
ICSAB %R < 50%	J-	R
ICS %R 50 - 84% [or ICS found value is < (true value - QL), whichever is lower]	J-	UJ
ICS %R 85 - 115%	No qualification	No qualification
ICS %R 116 - 150% [or ICS true value is > (true value + QL), whichever is greater]	J+	No qualification
ICS %R > 150%	J+	No qualification
ICSA results ≥ DLs or MDLs, but not present in ICS (potential false positives)	J+	No qualification
Negative ICSA results, but not present in ICS (potential false negatives)	J- for results < 10x (|negative sample result|)	UJ

VI. <u>Laboratory Control Sample</u>

A. Review Items

Laboratory LCS reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

The objective is to determine the validity of the analytical results based on the recovery of the digested Laboratory Control Sample (LCS).

C. Action

Refer to ICP-AES Table 6 for the evaluation criteria and corresponding actions for detected and non- detected target analyte results in the samples associated with deficient LCSs. For an LCS analysis that does not meet the technical criteria, apply the actions to all samples in the same preparation batch.

Matrix spike data can be reviewed to determine batch quality if an LCS was not prepared and analyzed with the samples.

ICP-AES Table 6. LCS Actions

Criteria	Action	
	Detect	Non-detect
LCS not prepared with sample	J	UJ
LCS not prepared at specified concentration	J	UJ
Aqueous/water and soil/sediment/waste %R < 40% (< 20% Ag, Sb)	J-	R
Aqueous/water and soil/sediment/waste %R 40 - 69% (20 - 49% Ag, Sb)	J-	UJ
Aqueous/water and soil/sediment/waste %R 70 - 130% (50 - 150% Ag, Sb)	No qualification	No qualification
Aqueous/water and soil/sediment/waste %R 131 - 150% (151 - 170% Ag, Sb)	J+	No qualification
Aqueous/water and soil/sediment/waste %R > 150% (170% Ag, Sb)	R	No qualification
Wipe %R < 40% (< 20% Ag, Sb)	J-	R
Wipe %R 40 - 69% (20 - 49% Ag, Sb)	J-	UJ
Wipe %R 70 - 130% (50 - 150% Ag, Sb)	No qualification	No qualification
Wipe %R > 130% (> 150% Ag, Sb)	J+	No qualification

VII. Laboratory Duplicate Sample Analysis

A. Review Items

Data Package Cover Page, laboratory duplicate reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

The objective of the laboratory duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis.

C. Action:

Refer to ICP-AES Table 7 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient duplicates.

1. For a laboratory duplicate sample analysis that does not meet the technical criteria, apply the actions to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making 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, E_h, 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]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package are similar to the duplicate sample, and that only these samples should be qualified; or 2) no samples are sufficiently similar to the sample used for the laboratory duplicate analysis, and thus only the field sample used to prepare the duplicate sample should be qualified.
2. For high RPDs (i.e., > 100%), use professional judgment to qualify the data as this may be indicative of a sampling problem.

NOTE: The laboratory duplicate analysis is not required for wipe nor air filter samples.

ICP-AES Table 7. Laboratory Duplicate Sample Actions

Criteria	**Action**	
	Detect	**Non-detect**
Laboratory duplicate analysis not performed at the specified frequency	J	UJ
Aqueous: Both original sample and duplicate sample results are ≥ 5x QL and 20% < RPD ≤ 100%	J	UJ
Soil/Sediment: Both original sample and duplicate sample results are ≥ 5x QL and 35% < RPD ≤ 100%	J	UJ
Aqueous: Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification
Soil/Sediment: Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 35%	No qualification	No qualification
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 100%	R	NA

Original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL	J	UJ
Original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

VIII. Spike Sample Analysis

A. Review Items

Data Package Cover Page, laboratory matrix spike reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

The objective of the spiked sample analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Action:

Refer to ICP-AES Table 8 below for the evaluation criteria and corresponding actions for detected and non-detected target and spike analyte results in the samples associated with deficient matrix spikes.

1. For a matrix spike sample analysis that does not meet the technical criteria, apply the actions to all samples of the same matrix, if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making 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, E_h, 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]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package are similar to the Matrix Spike sample, and that only these samples should be qualified; or 2) no samples are sufficiently similar to the sample used for the matrix spike analysis, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.

NOTE: Matrix spike analysis is not required for SDG that contains only field blank samples.

Qualifiers based on matrix spike sample analysis results are to be applied to original samples only.

Matrix spike analysis is not required for Calcium (Ca), Magnesium (Mg), Potassium (K), and Sodium (Na) for both matrices; Aluminum (Al) and Iron (Fe) for soil only.

Disregard the out-of-control spike recoveries for analytes whose unspiked concentrations are ≥ 4x the spike added.

ICP-AES Table 8. Spike Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike analysis not performed at the specified frequency (qualify all samples associated with the matrix spike)	J	UJ
Matrix Spike not prepared from field sample (qualify all samples associated with the matrix spike)	J	UJ
Matrix Spike %R < 30% Post-digestion spike %R < 75%	J-	R
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R 30-74% Post-digestion spike %R < 75%	J-	UJ
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R > 125% Post-digestion spike %R > 125%	J+	No qualification
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	J	No qualification
Matrix Spike %R < 30% No post-digestion spike performed [not required for Silver (Ag) and Antimony (Sb)]	J-	R
Matrix Spike %R 30 - 74% No post-digestion spike performed [not required for Silver (Ag) and Antimony (Sb)]	J-	UJ
Matrix Spike %R 75 - 125% No post-digestion spike is required	No qualification	No qualification
Matrix Spike %R > 125% No post-digestion spike performed [not required for Silver (Ag) and Antimony (Sb)]	J+	No qualification

IX. Serial Dilution

A. Review Items

Laboratory serial dilution reports (if available), instrument printouts, and raw data in the data package.

B. Objective

The objective of the serial dilution analysis is to determine if significant physical or chemical interferences exist due to sample matrix.

C. Action:

Refer to ICP-AES Table 9 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient serial dilution analyses.

1. For a serial dilution sample analysis that does not meet the technical criteria, apply the actions to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making 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, E_h, 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]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package are similar to the serial dilution sample, and that only these samples should be qualified; or 2) 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.

NOTE: Serial dilution analysis is not required for SDG that contains only field blank samples.

ICP-AES Table 9. Serial Dilution Actions

Criteria	Action	
	Detect	**Non-detect**
Serial Dilution analysis not performed at the specified frequency	J	UJ
Aqueous: Sample concentration > 50x MDL, serial dilution sample concentration ≥ QL, and 10% ≤ %D < 100%	J	NA
Aqueous: Sample concentration > 50x MDL, serial dilution sample concentration ≥ QL, and %D ≥ 100%	R	NA
Soil/Sediments: Sample concentration > 50x MDL, serial dilution sample concentration ≥ QL, and 15% ≤ %D < 100%	J	NA

Soil/Sediment: Sample concentration > 50x MDL, serial dilution sample concentration ≥ QL, and %D ≥ 100%	R	NA
Aqueous: Sample concentration > 50x MDL and serial dilution sample concentration ≥ QL, and %D < 10%	No qualification	No qualification
Soil/Sediment: Sample concentration > 50x QL and serial dilution sample concentration ≥ QL, and %D < 15%	No qualification	No qualification
Sample concentration > 5x QL and serial dilution sample concentration < QL	No qualification	No qualification
Interferences present	Use professional judgment	Use professional judgment

X. Field Duplicates

A. Review Items

Laboratory Results Reports, sampling documentation (e.g., COC Records), instrument printouts, and other raw data from QA/QC samples in data package.

B. Objective

The objective is to use results from the analysis of field and project QA/QC samples such as field blanks and field duplicates to determine the validity of the analytical results.

C. Action:

Refer to ICP-AES Table 10 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient field duplicates.

1. If a field duplicate samples pair was collected and analyzed, calculate, and report the RPD when the sample and its field duplicate values are both ≥ 5x QL. Calculate and report the absolute difference when at least one value (sample or its duplicate) < 5x QL.
2. Any action should be in accordance with the project specifications and the criteria for acceptable field duplicate sample results.
3. For field duplicate sample analysis that does not meet the technical criteria, apply the actions to the field sample and the field sample duplicate only. Exercise professional judgment in determining sample similarity when making 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]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. Two possible determinations are: 1) all of the

samples are considered sufficiently similar, and all samples should be qualified; or 2) only some of the samples in the data package are similar to the field duplicate sample, and that only these samples should be qualified.

4. In general, for QA/QC performance not within QAPP specification, qualify detects as estimated (J) and non-detects as estimated (UJ).

NOTE: Do not calculate RPD when both values are non-detects.

ICP-AES Table 10. Field Duplicate Actions

Criteria	Action	
	Detect	**Non-detect**
Aqueous: Both original sample and its field duplicate sample results are ≥ 5x QL and 20% < RPD < 100%	J	NA
Soil/Sediment: Both original sample and its field duplicate sample results are ≥ 5x QL and 50% < RPD < 100%	J	NA
Both original sample and its field duplicate sample results are ≥ 5x QL and RPD ≤ 20% (*Aqueous*) / RPD ≤ 50% (*Soil/Sediment*)	No qualification	No qualification
Aqueous: Original sample and/or its field duplicate sample result < 5x QL (including non-detects) and *absolute difference* between original sample and duplicate > QL	J	UJ
Soil/Sediment: Original sample and/or its field duplicate sample result < 5x QL (including non-detects) and *absolute difference* between original sample and duplicate > 2x QL	J	UJ
Original sample and its field duplicate ≥ 5x QL and RPD ≥ 100%	Use professional judgment to qualify other than R	NA

XI. Target Analyte Quantitation

A. Review Items

Laboratory result reports, sample preparation sheet, data package narrative, instrument printouts and raw data.

B. Objective

The objective is to ensure that the reported results and quantitation limits for target analytes reported by the laboratory are accurate and sufficient to meet requirements.

C. Action:

Refer to ICP-AES Table 11 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples with deficient quantitation limits. Apply the actions to the affected analytes for each sample that does not meet the quantitation criteria.

1. If sample results are < QLs and ≥ MDLs or limits in the QAPP, qualify as estimated (J).
2. If any sample result was greater than the linear range for ICP-AES and the sample was not diluted to obtain the result reported on Form I, qualify the affected results as estimated (J).
3. If the percent solids of sediment for a sample are < 50% but ≥ 30%, qualify the affected results ≥ MDL as estimated (J), and the non-detects as estimated (UJ).
4. If the sample's percent solids of sediment are < 30%, check if the sample was prepared at greater mass to maintain the QLs. Use professional judgment when this was not completed.

ICP-AES Table 11. Target Analyte Quantitation Percent Solids of Sediment Actions

Criteria	Action	
	Detect	Non-detect
Sample result < QLs and ≥ MDLs or limits in the QAPP	J	NA
Sample result > the linear range for ICP-AES and the sample was not diluted to obtain the result reported on Form I, qualify the affected results as estimated, J	J	NA
Percent solids of sediment sample < 50% but ≥ 30%	J	UJ
Percent solids of sediment sample < 30%, and was not prepared at greater mass to maintain QLs	Use professional judgment to qualify J or R	Use professional judgment to qualify UJ or R

Appendix B

Data Assessment Report Template

	UNITED STATES ENVIRONMENTAL PROTECTION AGENCY **REGION 2** **LSASD/HWSB/HWSS** **2890 Woodbridge Avenue, Edison, NJ 08837**

EXECUTIVE NARRATIVE

Case No.:
Site:
Number of Samples:
Analysis:

SDG No.:
Laboratory:
Sampling dates:
Validation SOP:

QAPP:
Contractor:
Reference: DCN Number

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

Major Findings:

Minor Findings:

COMMENTS:

Reviewer Name(s):

Approver's Signature:

Name: **Date:**

Affiliation: USEPA/R2/LSASD/HWSB/HWSS

Appendix C

Definitions/Glossary of Terms

Definitions/Glossary of Terms

Action Limit – A result for a Performance Evaluation (PE) sample that is outside the 99% (±3σ) control limits. The laboratory may be required to apply and document corrective actions to bring the analytical results back into control.

Analyte – The element or ion an analysis seeks to determine, the element of interest.

Analytical Services Branch (ASB) – Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).

Analytical Sample – Any prepared field sample or extract thereof that is introduced into an instrument for the purpose of measuring any target analyte. This definition excludes any instrument quality control samples (e.g., standards associated with initial calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), Continuing Calibration Blank (CCB), and tune verifications). The following are also defined as analytical samples: diluted samples; matrix spike and matrix spike duplicate samples; duplicate samples; serial dilution samples, post-digestion/post-distillation spike samples; Laboratory Control Samples (LCSs); Performance Evaluation (PE) samples; Preparation/Method Blanks; Field Blanks (FBs); and Leachate Extraction Blanks (LEBs).

Associated Samples – Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples analyzed under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.

Blank – An analytical sample that has negligible or unmeasurable amounts of a substance of interest. The blank is designed to assess specific sources of contamination. Types of blanks may include calibration blanks, preparation blanks, and field blanks. See the individual definitions for types of blanks.

Calibration – A set of operations that establish under specific conditions, the relationship between values indicated by a measuring instrument and the corresponding known values. The calibration standards should be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

Calibration Blank – A blank solution containing all reagents and in the same concentration as those used in the analytical sample preparation. This blank is digested/distilled for mercury and cyanide. Calibration blanks are used to verify that the instrument baseline is stable, and the instrument is free of contamination.

Calibration Curve – A plot of instrument response versus concentration of standards.

Calibration Standards – A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the calibration curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

Case – A finite, usually predetermined number of samples collected over a given period from a particular project site. A case numbers is assigned by the Sample Management Office (SMO) and consists of one or more Sample Delivery Groups (SDGs).

Chain of Custody (COC) Record – A sample identification form completed by the sampler, which accompanies the sample during shipment to the laboratory and is used to document sample identity, sample chain of custody, sample condition, and sample receipt by the laboratory.

Contamination – A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may result from other samples, sampling equipment, or from introduction while in transit, from laboratory reagents, from the laboratory environment, or from analytical instruments.

Continuing Calibration Blank (CCB) – A reagent water sample that is run at specified interval and designed to detect any carryover contamination.

Contract Compliance Screening (CCS) – A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under EPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.

Continuing Calibration Verification (CCV) – A single parameter or multi-parameter standard solution prepared from the same source as the initial calibration standards by the analyst and used to periodically verify the stability of the instrument calibration during analysis of samples. The CCV can be one of the calibration standards with the concentration near the middle of the calibration range. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples.

Control Limits – A range within which specified measurement results should fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

Contract Laboratory Program (CLP) – Supports the EPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.

Contract Required Quantitation Limit (CRQL) – Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

Data Package Narrative – Portion of the data package which includes laboratory information, sample identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Detection Limit (DL) - A generic term for the minimum measured concentration of a substance that can be reported with a specified confidence that the measured concentration is distinguishable from blank results. Includes Method Detection Limit (MDL), Limit of Detection (LOD), and other means of establishing this limit.

Duplicate – A second aliquot of a sample that is treated the same as the original sample in order to evaluate the precision.

Field Blank (FB) –A blank used to provide information about contaminants that may be introduced during sample collection, shipment, storage, and/or preparation and analysis in the laboratory. Examples of field blanks include trip blanks, rinse blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

Field Duplicate (FD) – A duplicate sample generated in the field, not in the laboratory.

Field Quality Control (FQC) – Any QC samples submitted from the field to the laboratory. Examples include, but are not limited to, field blanks, and field duplicates.

Field Sample – A portion of material received from the field to be analyzed for analytes of interest.

Holding Time – The maximum amount of time samples may be held before they are processed.

Holding Time (Contractual) – The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

Holding Time (Technical) – The maximum amount of time that samples may be held from the collection date until analysis.

Initial Calibration – Analysis of analytical standards at a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Initial Calibration Blank (ICB) – The first blank standard analysis to confirm the calibration curve.

Initial Calibration Verification (ICV) – The analysis of solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV solution(s) should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources.

Interference Check Sample (ICS) – A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

Internal Standard – A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

Laboratory Control Sample (LCS) – A reference matrix spiked with target analytes at a known concentration. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

Leachate Extraction Blank (LEB) – A blank carried through the entire Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction with the resulting leachate extracted, digested, or distilled by an appropriate aqueous method from the analytical method.

Matrix – The predominant material of which the sample to be analyzed is composed. For the purposes ofthis document, the matrices are aqueous/water, soil/sediment, and wipe. Matrix is <u>not</u> synonymous with phase (liquid or solid).

Matrix Spike – Aliquot of a sample (aqueous/water or soil/sediment) fortified (spiked) with known quantities of specific analytes and subjected to the entire analytical procedure to estimate recovery.

Method Detection Limit (MDL) – The minimum measured concentration of a substance that can be reported with 99% confidence such that the measured concentration is distinguishable from method blank results. Additional information about the procedure is provided in Title 40 of the Code of Federal Regulations (CFR), Chapter 1, Subchapter D, part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit, Revision 2.

Narrative (SDG Narrative) – Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Office of Solid Waste and Emergency Response (OSWER) – The EPA office that provides policy, guidance, and direction for the EPA's solid waste and emergency response programs, including Superfund.

Percent Difference (%D) – The relative difference between two values (e.g., a measured and expected value) expressed as a percentage of one of the values (e.g., expected value).

Percent Solids (%Solids) – The proportion of solid in a soil/sediment sample determined by drying an aliquot of the sample.

Performance Evaluation (PE) Sample – A sample prepared by a third party at known concentrations that are unknown to the analytical laboratory and is provided to test whether the laboratory can produce analytical results within specified performance limits.

Post-Digestion Spike/Post-Distillation Spike – The addition of a known amount of standard after digestion or distillation (also identified as an analytical spike).

Preparation Blank (PB) – An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Preparation Log – A record of sample preparation (e.g., digestion, extraction, distillation) at the laboratory.

Quality Assurance Project Plan (QAPP) – A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization, or laboratory for ensuring quality in its products and utility to its users.

Quantitation Limit (QL) – The minimum level of acceptable quantitation that is supported by the analysis of standards.

Raw Data – The originally recorded and unprocessed measurements from any measuring device such as analytical instruments, balances, pipettes, thermometers, etc. Reported data are processed raw measurement values that may have been reformatted from the original measurement to meet specific reporting requirements such as significant figures and decimal precision.

Relative Percent Difference (RPD) – The absolute of the relative difference between two values normalized to the mean of the two values expressed as a percentage.

Regional Sample Control Center Coordinator (RSCC) – In EPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.

Relative Standard Deviation (RSD) – As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.

Sample – A single, discrete portion of material to be analyzed that is contained in single or multiple containers and identified by a unique Sample Number.

Sample Delivery Group (SDG) – A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
b. Each 7-calendar day period (3-calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG. Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.

Sample Identifier – A unique identification number that appears on the Chain of Custody (COC) Records or sampling forms which documents information for a sample.

Sample Management Office (SMO) – A contractor-operated facility operated under the SMO contract, awarded, and administered by the EPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

Sampling and Analysis Plan (SAP) – A document which specifies the procedural and analytical requirements for one-time, or time-limited, projects involving the collection of water, soil, sediment, or other samples taken to characterize areas of potential environmental contamination.

Serial Dilution (SD) – The dilution of a sample by a factor of five. When corrected by the Dilution Factor (DF), the diluted sample should agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents [Inductively Coupled Plasma (ICP) only].

Soil – Synonymous with soil/sediment and sediment as used herein.

Statement of Work (SOW) – A document which specifies how laboratories analyze samples under a contract, such as the Contract Laboratory Program (CLP) analytical program.

Warning Limit – A result for a Performance Evaluation (PE) sample that is outside the 95% ($\pm 2\sigma$) control limits. The laboratory should apply and document corrective actions to bring the analytical results back into control.

Appendix D

SOP Change Request Form (CRF)

REQUEST FOR SOP CHANGE

Requestor Name:		Date of Initiation:	

Dept.:		SOP #:		Revision #:	Date:

SOP Title:	

Please Check One	MINOR REVISION ……	MAJOR REVISION …….

CHANGE(S) (Use attachment if necessary):
CHANGE FROM: CHANGE TO:
REASON(S) FOR CHANGE(S):

APPROVAL	**NAME**:	**Signature/Date**
EPA Branch Chief / Section Chief/Team Leader		
EPA TOCOR		
REQUESTOR		
Effective Date		