USEPA REGION 9

STANDARD OPERATING PROCEDURE 906

GUIDELINES FOR DATA REVIEW OF CONTRACT LABORATORY PROGRAM ANALYTICAL SERVICES INORGANIC DATA PACKAGES

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GUIDELINES FOR DATA REVIEW OF CONTRACT LABORATORY PROGRAM ANALYTICAL SERVICES INORGANIC DATA PACKAGES

1 INTRODUCTION

Data review provides information about limitations of analytical data based on specific quality criteria, data quality objectives, and data quality indicators. These guidelines for the review of data packages for ICP-AES, ICP-MS, CVAA, and spectrophotometric analysis of metals and cyanide in soil and water samples are based on the specific technical requirements listed in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October 204, as modified in Appendix A1 according to procedures of the EPA Region 9 Quality Assurance Program;
- USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, Multimedia, Multi-Concentration, ILM05.3, March 2004;
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, February 1994, as modified in Appendix A1 according to procedures of the EPA Region 9 Quality Assurance Program;
- USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, Multimedia, Multi-Concentration, ILM04.1, January 2000; and
- USEPA Region 9 Memorandum "Documentation of Data Validation Requirements in QAPPs, FSPs, and SAPs" dated January 14, 2000.

2 PURPOSE

This SOP provides guidance for reviewing CLP metals and cyanide inorganic data packages and preparing data validation reports according to Regional Tier 3 validation. A uniform procedure ensures that data reviews are technically accurate, complete, and consistent. The technical requirements in the documents cited above are default criteria when other contract (or project-specific) requirements are not specified.

Note: Determining contract compliance is not the intended objective of this document.

3 ACRONYMS

%D	-	Percent difference
%R	-	Percent recovery
%RSD	-	Percent relative standard deviation
BG	-	Background sample
CCV	-	Continuing calibration verification
CLP	-	Contract Laboratory Program
CLPAS	-	Contract Laboratory Program Analytical Services
CLP PO	-	Contract Laboratory Program Project Officer
CN	-	Cyanide
CRA	-	CRDL standard (SOW ILM04.X mercury only)
CRDL	-	Contract required detection limit (SOW ILM04.X only)
CRI	-	CRQL standard
CRQL	-	Contract required quantitation limit (SOW ILM05.X)
CVAA	-	Cold vapor atomic absorption spectrophotometry
DQI	-	Data quality indicators
EB	-	Equipment blank
EPA	-	Environmental Protection Agency
ESAT	-	Environmental Services Assistance Team
FB	-	Field blank
FG	-	Functional Guidelines for Organic Data Review
ICP-AES	-	Inductively coupled plasma-atomic emission spectrophotometry
ICP-MS	-	Inductively coupled plasma-mass spectrometry
ICS	-	Interference check sample
ICSA	-	ICS solution A
ICSAB	-	ICS solution AB
ICV	-	Initial calibration verification
IDL	-	Instrument detection limit (SOW ILM04.X only)
IS	-	Internal standard
LCS	-	Laboratory control sample
MDL	-	Method detection limit
MS	-	Matrix spike
PB	-	Preparation blank
PE	-	Performance evaluation
QA	-	Quality assurance
QC	-	Quality control
QAPP	-	Quality Assurance Project Plan
r	-	Correlation coefficient
RPD	-	Relative percent difference
SAP	-	Sampling Analysis Plan
SDG	-	Sample delivery group
SMC	-	System monitoring compound
SOP	-	Standard operating procedure
SOW	-	Statement of Work

TAL	-	Target analyte list
TAL	-	Target analyte list
TDF	-	Technical direction form
TR	-	Traffic report
TRL	-	Telephone record log
USEPA	-	United States Environmental Protection Agency

4 INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY DATA REVIEW

This section contains data validation requirements and procedures for metals analysis by inductively coupled plasma – atomic emission spectroscopy (ICP-AES). Validation criteria and appropriate actions for each SOW are provided in Appendices B1 and B2. Worksheets for each SOW are provided in Appendices C1 and C2.

4.1 PRESERVATION AND HOLDING TIMES

The objective of this section is to verify the validity of the results based on the preservation and holding time of the samples from the time of collection to the time of analysis. Holding time and preservation criteria and actions are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.1;
- Appendix B2 SOW ILM05.X Table 5.1.

4.1.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form XII-IN, Form XIII-IN, EPA Traffic Report and/or Chain of Custody (COC) record form(s), Form DC-1, Field QA/QC Summary Form, sample digestion log(s), raw data, and sample delivery group (SDG) Narrative.

- *SOW ILM05.X*: Form IA-IN, Form IB-IN, Form XII-IV, Form XIII-IN, EPA Traffic Report/Chain of Custody (TR/COC) record form(s), Form DC-1, Field QA/QC Summary Form, sample digestion log(s), raw data, and SDG Narrative.

4.1.2 Preservation Criteria:

4.1.2.1 Aqueous Samples

- pH: Examine Form DC-1 and the sample digestion log(s) to verify water samples were preserved to a pH < 2 with nitric acid.

- Temperature: Examine DC-1 and internal COC documents to confirm the cooler temperature upon receipt at the laboratory is 4±2°C.

4.1.2.2 Soil Samples

- Temperature: Examine DC-1 and internal COC documents to confirm the cooler temperature upon receipt at the laboratory is 4±2°C.

4.1.3 Holding Time Criteria

4.1.3.1 Verify and document in worksheets all technical holding times by comparing the dates of collection for all samples as reported on the EPA Traffic Reports/Chain of Custody (TR/COC) record form(s) to the dates of analysis from the instrument run logs and the raw data.

4.1.3.2 Technical holding time: If the sample holding time for water or soil is less than or equal to 180 days, no action is required.

4.1.3.3 If the sample holding time for water or soil is greater than 360 days, qualify results as rejected and comment in CLP PO ACTION section. Note that the Region 9 advisory limit for the rejection of data is two times the technical holding time for analyses that grossly exceed the holding time and are or are not properly preserved. (Region 9 Modification, see Appendix A.1.)

4.2 INSTRUMENT CALIBRATION

The objective of this section is to verify that the method requirements for satisfactory instrument calibration are followed and that the instrument is capable of producing acceptable quantitative data. Initial calibration verification (ICV) demonstrates that the instrument performance is acceptable at the beginning of the analytical run. Continuing calibration verification (CCV) demonstrates that the initial calibration continues to be valid by checking instrument performance at regular intervals. Initial calibration, initial calibration verification, continuing calibration verification and contract required detection limit (CRDL)/contract required quantitation limit (CRQL) verification (CRI) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.2;
- Appendix B2 SOW ILM05.X Table 5.2.

4.2.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form IIA-IN, Form IIB-IN, Form XI-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form IIA-IN, Form IIB-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

4.2.2 Initial Instrument Calibration

4.2.2.1 Verify that the instrument is successfully calibrated daily or once every 24 hours and each time the instrument is set up. Verify that a blank and at least one standard have been used to establish the standard curve and that sample results are within the linear working range of the instrument. Verify that the average of two replicate exposures is used for all results. Inspect the raw data to ensure that the calibration date and time are included. Analytical wavelengths used for calibration must be used throughout the analytical sequence.

4.2.3 Initial and Continuing Calibration Verification

4.2.3.1 If an ICV or CCV %R is outside of the control limits, determine if the laboratory stopped the analysis, corrected the problem, recalibrated, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION.

4.2.3.2 From the raw data, recalculate and document in worksheets (Appendix C) a minimum of 10% of the calibration verification percent recoveries (%Rs) using the equations below:

ICV %R or CCV %R = (*Found* value / *True* value) x 100

Found value = concentration of analyte measured in ICV or CCV standard.

True value = certified concentration of analyte in ICV or CCV standard.

4.2.3.3 Verify that an ICV standard, prepared from a USEPA or certified stock solution but from a different stock source or lot than used for calibration, is analyzed at the wavelengths used for sample analysis and reported immediately after ICP system calibration, but before the analysis of any analytical samples. Examine all ICV %Rs and compare to criteria in Appendix B.

4.2.3.4 Verify that CCV standards are analyzed and reported at the beginning and end of the analytical run, after an ICV, and at a frequency of 10% of samples analyzed or every two hours during an analytical run, whichever is more frequent. Verify that the CCV standards are prepared from a different source or lot than used for the ICV and are at or near the mid-range of the calibration curve.

4.2.4 Contract Required Detection Limit/Contract Required Quantitation Limit Verification

4.2.4.1 Verify that a CRDL/CRQL verification standard (CRI) was analyzed to demonstrate that the established calibration is linear and valid near the CRDL/CRQL.

4.2.4.2 Note: CRI is not required to contain Al, Ba, Ca, Fe, Mg, K, and Na.

4.2.4.3 Recalculate and document in worksheets a minimum of 10% of the CRI verification %Rs using the equation below:

$$\text{CRI \%R} = (\textit{Found}\text{ value} / \textit{True}\text{ value}) \times 100$$

Found value = concentration of analyte measured in CRI standard.

True value = certified concentration of analyte in CRI standard.

4.2.4.4 There are no EPA-established limits for CRI in SOW ILM04.X. Use the Region 9 advisory limits of 65-135%R. (Region 9 Modification, see Appendix A.2) Compare to criteria in Appendix B.

- Verify that the CRI is analyzed after the ICV and at the end of the run or a minimum of 2 times per 8 hour shift, whichever is more frequent. If the frequency is not met, note in CLP PO ATTENTION but do not qualify any results.

- The CRI concentration should be two times the CRDL specified for the target analyte, or two times the instrument detection limit (IDL), whichever is greater.

4.2.4.5 There are EPA established limits for CRI in SOW ILM05.X. Compare CRI results to criteria and actions in Appendix B.

- Verify that a CRI is analyzed after the ICV, but before the Interference Check Sample (ICS), every 20 analytical samples, and at the end of the run.

- If calibration criteria are grossly exceeded, include a comment in the CLP PO ACTION section of the report.

4.3 BLANKS

The objective of this section is to assess the results of blank analyses to determine the existence and magnitude of contamination from laboratory or field activities. The criteria for evaluation of blanks apply to any blank associated with the samples. Initial calibration blank (ICB), continuing calibration blank (CCB), preparation blank (PB), field blank (FB), and equipment blank (EB) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.3;
- Appendix B2 SOW ILM05.X Table 5.3.

4.3.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.*X: Form I-IN, Form III-IN, Form XIII-IN, Form XIV-IN, Field QA/QC Summary Form, sample preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.*X: Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, Field QA/QC Summary Form, sample preparation logs, instrument logs, instrument printouts, and raw data.

4.3.2 No contaminants should be found in any blank. If problems exist with any blank, evaluate all sample data associated with the blank to determine if the problem is system wide or an isolated occurrence.

4.3.3 Calibration Blanks

4.3.3.1 Verify that an ICB is analyzed after the ICV. Verify that if the absolute value of an analyte in an ICB is greater than the CRQL, the analysis is terminated, the problem corrected, and the associated samples reanalyzed. If the ICB does not meet criteria, apply action to all samples in the analytical run. If the ICB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

4.3.3.2 Verify that a CCB is analyzed after every CCV. If the absolute value of an analyte in a CCB is greater than the CRQL, verify that the analysis is terminated, the problem corrected, and the associated samples reanalyzed. Verify that the CCB is analyzed at a frequency of every ten samples or every two hours during the run, whichever is more frequent. If CCBs do not meet criteria, apply actions to all samples associated with the CCBs out of criteria. If the CCB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

4.3.4 Preparation Blank

4.3.4.1 Verify that a PB was digested and analyzed for each matrix per SDG or batch of samples digested, whichever is more frequent. If the absolute value of any analyte in the PB is less than or equal to the CRQL, no action is required. If the PB does not meet criteria, apply action to all samples prepared in the same preparation batch. If the PB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

4.3.4.2 Verify that analyte concentrations in the PB are less than the CRQL. Confirm that all samples associated with a contaminated PB contain the contaminated analyte at a concentration greater than 10 times the PB concentration. Verify that the all samples were re-digested and reanalyzed (except for identified field blanks). Note that the laboratory is not to correct any sample concentration by subtracting the blank value. Include a comment in the CLP PO ACTION section of the report if the laboratory failed to re-digest and reanalyze the affected samples.

- *Region 9 Modification*: For PB contamination, qualify associated sample results greater than the MDL but less than 5 times the amount in the blank as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.3).

4.3.4.3 If the analyte concentration in the PB is less than the negative CRQL value, all samples associated with the contaminated PB with contaminating analyte concentrations less than 10 times the CRQL should have been re-digested and reanalyzed. Include a comment in the CLP PO ACTION section of the report if the laboratory failed to re-digest and reanalyze the affected samples.

4.3.5 Field Blanks

4.3.5.1 Verify all field QC blank samples listed on the Field QA/QC Summary Form and identified on the spreadsheet. Check the Field QA/QC Summary Form to verify that all field QC samples have been properly identified in the report, and on the spreadsheet. Use professional judgment to determine whether to comment or estimate the associated sample data.

4.3.6 If the absolute value of the concentration of an analyte in a blank is greater than the CRQL, apply the following guidelines.

4.3.6.1 For PB contamination, qualify associated sample results greater than the MDL but less than 5 times the amount in the blank as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.3).

4.3.6.2 For ICB or CCB contamination, determine all sample numbers whose results were used from the analytical run between the last acceptable ICB or CCB and the next acceptable CCB. Associated sample results greater than the MDL but less than 5 times the amount in the blank are qualified as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.3).

4.3.6.3 For any laboratory blank (PB, ICB, CCB) with a negative result whose absolute value is greater than the CRQL, use professional judgment to determine the effect on data quality, i.e., low bias, and include a comment under CLP PO ACTION.

4.3.6.4 For FB or EB contamination, identify associated samples based on the same date of collection as the field QC. Associated sample results greater than the MDL but less than 5 times the amount in the blank are qualified as non-detected and estimated (UJ). Include a comment under SAMPLING ISSUES (Region 9 Modification, see Appendix A).

4.3.7 For sample results less than the CRQL and affected by blank contamination, apply the following guidelines.

4.3.7.1 For undiluted samples, increase associated sample results less than the CRQL to the CRQL and qualify as non-detected (UJ).

4.3.7.2 For diluted samples, increase associated sample results less than the CRQL to the product of the CRQL multiplied by the sample dilution factor (CRQL times df) and qualify as non-detected (UJ).

4.3.7.3 If an ICB or CCB analyte result is greater than the CRQL, and the analysis was not terminated and the affected samples were not reanalyzed, raise the CRQL of the affected samples to the concentration found in the ICB or CCB. Report non-detects and results greater than or equal to the MDL, but less than or equal to the CRQL as [CRQL]U. Include a comment in the CLP PO ACTION section of the report.

4.4 ICP-AES INTERFERENCE CHECK SAMPLE (ICSA AND ICSAB)

The objective of this section is to verify that the ICP interference check sample (ICS) demonstrates the ability of the analytical instrument to overcome typical interferences found in environmental samples. ICS criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.4;
- Appendix B2 SOW ILM05.X Table 5.4.

4.4.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form IV-IN, Forms XI-IN (Part A and B), Form XIV-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form IVA-IN, Form XA-IN, Form XB-IN, Form XIII-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

4.4.2 An ICS analysis consists of analyzing two solutions in sequence, solution A (interferents only) then solution AB (interferents and target analytes), for wavelengths used for each analyte reported by ICP-AES.

4.4.3 Recalculate and document in worksheets a minimum of 10% of the ICS %Rs using the equation below:

ICS %R = (ICSA/AB *Found* value / ICSA/AB *True* value) X 100

ICSA/AB *Found* Value = concentration of analyte measured in ICSA or AB standard.

ICSA/AB *True* Value = certified concentration of analyte in ICSA or AB standard.

4.4.4 In general, ICP-AES sample data can be accepted if the concentrations of Al, Ca, Fe, or Mg in the sample are found to be less than or equal to their respective concentrations in the ICS solutions. If the interferent concentration is greater

than the level in the ICS, or other elements present in the sample are found at concentrations greater than10 mg/L, estimate the concentration produced by the interfering element. If the estimate is two times CRQL and also greater than 10% of the reported concentration of the affected element, qualify affected sample results as estimated.

4.4.5 Note that professional judgment is required to determine if qualification is necessary, and it is recommended that an experienced reviewer be consulted before qualifying data until a feel for these criteria is gained. The following questions may be used to help determine if qualification is reasonable.

- How does the result for an analyte not truly in solution ICSA compare to the MDL and CRQL for that analyte?
- What concentrations of interferents are in the samples?
- What concentration of the analyte in question does the sample result exhibit, and could that quantity be altered substantially by the potential interference?
- Could there be false positives or negatives due to the potential interference?
- Is the analyte in question specifically affected by the interferent in question?

4.4.6 ICS: SOW ILM04.X

4.4.6.1 ICSAB percent recoveries must fall within the control limits of 80-120%. For analytes with ICSAB results outside of the control limits in samples with concentrations of Al, Ca, Fe, or Mg comparable to or greater than their respective levels in the ICSAB, qualify sample results according to the following guidelines.

4.4.6.2 Examine ICSA results to determine if false positives or negatives are being generated as a result of interference due to high quantities of the potential interferents Al, Ca, Fe, or Mg. Qualify sample results that fulfill all of the following criteria as estimated (J).

4.4.6.3 The absolute value of the concentration for an analyte is greater than the CRDL, but has a true value of zero.

4.4.6.4 The results for one or more interferents in a given sample are comparable to or greater than the quantities of interferents in ICSA.

4.4.6.5 The result for the analyte in that sample could be substantially affected by such potential interference. Typically, such results are near the CRDL.

4.4.6.6 The analyte in question has been shown to be affected by the interferent in question due to analysis at similar primary or secondary wavelengths. Refer to the ICP Interelement Correction Factors on Form 11 A and B submitted with the data package.

4.4.6.7 If the interferent concentration is greater than the level in the ICS, or other elements present in the sample are at concentrations greater than10 mg/L, estimate the concentration produced by the interfering element. If the estimate is greater than2x CRDL and also greater than 10% of the reported concentration of the affected element, qualify affected sample results as estimated (J).

4.4.7 ICS: SOW ILM05.X

4.4.7.1 Examine all ICP ICS results on Form 4A. The laboratory should have analyzed and reported ICS results for all analytes being reported from the analytical run and for all interferents (target and non target) for these reported analytes. Note that ICS solution A is analyzed before ICS solution AB.

4.4.7.2 ICSA and ICSAB solutions must be run at the beginning, the end, and after every 20 analytical samples of each analytical run. The ICS must be analyzed after the ICV/ICB, but before the CCV.

4.4.7.3 ICSA and ICSAB %Rs must fall within the control limits of two times (2 x) the CRQL, or 80-120% of the true value (whichever is greater) for the analytes and interferents in the solution. When an ICS %R is outside of the control limits, the laboratory is required to stop, correct the problem, recalibrate, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION.

4.5 SPIKE SAMPLE ANALYSIS

The objective of this section is to provide information about the effect of each sample matrix on the sample preparation procedures and the analytical method. Matrix spike (MS) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.5;
- Appendix B2 SOW ILM05.X Table 5.5.

4.5.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Forms V-IN (Parts A and B), Form XIII-IN, Form XIV- IN, Field QA/QC Summary Form, standard preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form I-IN (for QC sample), Form VA-IN, Form VB-IN, Form XII-IN, Form XIII-IN, Field QA/QC Summary Form, standard preparation logs, instrument logs, instrument printouts, and raw data.

4.5.2 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the MS %Rs using the equation below:

$$\text{Matrix spike \%R} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spiked sample result from the original sample.

SR = Sample result. Use SR = 0 when sample concentration is less than the MDL.

SA = Spike added.

4.5.3 Use the results of the sample designated as the "original sample" to perform re-calculations of the matrix spike. Do not use the average of the "original sample" and the laboratory duplicate sample results for the purpose of determining the matrix spike %R.

4.5.4 Do not use results from post-digestion spike analyses to qualify sample results. Comment about post-digestion spike results in the matrix spike section of the inorganic report.

Note: Post-digestion (analytical) spikes are <u>not</u> required for silver (Ag).

4.5.5 Do not evaluate matrix spike recovery in instances where a sample result exceeds four times the spiking concentration.

4.5.6 Verify that at least one matrix spike sample analysis has been performed on each group of samples of a similar matrix type and concentration, or for each SDG.

4.5.7 Check that the QC sample designated on the chain of custody is used for matrix spike analysis. Comment in "Additional Comments" if a different sample than the one designated on the COC is used

4.5.8 Verify that the spike was added before the digestion of the sample, and that the correct spike concentrations were used in spiking the matrix spike sample by examining the digestion logs and referring to the SOW.

4.5.9 Qualify samples designated as background samples for MS outliers since they reflect the matrix of the environmental samples.

4.5.10 Do not qualify field blank, equipment blank, or performance evaluation (PE) sample data due to MS outliers since these matrices are not representative of environmental samples. If an improper sample, i.e., field or equipment blank, was used for matrix spike analysis and there was no QC sample listed on the COC, comment in SAMPLING and include a comment in Section III, Validity and Comments, indicating that accuracy and precision parameters could not be evaluated due to improperly performed QC.

4.5.11 If inadequate QC has been performed (i.e., due to insufficient sample, etc.), use professional judgment to determine the effect on the data quality, and include a comment in CLP PO ATTENTION and in Section III, Validity and Comments.

4.5.12 If incorrect concentrations were used, use professional judgment to determine the effect on the data quality, and include a comment in CLP PO ATTENTION and in Section III, Validity and Comments.

4.5.13 If any analytes have not met matrix spike recovery criteria, check the post-digestion spike sample %Rs on Form 5B for those analytes (except Ag). Whether the results for these analytes do or do not achieve 75-125 %R, comment on the post-digestion spike recovery results in the matrix spike section of the inorganic template.

4.5.14 If incorrect concentrations were used, use professional judgment to determine the effect on the data quality, and include a comment in CLP PO ACTION section.

4.5.15 A post-digestion spike must be performed for any analyte that does not meet the specified MS %R criterion (except Ag). An aliquot of the remaining unspiked sample shall be spiked at two times (2x) the indigenous level or 2x the CRQL, whichever is greater. If a required post-digestion spike was not performed, comment in the CLP PO ACTION section of the report.

4.6 DUPLICATE SAMPLE ANALYSIS

The objective of this section is to verify that the laboratory demonstrated acceptable method precision at the time of sample analysis. Duplicate sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.6;
- Appendix B2 SOW ILM05.X Table 5.6.

4.6.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form VI-IN, Form XIII-IN, Form XIV-IN, QA/QC Summary Form, instrument logs, instrument printouts, and raw data.

  If inadequate QC has been performed, use professional judgment to determine the effect on the data quality, and include a comment for CLP PO ATTENTION and in Section III, Validity and Comments.

- *SOW ILM05.X*: Form I-IN, Form VI-IN, Form XII-IN, Form XIII-IN, QA/QC Summary Form, instrument logs, instrument printouts, and raw data.

  If inadequate QC has been performed, use professional judgment to determine the effect on the data quality, and include a comment in the CLP PO ACTION section of the report.

4.6.2 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the RPDs using the equation below:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

S = Sample result (original).

D = Duplicate result.

4.6.3 Do not report an RPD of 200 when one result is non-detected and the other result is detected greater than or equal to the CRQL. State in the duplicate sample comment that the analyte was reported at a given concentration in one duplicate analysis, but not detected in the other.

4.6.4 Verify that duplicate sample analyses are performed for Percent Solids.

4.6.5 Verify that at least one duplicate sample analysis has been performed on each group of samples of a similar matrix type and concentration, or for each SDG. Check to make sure that the QC sample designated on the chain of custody was used. Duplicate analysis does not need to be performed on samples labeled as rinsates in an SDG of soil samples.

4.6.6 Qualify Background samples for duplicate outliers since the background sample matrix reflects the matrix of the environmental samples.

4.6.7 Do not qualify results for field blank, equipment blank, or PE sample data for duplicate outliers since the matrix of the blanks is not similar to the environmental samples. If a field blank was used for duplicate sample analysis, note such use of a field blank in CLP PO ACTION and include a statement in Section III, Validity and Comments, indicating that this precision parameter could not be evaluated due to improperly performed QC.

4.7 LABORATORY CONTROL SAMPLE (LCS)

The objective of this section is to monitor the laboratory's overall performance at each step of the analysis, including sample preparation. Laboratory control sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.7;
- Appendix B2 SOW ILM05.X Table 5.7.

4.7.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form VII-IN, Form XIII-IN, Form XIV-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form VII-IN, Form XII-IN, Form XIII-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

4.7.2 Verify that one aqueous LCS was prepared and analyzed for every group of aqueous samples in an SDG, or each batch of aqueous samples digested, whichever is more frequent. The aqueous LCS must be prepared and analyzed using the identical preparation and analytical methods used for the water samples. The aqueous LCS solution should be obtained from the USEPA, if available. If the LCS is unavailable from the USEPA, the ICV solution may be used.

4.7.3 Aqueous LCS %Rs must be within the 80-120% control limits. If the %R are outside the control limits (except for Sb and Ag) the laboratory is required to stop, correct the problem, recalibrate, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION section of the report. Use professional judgment to assess the data.

Note: Analytes Sb and Ag do not have fixed LCS control limits. If Sb and Ag exceed the 80-120%R control limits, estimate the results in the associated samples. However, the laboratory is not required to repeat the analysis for Sb and Ag.

4.7.4 For soils sample analysis, verify that one solid LCS was prepared and analyzed for every group of soil/sediment samples in a SDG, or with each batch of soil samples digested, whichever is more frequent. The solid LCS must be prepared and analyzed using the identical preparation and analytical methods used for the soil/sediment samples. The solid LCS should be obtained from the USEPA, if available. If the LCS is unavailable from the USEPA, other USEPA QA samples or certified materials may be used.

4.7.5 Solid LCS results must be within the control limits reported on Form VII-IN. If a result is outside the control limit, the laboratory is required to stop, correct the problem, recalibrate, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION section of the report. Use professional judgment to assess the data.

Note: The percent solids determination is not required for the solid LCS.

4.7.6 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the %Rs using the equation below:

LCS %R = (*Found* value/ *True* value) X 100

Found value = concentration of analyte measured in LCS sample.

True value = certified concentration of analyte in LCS source.

4.7.7 Verify that at least one LCS has been analyzed for each group of samples of a similar matrix type (soil or water) and concentration, or for each SDG.

4.8 ICP SERIAL DILUTION ANALYSIS

The objective of this section is to verify, through a serial dilution of a sample, whether or not significant physical or chemical interferences exist due to sample matrix for samples analyzed by ICP. ICP serial dilution analysis criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.8;
- Appendix B2 SOW ILM05.X Table 5.8.

4.8.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form IX-IN, Form XIV-IN, Field QA/QC Summary Form, instrument printouts, and raw data.

- *SOW ILM05.X*: Form I-IN, Form VIII-IN, Form XIII-IN, Field QA/QC Summary Form, instrument printouts, and raw data.

4.8.2 Verify that an ICP serial dilution sample is prepared and analyzed for every group of samples with a similar matrix type (soil or water) or for each SDG, or with each batch of aqueous samples digested, whichever is more frequent.

4.8.3 If the analyte concentration in the original sample is greater than fifty times (50x) the method detection limit (MDL), the serial dilution results must be within a ten percent difference (%D) of the original sample results after correction for dilution. Serial dilution results for soil and water samples are reported in ug/L.

4.8.4 Examine the raw data for evidence of interference; i.e., for results of the diluted sample which are significantly higher or lower than the results of the original sample. Use professional judgment and document when qualifying sample results. Indicate in the report if there is a possible high or low bias in the results.

4.8.5 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the percent differences (%Ds) using the equation below:

$$\%D = \frac{I - S}{I} \times 100$$

I = Initial Sample Result.

S = Serial Dilution Result (Instrument Reading times 5).

4.8.6 Qualify background samples for serial dilution outliers since background samples reflect the matrix of the environmental samples.

4.8.7 Do not qualify field blank, equipment blank, or PE samples for serial dilution outliers since the matrix of these samples are not representative of the environmental samples. If field blanks or PE samples were analyzed for serial dilution, include a comment in CLP PO ACTION section of the report.

4.9 FIELD DUPLICATES

The objective of this section is to evaluate overall precision, including both field and laboratory precision. The results may have more variability than laboratory duplicates. Duplicate sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.6;
- Appendix B2 SOW ILM05.X Table 5.6.

4.9.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form XIV-IN, Field QA/QC Summary Form, instrument printouts, and raw data.
- *SOW ILM05.X*: Form I-IN, Form XIII-IN, Field QA/QC Summary Form, instrument printouts, and raw data.

4.9.2 Aqueous field sample duplicate analyses have control limits of ±20 RPD (or ±CRQL for sample results less than 5 times the CRQL).

4.9.3 Solid field sample duplicate analyses have Region 9 control limits of ±35 RPD (or ±2 times the CRQL for sample results less than 5 times the CRQL), (Region 9 Modification, see Appendix A).

4.9.4 Examine the raw data and recalculate and document in worksheets all of the Field Duplicate RPDs using the equation below:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

S = Field sample result.

D = Field duplicate result.

4.9.5 Verify that all field duplicate QC samples have been properly identified in the report and on the spreadsheet by comparing with the Field QA/QC Summary Form. Label the first duplicate pair as "D1", the second pair as "D2", etc, on Table 1A next to the sample identification.

4.9.6 Do not qualify as estimated or rejected any analyte that exceeds the control limits for laboratory duplicates. Comment on the outlier and provide the RPD in the comment.

4.10 OVERALL ASSESSMENT

Review the entire data package and the data review results and use professional judgment to identify any inconsistencies, anomalies, additive effects of technical problems, impacts on data quality, or other concerns which should be brought to the attention of the data user. Determine whether there is any need to qualify data that were not qualified based on the criteria previously assessed. (Region 9 Modification, see Appendix A).

4.10.1 Inspect Data package to verify the presence of the following review items:

- Complete data package, Field QA/QC Summary Form, preparation logs, calibration standard and spiking standard logs, instrument logs, instrument printouts, and raw data.

4.10.2 Sample Result Verification

Examine the raw data and recalculate and document in worksheets a minimum of 10% of the sample results using the equations below:

4.10.2.1 Water samples:

$$\text{Concentration } \mu g/L = C \times ((Vol._f / Vol._i)) \times DF$$

C = Instrument value in μg/L.

$Vol._f$ = Final digestion volume (mL).

$Vol._i$ = Initial digestion volume (mL).

DF = Dilution factor.

4.10.2.2 Soil samples:

$$\text{Concentration (dry weight) mg/kg} = ((C \times V)/ (W \times S)) \times DF$$

C = Instrument result in (mg/L).

V = Final sample volume (L).

W = Wet sample weight (kg).

S = Percent solids result/100.

DF = Dilution factor.

4.10.2.3 Percent solids calculation for soil samples:

Percent Solids = (Sample *dry* weight / Sample *wet* weight) X 100

4.10.3 Verify that all results on the spreadsheet (Table 1A) were transcribed correctly from the Form 1s. Be sure all "U" and "B" qualifiers have been transposed to the spreadsheet correctly and that "B" flags have been changed to "L" flags.

4.10.4 Verify that preparation blank values from Form 3 are reported on Table 1A.

4.10.5 Verify that every sample has been analyzed, and that results that exceed the calibration range have been diluted (but not over-diluted) and reanalyzed. If not, qualify such results as estimated and comment in CLP PO ACTION.

4.10.6 If any values have been incorrectly recorded, especially those that will cause the qualification of data, notify the laboratory to have the appropriate forms regenerated and resubmitted.

4.10.7 Do not make any corrections on any forms without first checking with the laboratory. Document all communication on a TRL. In general, have the laboratory resubmit any form or page of raw data that requires correction.

4.10.8 When corrected forms and raw data are received by the reviewer, strike each page to be replaced with a single line, mark the page "Replaced," and initial and date. Also, date and initial the replacement page.

4.10.9 Verify the values for all MDLs and CRQLs are transferred from the appropriate Forms to the Table 1A. Form 9 (Form 10 for ILM04.X) to the spreadsheet.

4.10.9.1 Verify that all non-detected results listed on Form 1s and Table 1A have the same value as the IDL, MDL, and CRQL values listed on the appropriate forms.

- If the listed non-detected results (U) Form 1s do not agree with the values listed on Form 9, contact the laboratory via TRL to explain the discrepancy.

- Do not make any corrections on Form 1s without first contacting the laboratory via TRL.

4.10.9.2 Examine the dates on the Form 9s to be sure they are current. MDL studies are to be performed quarterly, and if they are not, this should be noted for CLP PO ACTION. Use professional judgment to determine the effect on day quality.

4.10.10 Examine Form 12 (Form 13 for ILM04.X) and the raw data to determine whether the correct volumes for waters or weights for soils have been used. Refer to the SOW (Exhibit D) for guidance. Note any irregularities in ADDITIONAL COMMENTS or CLP PO ACTION.

4.10.11 Examine Form 13 (Form 14 for ILM04.X) to see if the following SOW protocols have been followed (refer to the SOW for further details).

4.10.11.1 Verify that the correct number of calibration standards and calibration verification standards have been analyzed before the environmental samples: these include the calibration standards, ICV/ICB, CRI, ICSA/ICSAB, and the first CCV and CCB. The LCS and PB are considered samples, and must be run after the first CCV/CCB. If this sample order sequence is not followed, include a comment under CLP PO ACTION.

4.10.11.2 Verify that the CCVs/CCBs were analyzed at the proper frequency during each analytical run. Use professional judgment to determine whether to comment or estimate the associated sample data.

4.10.11.3 Verify that an LCS, a PB, a matrix spike, a laboratory duplicate, and a serial dilution, were analyzed for each matrix. Verify that all analyses of preparation blanks and LCSs are reported on the appropriate forms. Use professional judgment to determine whether to comment or estimate the associated sample data.

4.10.11.4 Verify the dates and times of analysis on the Run Logs against the raw data. Use professional judgment to determine whether to comment or estimate the associated sample data.

4.10.12 If solid samples are included in the SDG, verify calculations for percent solids in the raw data.

4.10.12.1 Verify that the samples were dried for a minimum of 12 hours, but not more than 24 hours in a drying oven maintained at 103-105°C or that there is documentation showing that constant weight was attained.

4.10.12.2 Verify that percent solids values have been transcribed correctly onto the Form 1s and the spreadsheet.

4.10.12.3 If any errors have occurred, contact the laboratory via TRL to confirm the error and to have all relevant forms regenerated.

4.10.12.4 Examine the raw data for sample, standards, and spike preparation and digestion to verify that the correct weights and volumes were used and transcribed to Form 12 (Form 13 for ILM04.X); and that spike levels and calculations are correct. Check for current stock standard true value and traceability certificate. (Region 9 Modification, see Appendix A.)

4.10.12.5 Examine the chain of custody forms to verify that the samples were received intact, and to verify sample type, sample preservation, sample location, laboratory QC sample, dates of sample collection and receipt by the laboratory, and sampler and laboratory receipt signatures. If any of the information is incorrect or missing, including signatures, comment that the effect on the legal defensibility of the data is unknown in ADDITIONAL COMMENTS.

5 INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY DATA REVIEW

This section contains data validation requirements and procedures for metals analysis by inductively coupled plasma – mass spectrometry (ICP-MS). Validation criteria and appropriate actions for each SOW are provided in Appendix B2. Worksheets for each SOW are provided in Appendix C2.

5.1 PRESERVATION AND HOLDING TIMES

The objective of this section is to verify the validity of the results based on the preservation and holding time of the samples from the time of collection to the time of analysis. Holding time and preservation criteria and actions are contained in Appendix B2 SOW ILM05.X Table 5.9. *NOTE: At this time, ICP-MS method in SOW ILM05.2 is for water samples only.*

5.1.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form IA-IN, Form IB-IN, Form XII-IV, Form XIII-IN, EPA Traffic Report/Chain of Custody (TR/COC) record form(s), Form

DC-1, Field QA/QC Summary Form, sample digestion log(s), raw data, and SDG Narrative.

5.1.2 Preservation Criteria:

5.1.2.1 Aqueous Samples

- pH: Examine Form DC-1 and the sample digestion log(s) to verify water samples were preserved to a pH < 2 with nitric acid.

- Temperature: Examine DC-1 and internal COC documents to confirm the cooler temperature upon receipt at the laboratory is 4±2 °C.

5.1.2.2 Soil Samples [There are no soils criteria provided for ICP-MS at this time.]

5.1.3 Holding Time Criteria

5.1.3.1 Verify and document in worksheets all technical holding times by comparing the dates of collection for all samples as reported on the EPA Traffic Reports/Chain of Custody (TR/COC) record form(s) to the dates of analysis from the instrument run logs and the raw data.

5.1.3.2 Technical holding time: If the sample holding time for water or soil is less than or equal to 180 days, no action is required.

5.1.3.3 If the sample holding time for water is greater than 360 days, qualify results as rejected and comment in CLP PO ACTION section. Note that the Region 9 advisory limit for the rejection of data is two times the technical holding time for analyses that grossly exceed the holding time and are or are not properly preserved. (Region 9 Modification, see Appendix A.)

5.2 ICP-MS TUNE ANALYSIS

The objective of this section is to verify that the method requirements for satisfactory instrument tuning are followed and serves as an initial demonstration of instrument stability and precision. ICP-MS tune criteria are contained in Appendix B2 SOW ILM05.X Table 5.10.

5.2.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form XIV-IN, instrument logs, instrument printouts, and raw data.

5.2.2 Instrument Tune Analysis

5.2.2.1 Verify that the ICP-MS tuning solution was analyzed at least five times consecutively prior to instrument calibration. Verify that the tuning solution contains 100 μg/L of beryllium, magnesium (three isotopes), cobalt, indium (two isotopes), and lead (three isotopes). Verify that the peak width is 0.75 atomic mass unit (amu) at five percent (5%) peak height and the mass resolution within 0.1 amu over the range of six to two hundred ten (6-210) amu. [*Note*: the manufacturer some instruments may be required to use 0.65-0.80 amu at 10% peak height criteria.] Check tuning raw data, if provided, and from the raw data, recalculate and document in worksheets (Appendix C) a minimum of 10% of the average isotope mass (%Rs) using the equation below:

$$\text{Average measured mass} = \frac{\sum x}{n}$$

x = Isotope mass from analysis

n = Number of tuning analyses.

5.2.2.2 The Percent Relative Standard Deviation (%RSD) of the absolute signals for all tuning solution analytes must be less than 5 percent (<5%).

Calculate %/RSD using the equation below:

$$\% \text{ RSD} = \frac{\sigma_{n-1} \times 100}{\bar{x}}$$

σ_{n-1} = Standard Deviation

$\bar{x}$ = Mean.

5.3 INSTRUMENT CALIBRATION

The objective of this section is to verify that the method requirements for satisfactory instrument calibration are followed and that the instrument is capable of producing acceptable quantitative data. Initial calibration verification (ICV) demonstrates that the instrument performance is acceptable at the beginning of the analytical run.

Continuing calibration verification (CCV) demonstrates that the initial calibration continues to be valid by checking instrument performance at regular intervals. Initial calibration, initial calibration verification, continuing calibration verification and contract required quantitation limit (CRQL) verification (CRI) criteria are contained in Appendix B2 SOW ILM05.X Table 5.11.

5.3.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form IIA-IN, Form IIB-IN, Form XI-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

5.3.2 Initial Instrument Calibration

5.3.2.1 Verify that the instrument is successfully calibrated daily or once every 24 hours and each time the instrument is set up. Verify that a blank and at least one standard have been used to establish the standard curve and that sample results are within the linear working range of the instrument. Verify that the average of three (minimum) replicate scans is used for all results (standardization, QC, and samples). Inspect the raw data to ensure that the calibration date and time are included.

5.3.3 Initial and Continuing Calibration Verification

5.3.3.1 If an ICV or CCV %R is outside of the control limits, determine if the laboratory stopped the analysis, corrected the problem, recalibrated, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION.

5.3.3.2 From the raw data, recalculate and document in worksheets (Appendix C) a minimum of 10% of the calibration verification percent recoveries (%Rs) for the ICV and CCV using the equation below:

$$\%R = (\textit{Found}\text{ value} / \textit{True}\text{ value}) \times 100$$

Found value = concentration of analyte measured in sample.

True value = certified concentration of analyte(s) in standard.

5.3.3.3 Verify that an ICV standard, prepared from a USEPA or certified stock solution but from a different stock source or lot than used for calibration, is analyzed at the analytical mass used for sample analysis and reported immediately after ICP system calibration, but before the analysis of any analytical samples. Examine and compare all ICV %Rs to values reported by the laboratory on Form IIA and criteria in Appendix B2.

5.3.3.4 Verify that CCV standards are analyzed and reported at the beginning and end of the analytical run, after an ICV, and at a frequency of 10% of samples analyzed or every two hours during an analytical run, whichever is more frequent. Verify that the CCV standards are prepared from a different source or lot than used for the ICV, are at or near the mid-range of the calibration curve, and is analyzed at the analytical mass used for sample analysis.

5.3.4 Contract Required Quantitation Limit Verification

5.3.4.1 Verify that a CRQL verification standard (CRI) was analyzed to demonstrate that the established calibration is linear and valid near the CRQL.

5.3.4.2 Recalculate and document in worksheets a minimum of 10% of the CRI verification %Rs using the equation below:

$$\text{CRI \%R} = (\textit{Found} \text{ value} / \textit{True} \text{ value}) \times 100$$

5.3.4.3 There are EPA established limits for CRI in SOW ILM05.X. Compare CRI results to criteria and actions in Appendix B ICP-MS Table 5.3.

- Verify that a CRI is analyzed after the ICV, but before the Interference Check Sample (ICS), every 20 analytical samples, and at the end of the run.

- If calibration criteria are grossly exceeded, include a comment in the CLP PO ACTION section of the report.

5.4 BLANKS

The objective of this section is to assess the results of blank analyses to determine the existence and magnitude of contamination from laboratory or field activities. The criteria for evaluation of blanks apply to any blank associated with the samples. Initial calibration blank (ICB), continuing calibration blank (CCB), preparation blank (PB), field blank (FB), and equipment blank (EB) criteria are contained in Appendix B2 SOW ILM05.X Table 5.12.

5.4.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, Field QA/QC Summary Form, sample preparation logs, instrument logs, instrument printouts, and raw data.

5.4.2 No contaminants should be found in any blank. If problems exist with any blank, evaluate all sample data associated with the blank to determine if the problem is system wide or an isolated occurrence.

5.4.3 Calibration Blanks

5.4.3.1 Verify that an ICB is analyzed after the ICV. Verify that if the absolute value of an analyte in an ICB is greater than the CRQL, the analysis is terminated, the problem corrected, and the associated samples reanalyzed. If the ICB does not meet criteria, apply action to all samples in the analytical run. If the ICB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

5.4.3.2 Verify that a CCB is analyzed after every CCV. If the absolute value of an analyte in a CCB is greater than the CRQL, verify that the analysis is terminated, the problem corrected, and the associated samples reanalyzed. Verify that the CCB is analyzed at a frequency of every ten samples or every two hours during the run, whichever is more frequent. If CCBs do not meet criteria, apply actions to all samples associated with the CCBs out of criteria. If the CCB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

5.4.4 Preparation Blank

5.4.4.1 Verify that a PB was digested and analyzed for each matrix per SDG or batch of samples digested, whichever is more frequent. If the absolute value of any analyte in the PB is less than or equal to the CRQL, no action is required. If the PB does not meet criteria, apply action to all samples prepared in the same preparation batch. If the PB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

5.4.4.2 Verify that analyte concentrations in the PB are less than the CRQL. Confirm that all samples associated with a contaminated PB contain the contaminated analyte at a concentration greater than 5 times the PB concentration. Verify that the all samples were re-digested and reanalyzed (except for identified field blanks). Note that the laboratory is not to correct any sample concentration by subtracting the blank value. Include a comment in the CLP PO ACTION section of the report if the laboratory failed to re-digest and reanalyze the affected samples.

- *Region 9 Modification*: For PB contamination, qualify associated sample results greater than the MDL but less than 5 times the amount in the blank as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.)

5.4.4.3 If the analyte concentration in the PB is less than the negative CRQL value, all samples associated with the contaminated PB with contaminating analyte concentrations less than 5 times the CRQL should have been re-digested and reanalyzed. Include a comment in the CLP PO ACTION section of the report if the laboratory failed to re-digest and reanalyze the affected samples.

5.4.5 Field Blanks

5.4.5.1 Verify all field QC blank samples listed on the Field QA/QC Summary Form and identified on the spreadsheet. Check the Field QA/QC Summary Form to verify that all field QC samples have been properly identified in the report, and on the spreadsheet. Use professional judgment to determine whether to comment or estimate the associated sample data.

5.4.6 If the absolute value of the concentration of an analyte in a blank is greater than the CRQL, apply the following guidelines.

5.4.6.1 For PB contamination, qualify associated sample results greater than the MDL but less than 5 times the amount in the blank as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.)

5.4.6.2 For ICB or CCB contamination, determine all sample numbers whose results were used from the analytical run between the last acceptable ICB or CCB and the next acceptable CCB. Associated sample results greater than the MDL but less than 5 times the amount in the blank are qualified as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.)

5.4.6.3 For any laboratory blank (PB, ICB, CCB) with a negative result whose absolute value is greater than the CRQL, use professional judgment to determine the effect on data quality, i.e., low bias, and include a comment under CLP PO ACTION.

5.4.6.4 For FB or EB contamination, identify associated samples based on the same date of collection as the field QC. Associated sample results greater than the MDL but less than 5 times the amount in the blank are qualified as non-detected and estimated (UJ). Include a comment under SAMPLING ISSUES (Region 9 Modification, see Appendix A.)

5.4.7 For sample results greater than the CRQL and affected by blank contamination, apply the following guidelines.

5.4.7.1 For undiluted samples, increase associated sample results less than the CRQL to the CRQL and qualify as non-detected (UJ).

5.4.7.2 For diluted samples, increase associated sample results less than the CRQL to the product of the CRQL multiplied by the sample dilution factor (CRQL times df) and qualify as non-detected (UJ).

5.4.7.3 If an ICB or CCB analyte result is greater than the CRQL, and the analysis was not terminated and the affected samples were not reanalyzed, raise the CRQL of the affected samples to the concentration found in the ICB or CCB. Report non-detects and results greater than or equal to the MDL, but less than or equal to the CRQL as [CRQL]U. Include a comment in the CLP PO ACTION section of the report.

5.5 ICP-MS INTERFERENCE CHECK SOLUTION (ICSA AND ICSAB)

The objective of this section is to verify that the ICP interference check solution (ICS) demonstrates the ability of the analytical instrument to overcome typical interferences found in environmental samples. ICS criteria are contained in Appendix B2 SOW ILM05.X Table 5.13.

5.5.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form IVB-IN, Form XIII-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

5.5.2 An ICS analysis consists of analyzing two solutions in sequence, solution A (interferents only) then solution AB (interferents and target analytes), for all masses used for each analyte or interferent reported by ICP-MS.

5.5.2.1 Examine all ICP ICS results on Form 4B. The laboratory should have analyzed and reported ICS results for all analytes being reported from the analytical run and for all interferents (target and non target) for these reported analytes. Note that ICS solution A is analyzed before ICS solution AB.

5.5.2.2 ICSA and ICSAB solutions must be run at the beginning, the end, and after every 20 analytical samples of each analytical run. The ICS must be analyzed after the ICV/ICB, but before the CCV.

5.5.2.3 Recalculate and document in worksheets a minimum of 10% of the ICS %Rs using the equation below:

ICS %R = (ICS *Found* Value / ICS *True* Value) X 100

5.5.2.4 ICSA and ICSAB %Rs must fall within the control limits of three times (3 times) the CRQL, or 80-120% of the true value (whichever is greater) for the analytes and interferents in the solution. When an ICS %R is outside of the control limits, the laboratory is required to stop, correct the problem, recalibrate, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION.

5.6 LABORATORY CONTROL SAMPLE (LCS)

The objective of this section is to monitor the laboratory's overall performance at each step of the analysis, including sample preparation. Laboratory control sample criteria are contained in Appendix B2 SOW ILM05.X Table 5.14.

5.6.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form VII-IN, Form XII-IN, Form XIII-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

5.6.2 Verify that one aqueous LCS was prepared and analyzed for every group of aqueous samples in an SDG or with each batch of aqueous samples digested, whichever is more frequent. The aqueous LCS must be prepared and analyzed using the identical preparation and analytical methods used for the water samples. The aqueous LCS solution should be obtained from the USEPA, if available. If the LCS is unavailable from the USEPA, the ICV solution may be used.

5.6.3 Aqueous LCS %Rs must be within the 80-120% control limits. If the %R is outside the control limits the laboratory is required to stop, correct the problem, recalibrate, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION section of the report. Use professional judgment to assess the data.

5.6.4 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the %Rs using the equation below:

LCS %R = (LCS *Found* / LCS *True* Value) X 100

LCS *Found* = concentration of analyte measured in LCS sample.

LCS *True* Value = certified concentration of analyte in LCS source.

5.6.5 Verify that at least one LCS has been analyzed for each group of samples of similar matrix type and concentration, or for each SDG.

5.7 DUPLICATE SAMPLE ANALYSIS

The objective of this section is to verify that the laboratory demonstrated acceptable method precision at the time of sample analysis. Duplicate sample criteria are contained in Appendix B2 SOW ILM05.X Table 5.15.

5.7.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form I-IN, Form VI-IN, Form XII-IN, Form XIII-IN, QA/QC Summary Form, instrument logs, instrument printouts, and raw data.

5.7.2 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the relative percent deviations (RPDs) using the equation below:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

S = Sample result (original).

D = Duplicate result.

5.7.3 Do not report an RPD of 200 when one result is non-detected and the other result is detected greater than or equal to the CRQL. State in the duplicate sample comment that the analyte was reported at a given concentration in one duplicate analysis, but not detected in the other.

5.7.4 Verify that at least one duplicate sample analysis has been performed on each group of samples of a similar matrix type and concentration, or for each SDG. Check to make sure that the QC sample designated on the chain of custody was used. If inadequate QC has been performed, use professional judgment to determine the effect on the data quality, and include a comment in the CLP PO ACTION section of the report.

5.7.5 Qualify Background samples for duplicate outliers if the background sample matrix reflects the matrix of the environmental samples.

5.7.6 Do not qualify results for field blank, equipment blank or PE sample data for duplicate outliers since the matrix of the blanks is not similar to the environmental samples. If a field blank was used for duplicate sample analysis, note such use of a field blank in CLP PO ACTION and include a statement in Section III, Validity and Comments, indicating that this precision parameter could not be evaluated due to improperly performed QC.

5.8 SPIKE SAMPLE ANALYSIS

The objective of this section is to provide information about the effect of each sample matrix on the sample preparation procedures and the analytical method. Matrix spike (MS) criteria are contained in Appendix B2 SOW ILM05.X Table 5.16.

5.8.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form I-IN (for QC sample), Form VA-IN, Form VB-IN, Form XII-IN, Form XIII-IN, Field QA/QC Summary Form, standard preparation logs, instrument logs, instrument printouts, and raw data.

5.8.2 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the MS %Rs using the equation below:

$$\text{Matrix spike \%R} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spiked sample result from the original sample.

SR = Sample result. Use SR = 0 when sample concentration is Less than the MDL.

SA = Spike added.

5.8.3 Use the results of the sample designated as the "original sample" to perform re-calculations of the matrix spike. Do not use the average of the "original sample" and the laboratory duplicate sample results for the purpose of determining the matrix spike %R.

5.8.4 When the matrix spike recovery of an analyte falls outside the 75-125 %R control limits and the analyte result is less than four times the spike added, verify that a post-digestion spike was performed for those analytes. Verify that an aliquot of the remaining unspiked sample was spiked at two times (2 times) the indigenous level or two times (2 times) the analyte CRQL whichever is greater.

5.8.5 Do not qualify matrix spike recovery in instances where an analyte result exceeds four times the spiking concentration.

5.8.6 A post-digestion spike must be performed for any analyte that does not meet the specified MS %R criterion. An aliquot of the remaining unspiked sample shall be spiked at two times (2 times) the indigenous level or 2 times the CRQL, whichever is greater. If a required post-digestion spike was not performed, comment in the CLP PO ACTION section of the report.

5.8.7 Verify that the spike was added before the digestion of the sample, and that the correct spike concentrations were used in spiking the matrix spike sample by examining the digestion logs and referring to the SOW.

5.8.8 Qualify samples designated as background samples for MS outliers if they reflect the matrix of the environmental samples.

5.8.9 Do not qualify field blank, equipment blank, or performance evaluation (PE) sample data due to MS outliers since these matrices are not representative of environmental samples. If an improper sample, i.e., field or equipment blank, was used for matrix spike analysis and there was no QC sample listed on the COC, comment in SAMPLING and include a comment in Section III, Validity and Comments, indicating that accuracy and precision parameters could not be evaluated due to improperly performed QC.

5.8.10 If inadequate QC has been performed (i.e., due to insufficient sample, etc.), use professional judgment to determine the effect on the data quality, and include a comment in CLP PO ACTION and in Section III, Validity and Comments.

5.8.11 If incorrect concentrations were used, use professional judgment to determine the effect on the data quality, and include a comment in CLP PO ACTION.

5.9 ICP SERIAL DILUTION ANALYSIS

The objective of this section is to verify, through a serial dilution of a sample, whether or not significant physical or chemical interferences exist due to sample matrix for samples analyzed by ICP-MS. ICP serial dilution analysis criteria are contained in Appendix B2 SOW ILM05.X Table 5.17.

5.9.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form I-IN, Form VIII-IN, Form XIII-IN, Field QA/QC Summary Form, instrument printouts, and raw data.

5.9.2 Verify that an ICP serial dilution sample is prepared and analyzed for every group of samples with a similar matrix type or for each SDG, or with each batch of aqueous samples digested, whichever is more frequent.

5.9.3 If the analyte concentration in the original sample is greater than fifty times (50 times) the method detection limit (MDL), the serial dilution results must be within a ten percent difference (%D) of the original sample results after correction for dilution.

5.9.4 Examine the raw data for evidence of interference; i.e., for results of the diluted sample which are significantly higher or lower than the results of the original sample. Use professional judgment and document when qualifying sample results. Indicate in the report if there is a possible high or low bias in the results.

5.9.5 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the percent differences (%Ds) using the equation below:

$$\%D = (I - S/ I) \times 100$$

I = Initial Sample Result.

S = Serial Dilution Result (Instrument Reading times 5).

5.9.6 Qualify background samples for serial dilution outliers if background samples reflect the matrix of the environmental samples.

5.9.7 Do not qualify field blank, equipment blank, or PE samples for serial dilution outliers since the matrix of these samples are not representative of the environmental samples. If field blanks or PE samples were analyzed for serial dilution, include a comment in CLP PO ACTION section of the report.

5.10 ICP-MS INTERNAL STANDARDS

The objective of this section is to determine the existence and magnitude of instrument drift and physical interferences to samples analyzed by ICP-MS. The evaluation of internal standard results applies to all analytical samples, QC samples, and calibration standards. Internal standards criteria are contained in Appendix B2 SOW ILM05.X Table 5.18.

5.10.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form XV-IN, instrument printouts, and raw data.

5.10.2 Verify using Form XV-IN and instrument raw data that all samples analyzed, except the ICP-MS tune solution, contain a minimum of five internal standards. The SOW allows the following internal standards: enriched lithium (Li^6 isotope), scandium (Sc), yttrium (Y), rhodium (Rh), indium (In^{115} isotope), terbium (Tb), holmium (Ho), lutetium (Lu), and bismuth (Bi). The masses of the internal standards must bracket the masses of the target analytes.

5.10.3 If no internal standards were analyzed, less than five allowed internal standards were analyzed, or the internal standards do not bracket the target analyte masses, note in the CLP PO ACTION section.

5.10.4 Verify the percent relative intensity (%RI) for all samples and standards, except the tuning solution, fall within 60-125% of the response in the calibration blank.

5.10.5 If the %RI of an internal standard of a sample falls outside the 60-125% limits, verify that the sample was reanalyzed at a two times (2 times) dilution.

5.11 FIELD DUPLICATES

The objective of this section is to evaluate overall precision, including both field and laboratory precision. The results may have more variability than laboratory duplicates. Duplicate sample criteria are contained in Appendix B2 SOW ILM05.X Table 5.15.

5.11.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM05.X*: Form I-IN, Form XIII-IN, Field QA/QC Summary Form, instrument printouts, and raw data.

5.11.2 Aqueous field sample duplicate analyses have control limits of ±20 RPD (or ±CRQL for sample results less than 5 times the CRQL).

5.11.3 Solid field sample duplicate analyses have Region 9 control limits of ±35 RPD (or ±2 times the CRQL for sample results less than 5 times the CRQL). (Region 9 Modification, see Appendix A).

5.11.4 Examine the raw data and recalculate and document in worksheets all of the Field Duplicate RPDs using the equation below:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

S = Field sample result.

D = Field duplicate result.

5.11.5 Verify that all field duplicate QC samples have been properly identified in the report and on the spreadsheet by comparing with the Field QA/QC Summary Form. Label the first duplicate pair as "D1", the second pair as "D2", etc. on Table 1A next to the sample identification.

5.11.6 Do not qualify as estimated or rejected any analyte, which exceeds the control, limits for laboratory duplicates. Comment on the outlier and provide the RPD in the comment.

5.12 OVERALL ASSESSMENT

Review the entire data package and the data review results and use professional judgment to identify any inconsistencies, anomalies, additive effects of technical problems, impacts on data quality, or other concerns which should be brought to the attention of the data user. Determine whether there is any need to qualify data, which were not qualified, based on the criteria previously assessed. (Region 9 Modification, see Appendix A.)

5.12.1 Inspect Data package to verify the presence of the following review items:

- Complete data package, Field QA/QC Summary Form, preparation logs, calibration standard and spiking standard logs, instrument logs, instrument printouts, and raw data.

5.12.2 Sample Result Verification

Examine the raw data and recalculate and document in worksheets a minimum of 10% of the sample results using the equations below:

5.12.2.1 Water samples prepared using Method HW2:

Concentration μg/L = C x (V_f / V_i) x (V_f /20) x DF

C = Averaged Instrument value in μg/L.

V_f = Final digestion volume (50 mL).

V_i = Initial digestion volume (100 mL).

DF = Dilution factor.

5.12.2.2 Water samples prepared using Method HW3:

Concentration μg/L = C x DF

C = Averaged Instrument value in μg/L.

DF = Dilution factor.

5.12.2.3 Percent solids calculation for soil samples: [Not required at this time.]

5.12.3 Verify that all results on the spreadsheet (Table 1A) were transcribed correctly from the Form 1s. Be sure all "U" and "B" qualifiers have been transposed to the spreadsheet correctly and that "B" flags have been changed to "L" flags.

5.12.4 Verify that preparation blank values from Form 3 are reported on Table 1A.

5.12.5 Verify that every sample has been analyzed, and that results, which exceed the calibration range, have been diluted (but not over-diluted) and reanalyzed. If not, qualify such results as estimated and discuss them in CLP PO ACTION.

5.12.6 If any values have been incorrectly recorded, especially those, which will cause the qualification of data, notify the laboratory to have the appropriate forms regenerated and resubmitted.

5.12.7 Do not make any corrections on any forms without first checking with the laboratory. Document all communication on a TRL. In general, have the laboratory resubmit any form or page of raw data that requires correction.

5.12.8 When corrected forms and raw data are received by the reviewer, strike each page to be replaced with a single line, mark the page "Replaced," and initial and date. Also, date and initial the replacement page.

5.12.9 Verify the values for all MDLs and CRQLs are transferred from the Form 9 to the Table 1A spreadsheet. *Note that the analyte MDL must be less than one half the CRQL.*

5.12.9.1 Verify that all non-detected results listed on Form 1s and Table 1A have the same value as the CRQL values listed on the Form 9.

- If the listed non-detected results (U) Form 1s do not agree with the values listed on Form 9, contact the laboratory via TRL to explain the discrepancy.

- Do not make any corrections on Form 1s without first contacting the laboratory via TRL.

5.12.9.2 Examine the dates on the Form 9s to be sure they are current. MDL studies are to be performed quarterly, and if they are not, this should be noted for CLP PO ACTION. Use professional judgment to determine the effect on data quality.

5.12.10 Examine Form 12 and the raw data to determine whether the correct volumes for waters have been used. Refer to the SOW (Exhibit D) for guidance. Note any irregularities in ADDITIONAL COMMENTS or CLP PO ACTION.

5.12.11 Examine Form 13 to see if the following SOW protocols have been followed (refer to the SOW for further details).

5.12.11.1 Verify that the correct number of calibration standards and calibration verification standards were analyzed before the environmental samples: these include the calibration standards, ICV/ICB, CRI, ICSA/ICSAB, and the first CCV and CCB. The LCS and PB are considered samples, and must be run after the first CCV/CCB. If this sample order sequence is not followed, include a comment under CLP PO ACTION.

5.12.11.2 Verify that the CCVs/CCBs were analyzed at the proper frequency during each analytical run. Use professional judgment to determine whether to comment or estimate the associated sample data.

5.12.11.3 Verify that an LCS, a PB, a matrix spike, a laboratory duplicate, and a serial dilution, were analyzed for each matrix. Verify that all analyses of preparation blanks and LCSs are reported on the appropriate forms. Use professional judgment to determine whether to comment or estimate the associated sample data.

5.12.11.4 Verify the dates and times of analysis on the Run Logs against the raw data. Use professional judgment to determine whether to comment or estimate the associated sample data.

5.12.11.5 If any errors have occurred, contact the laboratory via TRL to confirm the error and to have all relevant forms regenerated.

5.12.11.6 Examine the raw data for sample, standards, and spike preparation and digestion to verify that the correct volumes were used and transcribed to Form 12; and that spike levels and calculations are correct. Check for current stock standard true value and traceability certificate.

5.12.11.7 Examine the chain of custody forms to verify that the samples were received intact, and to verify sample type, sample preservation, sample location, laboratory QC sample, dates of sample collection and receipt by the laboratory, and sampler and laboratory receipt signatures. If any of the information is incorrect or missing, including signatures, comment that the effect on the legal defensibility of the data is unknown in ADDITIONAL COMMENTS.

6 MERCURY DATA REVIEW

This section contains data validation requirements and procedures for mercury analysis by cold vapor atomic absorption spectroscopy (CVAA). Validation criteria and appropriate actions for each SOW are provided in Appendices B1 and B2. Worksheets for each SOW are provided in Appendices C1 and C2.

6.1 PRESERVATION AND HOLDING TIMES

The objective of this section is to verify the validity of the results based on the preservation and holding time of the samples from the time of collection to the time of analysis. Holding time and preservation criteria and actions are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.9;
- Appendix B2 SOW ILM05.X Table 5.19.

6.1.1 Inspect Data Package to verify the presence of the following review items:

- *ILM04.X*: Form I-IN, Form XII-IN, Form XIII-IN, EPA Traffic Report and/or Chain of Custody (COC) record form(s), Form DC-1, Field QA/QC Summary Form, sample digestion log(s), raw data, and sample delivery group (SDG) Narrative.

- *ILM05.X*: Form IA-IN, Form IB-IN, Form XII-IV, Form XIII-IN, EPA Traffic Report/Chain of Custody (TR/COC) record form(s), Form DC-1, Field QA/QC Summary Form, sample digestion log(s), raw data, and SDG Narrative.

6.1.2 Preservation Criteria:

6.1.2.1 Aqueous Samples

- pH: Examine Form DC-1 and the sample digestion log(s) to verify water samples were preserved to a pH < 2 with nitric acid.

- Temperature: Examine DC-1 and internal COC documents to confirm the cooler temperature upon receipt at the laboratory is 4±2°C.

6.1.2.2 Soil Samples

- Temperature: Examine DC-1 and internal COC documents to confirm the cooler temperature upon receipt at the laboratory is 4±2°C.

6.1.3 Holding Time Criteria

6.1.3.1 Verify and document in worksheets all technical holding times by comparing the dates of collection for all samples as reported on the EPA Traffic Reports/Chain of Custody (TR/COC) record form(s) to the dates of analysis from the instrument run logs and the raw data.

6.1.3.2 Technical holding time: If the sample holding time for water is less than or equal to 28 days and preserved with nitric acid, no action is required.

6.1.3.3 If the sample holding time for water is greater than 56 days, qualify results as rejected and comment in CLP PO ACTION section. Note that the Region 9 advisory limit for the rejection of data is two times the technical holding

time for analyses that grossly exceed the holding time and are or are not properly preserved. (Region 9 Modification, see Appendix A.)

6.2 INSTRUMENT CALIBRATION

The objective of this section is to verify that the method requirements for satisfactory instrument calibration are followed and that the instrument is capable of producing acceptable quantitative data. Initial calibration verification (ICV) demonstrates that the instrument performance is acceptable at the beginning of the analytical run. Continuing calibration verification (CCV) demonstrates that the initial calibration continues to be valid by checking instrument performance at regular intervals. Initial calibration, initial calibration verification, continuing calibration verification and contract required detection limit (CRDL)/contract required quantitation limit (CRQL) verification (CRI) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.10;
- Appendix B2 SOW ILM05.X Table 5.20 and Table 5.21.

6.2.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form IIA-IN, Form IIB-IN, Form XII-IN, Form XIV-IN, calibration standard logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form IIA-IN, Form IIB-IN, Form XIII-IN, calibration standard logs, instrument logs, instrument printouts, and raw data.

6.2.2 Initial Instrument Calibration

6.2.2.1 Verify that the instrument is successfully calibrated daily or once every 24 hours and each time the instrument is set up. Verify that a blank and at least four standards have been used to establish the standard curve and that sample results are within the linear working range of the instrument. One calibration standard must be at the CRQL concentration. Verify that the average of two replicate exposures (minimum) is used for all results. The calibration correlation coefficient must be greater than or equal to 0.995. Inspect the raw data to ensure that the calibration date and time are included. Analytical wavelength used for calibration must be used throughout the analytical sequence.

6.2.3 Initial and Continuing Calibration Verification

6.2.3.1 If an ICV or CCV %R is outside of the control limits, determine if the laboratory stopped the analysis, corrected the problem, recalibrated, and

reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION.

6.2.3.2 From the raw data, recalculate and document in worksheets (Appendix C) a minimum of 10% of the calibration verification percent recoveries (%Rs) using the equations below:

ICV %R = (*Found* value / *True* value) x 100

CCV %R = (*Found* value / *True* value) x 100

6.2.3.3 Verify that an ICV standard, prepared from a USEPA or certified stock solution but from a different stock source or lot than used for calibration, is analyzed at the wavelengths used for sample analysis and reported immediately after ICP system calibration, but before the analysis of any analytical samples. Examine all ICV %Rs and compare to criteria in appendix.

6.2.3.4 Verify that CCV standards are analyzed and reported at the beginning and end of the analytical run, after an ICV, and at a frequency of 10% of samples analyzed or every two hours during an analytical run, whichever is more frequent. Verify that the CCV standards are prepared from a different source or lot than used for the ICV and are at or near the mid-range of the calibration curve.

6.2.4 Contract Required Detection Limit/Contract Required Quantitation Limit Verification

6.2.4.1 Verify CRDL/CRQL verification standard (CRA/CRI) was analyzed to demonstrate that the established calibration is linear and valid near the CRDL/CRQL.

6.2.4.2 Recalculate and document in worksheets a minimum of 10% of the CRA/CRI verification %Rs using the equation below:

CRA/CRI %R = (*Found* value / *True* value) x 100

6.2.4.3 There are no EPA-established limits for CRA in *SOW ILM04.X*. Use the Region 9 advisory limits of 65-135%R. (Region 9 Modification, see Appendix A.2) Compare to criteria in appendix.

- Verify that the CRA is analyzed after the ICV and at the end of the run or a minimum of 2 times per 8 hour shift, whichever is more frequent. If the frequency is not met, note in CLP PO ATTENTION but do not qualify any results.

- The CRA concentration should be two times the CRDL specified for the target analyte, or two times the instrument detection limit (IDL), whichever is greater.

6.2.4.4 There are EPA established limits for CRI in SOW ILM05.X. Compare CRI results to criteria and actions in appendix.

- Verify that a CRI is analyzed after the ICV, every 20 analytical samples, and at the end of the run. The initial CRI must immediately follow the ICV/ICB analysis.

- If CRI results are outside acceptance limits, the laboratory shall reanalyze the CRI standard. If the CRI reanalysis results are still outside acceptance limits, the laboratory should terminate analysis, correct problem, recalibrate instrument, and verify calibration.

6.3 BLANKS

The objective of this section is to assess the results of blank analyses to determine the existence and magnitude of contamination from laboratory or field activities. The criteria for evaluation of blanks apply to any blank associated with the samples. Initial calibration blank (ICB), continuing calibration blank (CCB), preparation blank (PB), field blank (FB), and equipment blank (EB) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.11;
- Appendix B2 SOW ILM05.X Table 5.22.

6.3.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form III-IN, Form XIII-IN, Form XIV-IN, Field QA/QC Summary Form, sample preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, Field QA/QC Summary Form, sample preparation logs, instrument logs, instrument printouts, and raw data.

6.3.2 No contaminants should be found in any blank. If problems exist with any blank, evaluate all sample data associated with the blank to determine if the problem is system wide or an isolated occurrence.

6.3.3 Calibration Blanks

6.3.3.1 Verify that an ICB is analyzed after the ICV. Verify that if the absolute value of an analyte in an ICB is greater than the CRQL, the analysis is terminated, the problem corrected, and the associated samples reanalyzed. If the ICB does not meet criteria, apply action to all samples in the analytical run. If the ICB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

6.3.3.2 Verify that a CCB is analyzed after every CCV. If the absolute value of an analyte in a CCB is greater than the CRQL, verify that the analysis is terminated, the problem corrected, and the associated samples reanalyzed. Verify that the CCB is analyzed at a frequency of every ten samples or every two hours during the run, whichever is more frequent. If CCBs do not meet criteria, apply actions to all samples analyzed between the previous acceptable CCB and the subsequent acceptable CCB in the analytical run. If the CCB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

6.3.4 Preparation Blank

6.3.4.1 Verify that a PB was digested and analyzed for each matrix per SDG or batch of samples digested, whichever is more frequent. If the absolute value of any analyte in the PB is less than or equal to the CRQL, no action is required. If the PB does not meet criteria, apply action to all samples prepared in the same preparation batch. If the PB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

6.3.4.2 Verify that analyte concentrations in the PB are less than the CRQL. Confirm that all samples associated with the contaminated PB contain the contaminated analyte at a concentration greater than 5 times the PB concentration. Verify that all samples were re-digested and reanalyzed (except for identified field blanks). Note that the laboratory is not to correct any sample concentration by subtracting the blank value. Include a

comment in the CLP PO ACTION section of the report if the laboratory failed to re-digest and reanalyze the affected samples.

- *Region 9 Modification*: For PB contamination, qualify associated sample results greater than the MDL but less than 5 times the amount in the blank as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.)

6.3.4.3 If the analyte concentration in the PB is less than the negative CRQL value, all samples associated with the contaminated PB with contaminating analyte concentrations less than 5 times the CRQL should have been re-digested and reanalyzed. Include a comment in the CLP PO ACTION section of the report if the laboratory failed to re-digest and reanalyze the affected samples.

6.3.5 Field Blanks

6.3.5.1 Verify all field QC blank samples listed on the Field QA/QC Summary Form and identified on the spreadsheet. Check the Field QA/QC Summary Form to verify that all field QC samples have been properly identified in the report, and on the spreadsheet. Use professional judgment to determine whether to comment or estimate the associated sample data.

6.3.6 If the absolute value of the concentration of an analyte in a blank is greater than the CRQL, apply the following guidelines.

6.3.6.1 For PB contamination, qualify associated sample results greater than the MDL but less than 5 times the amount in the blank as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.)

6.3.6.2 For ICB or CCB contamination, determine all sample numbers whose results were used from the analytical run between the last acceptable ICB or CCB and the next acceptable CCB. Associated sample results greater than the MDL but less than 5 times the amount in the blank are qualified as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.)

6.3.6.3 For any laboratory blank (PB, ICB, CCB) with a negative result whose absolute value is greater than the CRQL, use professional judgment to determine the effect on data quality, i.e., low bias, and include a comment under CLP PO ACTION.

6.3.6.4 For FB or EB contamination, identify associated samples based on the same date of collection as the field QC. Associated sample results greater than

the MDL but less than 5 times the amount in the blank are qualified as non-detected and estimated (UJ). Include a comment under SAMPLING ISSUES (Region 9 Modification, see Appendix A.).

6.3.7 For sample results less than the CRQL and affected by blank contamination, apply the following guidelines.

6.3.7.1 For undiluted samples, increase associated sample results < the CRQL to the CRQL and qualify as non-detected (UJ).

6.3.7.2 For diluted samples, increase associated sample results < the CRQL to the product of the CRQL multiplied by the sample dilution factor (CRQL times df) and qualify as non-detected (UJ).

6.3.7.3 If an ICB or CCB analyte result is greater than the CRQL, and the analysis was not terminated and the affected samples were not reanalyzed, raise the CRQL of the affected samples to the concentration found in the ICB or CCB. Report non-detects and results greater than or equal to the MDL, but less than or equal to the CRQL as [CRQL]U. Include a comment in the CLP PO ACTION section of the report.

6.4 SPIKE SAMPLE ANALYSIS

The objective of this section is to provide information about the effect of each sample matrix on the sample preparation procedures and the analytical method. Matrix spike (MS) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.12;
- Appendix B2 SOW ILM05.X Table 5.23.

6.4.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN; Forms V-IN (Parts A and B), Form XIII-IN, Form XIV- IN, Field QA/QC Summary Form, standard preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form I-IN (for QC sample), Form VA-IN, Form VB-IN, Form XII-IN, Form XIII-IN, Field QA/QC Summary Form, standard preparation logs, instrument logs, instrument printouts, and raw data.

6.4.2 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the MS %Rs using the equation below:

$$\text{Matrix spike \%R} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spiked sample result from the original sample.

SR = Sample result. Use SR = 0 when sample concentration is less than the MDL.

SA = Spike added.

6.4.3 Use the results of the sample designated as the "original sample" to perform re-calculations of the matrix spike. Do not use the average of the "original sample" and the laboratory duplicate sample results for the purpose of determining the matrix spike %R.

6.4.4 Post-digestion (analytical) spikes are not required for mercury.

6.4.5 Do not evaluate matrix spike recovery in instances where a sample result exceeds four times the spiking concentration.

6.4.6 Verify that at least one matrix spike sample analysis has been performed on each group of samples of a similar matrix type and concentration, or for each SDG.

6.4.7 Check that the QC sample designated on the chain of custody is used for matrix spike analysis. Comment in "Additional Comments" if a different sample than the one designated on the COC is used

6.4.8 Verify that the spike was added before the digestion of the sample, and that the correct spike concentrations were used in spiking the matrix spike sample by examining the digestion logs and referring to the SOW.

6.4.9 Qualify samples designated as background samples for MS outliers since they reflect the matrix of the environmental samples.

6.4.10 Do not qualify field blank, equipment blank, or performance evaluation (PE) sample data due to MS outliers since these matrices are not representative of environmental samples. If an improper sample, i.e., field or equipment blank, was used for matrix spike analysis and there was no QC sample listed on the COC, comment in SAMPLING and include a comment in Section III, Validity and Comments, indicating that accuracy and precision parameters could not be evaluated due to improperly performed QC.

6.4.11 If inadequate QC has been performed (i.e., due to insufficient sample, etc.), use professional judgment to determine the effect on the data quality, and include a comment in Section III, Validity and Comments.

6.4.12 If incorrect concentrations were used, use professional judgment to determine the effect on the data quality, and include a comment in Section III, Validity and Comments.

6.5 DUPLICATE SAMPLE ANALYSIS

The objective of this section is to verify that the laboratory demonstrated acceptable method precision at the time of sample analysis. Duplicate sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.13;
- Appendix B2 SOW ILM05.X Table 5.24.

6.5.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form VI-IN, Form XIII-IN, Form XIV-IN, QA/QC Summary Form, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form I-IN, Form VI-IN, Form XII-IN, Form XIII-IN, QA/QC Summary Form, instrument logs, instrument printouts, and raw data.

6.5.2 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the RPDs using the equation below:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

S = Sample result (original).

D = Duplicate result.

6.5.3 Do not report an RPD of 200 when one result is non-detected and the other result is detected greater than or equal to the CRQL. State in the duplicate sample comment that the analyte was reported at a given concentration in one duplicate analysis, but not detected in the other. (Region 9 Modification, see Appendix A.)

6.5.4 For solid samples, verify that duplicate sample analyses are performed for Percent Solids.

6.5.5 Verify that at least one duplicate sample analysis has been performed on each group of samples of a similar matrix type and concentration, or for each SDG.

Check to make sure that the QC sample designated on the chain of custody was used. Duplicate analysis does not need to be performed on samples labeled as rinsates in an SDG of soil samples.

6.5.6 If inadequate QC has been performed, use professional judgment to determine the effect on the data quality, and include a comment in Section III, Validity and Comments.

6.5.7 Qualify Background samples for duplicate outliers since the background sample matrix reflects the matrix of the environmental samples.

6.5.8 Do not qualify results for field blank, equipment blank, or PE sample data for duplicate outliers since the matrix of the blanks is not similar to the environmental samples. If a field blank was used for duplicate sample analysis, note such use of a field blank in CLP PO ACTION and include a statement in Section III, Validity and Comments, indicating that this precision parameter could not be evaluated due to improperly performed QC.

6.6 LABORATORY CONTROL SAMPLE (LCS)

The objective of this section is to monitor the laboratory's overall performance at each step of the analysis, including sample preparation. Laboratory control sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.14;
- Appendix B2 SOW ILM05.X Table 5.25.

6.6.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form VII-IN, Form XIII-IN, Form XIV-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form VII-IN, Form XII-IN, Form XIII-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

6.6.2 For soils sample analysis, verify that one solid LCS was prepared and analyzed for every group of soil/sediment samples in a SDG, or with each batch of soil samples digested, whichever is more frequent. The solid LCS must be prepared and analyzed using the identical preparation and analytical methods used for the soil/sediment samples. The solid LCS should be obtained from the USEPA, if available. If the LCS is unavailable from the USEPA, other USEPA QA samples or certified materials may be used.

Note: The SOW does not require the analysis of an aqueous LCS with water samples.

6.6.3 Solid LCS results must be within the control limits reported on Form VII-IN. If a result is outside the control limit, the laboratory is required to stop, correct the problem, recalibrate, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION section of the report. Use professional judgment to assess the data.

Note: The percent solids determination is not required for the solid LCS.

6.6.4 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the %Rs using the equation below:

LCS %R = (LCS *Found* / LCS *True* Value) X 100

LCS *Found* = concentration of analyte measured in LCS sample.

LCS *True* Value = certified concentration of analyte in LCS source.

6.6.5 Verify that at least one LCS has been analyzed for each group of samples of a similar matrix type (soil or water) and concentration, or for each SDG.

6.7 FIELD DUPLICATES

The objective of this section is to evaluate overall precision, including both field and laboratory precision. The results may have more variability than laboratory duplicates. Duplicate sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.13;
- Appendix B2 SOW ILM05.X Table 5.24.

6.7.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form XIV-IN, Field QA/QC Summary Form, instrument printouts, and raw data.

- *SOW ILM05.X*: Form I-IN, Form XIII-IN, Field QA/QC Summary Form, instrument printouts, and raw data.

6.7.2 Aqueous field sample duplicate analyses have control limits of ±20 RPD (or ±CRQL for sample results less than 5 times the CRQL).

6.7.3 Solid field sample duplicate analyses have Region 9 control limits of ±35 RPD (or ±2x the CRQL for sample results less than 5 times the CRQL). (Region 9 Modification, see Appendix A).

6.7.4 Examine the raw data and recalculate and document in worksheets all of the Field Duplicate RPDs using the equation below:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

S = Field sample result.

D = Field duplicate result.

6.7.5 Verify that all field duplicate QC samples have been properly identified in the report and on the spreadsheet by comparing with the Field QA/QC Summary Form. Label the first duplicate pair as "D1", the second pair as "D2" etc., on Table 1A next to the sample identification.

6.7.6 Do not qualify as estimated or rejected any analyte that exceeds the control limits for laboratory duplicates. Comment on the outlier and provide the RPD in the comment.

6.8 OVERALL ASSESSMENT

Review the entire data package and the data review results and use professional judgment to identify any inconsistencies, anomalies, additive effects of technical problems, impacts on data quality, or other concerns which should be brought to the attention of the data user. Determine whether there is any need to qualify data that were not qualified based on the criteria previously assessed. (Region 9 Modification, see Appendix A.)

6.8.1 Inspect Data package to verify the presence of the following review items:

- Complete data package, Field QA/QC Summary Form, preparation logs, calibration standard and spiking standard logs, instrument logs, instrument printouts, and raw data.

6.8.2 Sample Result Verification

Examine the raw data and recalculate and document in worksheets a minimum of 10% of the sample results using the equations below:

6.8.2.1 Water samples:

$$\text{Concentration } \mu g/L = \frac{\mu g\ Hg,\ curve}{Aliquot\ volume,\ mL} \times \frac{1000mL}{1L}$$

μg Hg, curve = Amount of mercury as determined from calibration curve.

Aliquot volume, mL = Sample aliquot volume in mL.

6.8.2.2 Soil samples:

$$\text{Concentration (dry weight) mg/kg} = \frac{C}{W \times S} \times 0.1L \times DF$$

C = Instrument result in (μg/L).

W = Wet sample weight (g).

S = Percent solids result/100.

DF = Dilution factor (as required).

6.8.2.3 Percent solids calculation for soil samples:

Percent Solids = (Sample dry weight / Sample wet weight) X 100

6.8.3 Verify that all results on the spreadsheet (Table 1A) were transcribed correctly from the Form 1s. Be sure all "U" and "B" qualifiers have been transposed to the spreadsheet correctly and that "B" flags have been changed to "L" flags.

6.8.4 Verify that preparation blank values from Form 3 are reported on Table 1A.

6.8.5 Verify that every sample has been analyzed, and results that exceed the calibration range have been diluted (but not over-diluted) and reanalyzed. If not, qualify such results as estimated and comment in CLP PO ACTION.

6.8.6 If any values have been incorrectly recorded, especially those that will cause the qualification of data, notify the laboratory to have the appropriate forms regenerated and resubmitted.

6.8.7 Do not make any corrections on any forms without first checking with the laboratory. Document all communication on a TRL. In general, have the laboratory resubmit any form or page of raw data that requires correction.

6.8.8 When corrected forms and raw data are received by the reviewer, strike each page to be replaced with a single line, mark the page "Replaced," and initial and date. Also, date and initial the replacement page.

6.8.9 Verify the values for all MDLs and CRQLs are transferred from the appropriate Forms to the Table 1A. Form 9 (Form 10 for ILM04.X) to the spreadsheet.

6.8.9.1 Verify that all non-detected results listed on Form 1s and Table 1A have the same value as the IDL, MDL, and CRQL values listed on the appropriate forms.

- If the listed non-detected results (U) Form 1s do not agree with the values listed on Form 9, contact the laboratory via TRL to explain the discrepancy.

- Do not make any corrections on Form 1s without first contacting the laboratory via TRL.

6.8.9.2 Examine the dates on the Form 9s to be sure they are current. MDL studies are to be performed quarterly, and if they are not, this should be noted for CLP PO ACTION. Use professional judgment to determine the effect on day quality.

6.8.10 Examine Form 12 (Form 13 for ILM04.X) and the raw data to determine whether the correct volumes for waters or weights for soils have been used. Refer to the SOW (Exhibit D) for guidance. Note any irregularities in ADDITIONAL COMMENTS or CLP PO ACTION.

6.8.11 Examine Form 13 (Form 14 for ILM04.X) to see if the following SOW protocols have been followed (refer to the SOW for further details).

6.8.11.1 Verify that the correct number of calibration standards and calibration verification standards have been analyzed before the environmental samples: these include the calibration standards, ICV/ICB, CRI, and the first CCV and CCB. The LCS and PB are considered samples, and must be run after the first CCV/CCB. If this sample order sequence is not followed, include a comment under CLP PO ACTION.

6.8.11.2 Verify that the CCVs/CCBs were analyzed at the proper frequency during each analytical run. Use professional judgment to determine whether to comment or estimate the associated sample data.

6.8.11.3 Verify that a PB, a LCS, a matrix spike, and a laboratory duplicate were analyzed for each matrix. Verify that all analyses of preparation blanks and LCSs are reported on the appropriate forms. (Note: aqueous LCS not required for aqueous samples.) Use professional judgment to determine whether to comment or estimate the associated sample data.

6.8.11.4 Verify the dates and times of analysis on the Run Logs against the raw data. Use professional judgment to determine whether to comment or estimate the associated sample data.

6.8.12 If solid samples are included in the SDG, verify calculations for percent solids in the raw data.

6.8.12.1 Verify that the samples were dried for a minimum of 12 hours, but not more than 24 hours in a drying oven maintained at 103-105°C or that there is documentation showing that constant weight was attained.

6.8.12.2 Verify that percent solids values have been transcribed correctly onto the Form 1s and the spreadsheet.

6.8.12.3 If any errors have occurred, contact the laboratory via TRL to confirm the error and to have all relevant forms regenerated.

6.8.12.4 Examine the raw data for sample, standards, and spike preparation and digestion to verify that the correct weights and volumes were used and transcribed to Form 12 (Form 13 for ILM04.X); and that spike levels and calculations are correct. Check for current stock standard true value and traceability certificate.

6.8.12.5 Examine the chain of custody forms to verify that the samples were received intact, and to verify sample type, sample preservation, sample location, laboratory QC sample, dates of sample collection and receipt by the laboratory, and sampler and laboratory receipt signatures. If any of the information is incorrect or missing, including signatures, comment that the effect on the legal defensibility of the data is unknown in ADDITIONAL COMMENTS.

7 CYANIDE DATA REVIEW

This section contains data validation requirements and procedures for cyanide analysis by sample distillation followed by spectrophotometric analysis. Validation criteria and appropriate actions for each SOW are provided in Appendices B1 and B2. Worksheets for each SOW are provided in Appendices C1 and C2.

7.1 PRESERVATION AND HOLDING TIMES

The objective of this section is to verify the validity of the results based on the preservation and holding time of the samples from the time of collection to the time of analysis. Holding time and preservation criteria and actions are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.15;
- Appendix B2 SOW ILM05.X Table 5.26.

7.1.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form XIII-IN, EPA Traffic Report and/or Chain of Custody (COC) record form(s), Form DC-1, Field QA/QC Summary Form, sample distillation log, raw data, and sample delivery group (SDG) Narrative.

- *SOW ILM05.X*: Form IA-IN, Form IB-IN, Form XII-IV, Form XIII-IN, EPA Traffic Report/Chain of Custody (TR/COC) record form(s), Form DC-1, Field QA/QC Summary Form, sample distillation log, raw data, and SDG Narrative.

7.1.2 Preservation Criteria:

7.1.2.1 Aqueous Samples

- pH: Examine Form DC-1 and the sample distillation log to verify water samples were preserved to a pH greater than 12 with sodium hydroxide.

- Temperature: Examine DC-1 and internal COC documents to confirm the cooler temperature upon receipt at the laboratory is 4 ± 2 °C.

- Oxidizing agents and sulfide: Examine sample preparation and distillation logs to confirm the samples were screened and treated for oxidizing agents and sulfides.

7.1.2.2 Soil Samples

- Temperature: Examine DC-1 and internal COC documents to confirm the cooler temperature upon receipt at the laboratory is 4±2°C.

7.1.3 Holding Time Criteria

7.1.3.1 Verify and document in worksheets all technical holding times by comparing the dates of collection for all samples as reported on the EPA Traffic Reports/Chain of Custody (TR/COC) record form(s) to the dates of analysis from the instrument run logs and the raw data.

7.1.3.2 Technical holding time: If the sample holding time for water is less than or equal to 14 days and preserved with sodium hydroxide, no action is required.

7.1.3.3 If the sample holding time for water or soil is greater than 28 days, qualify results as rejected and comment in CLP PO ACTION section. Note that the Region 9 advisory limit for the rejection of data is two times the technical holding time for analyses that grossly exceed the holding time and are or are not properly preserved. (Region 9 Modification, see Appendix A.)

7.2 INSTRUMENT CALIBRATION

The objective of this section is to verify that the method requirements for satisfactory instrument calibration are followed and that the instrument is capable of producing acceptable quantitative data. Initial calibration verification (ICV) demonstrates that the instrument performance is acceptable at the beginning of the analytical run. Continuing calibration verification (CCV) demonstrates that the initial calibration continues to be valid by checking instrument performance at regular intervals. Initial calibration, initial calibration verification, continuing calibration verification and contract required detection limit (CRDL)/contract required quantitation limit (CRQL) verification (CRI) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.16;
- Appendix B2 SOW ILM05.X Tables 5.27 and 5.28.

7.2.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form IIA-IN, Form IIB-IN, Form XIII-IN, preparation logs, distillation log, calibration standard logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form IIA-IN, Form IIB-IN, Form XII-IN, Form XIII-IN, preparation logs, distillation log, calibration standard logs, instrument logs, instrument printouts, and raw data.

7.2.2 Initial Instrument Calibration

7.2.2.1 Verify that the instrument is successfully calibrated daily or once every 24 hours and each time the instrument is set up. Verify that a blank and at least three standards have been used to establish the standard curve and that sample results are within the linear working range of the instrument. One calibration standard must be at the CRQL concentration. Verify that the average of two replicate exposures (minimum) is used for all results. The calibration correlation coefficient must be greater than or equal to 0.995. Inspect the raw data to ensure that the calibration date and time are included. An analytical wavelength between 570 and 580 nanometers used for calibration must be used throughout the analytical sequence.

7.2.3 Initial and Continuing Calibration Verification

7.2.3.1 If an ICV or CCV %R is outside of the control limits, determine if the laboratory stopped the analysis, corrected the problem, recalibrated, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION.

7.2.3.2 From the raw data, recalculate and document in worksheets (Appendix C) a minimum of 10% of the calibration verification percent recoveries (%Rs) using the equations below:

$$\text{ICV \%R} = (\textit{Found} \text{ value} / \textit{True} \text{ value}) \times 100$$

$$\text{CCV \%R} = (\textit{Found} \text{ value} / \textit{True} \text{ value}) \times 100$$

7.2.3.3 Verify that an ICV standard, prepared from a USEPA or certified stock solution but from a different stock source or lot than used for calibration, is analyzed at the wavelengths used for sample analysis and reported immediately after ICP system calibration, but before the analysis of any analytical samples. Examine all ICV %Rs and compare to criteria in Appendix B.

7.2.3.4 Verify that CCV standards are analyzed and reported at the beginning and end of the analytical run, after an ICV, and at a frequency of 10% of samples analyzed or every two hours during an analytical run, whichever is more frequent. Verify that the CCV standards are prepared from a different source or lot than used for the ICV and are at or near the mid-range of the calibration curve.

7.2.3.5 The efficiency of the distillation must be verified by distilling at least one mid-level calibration standard. The distilled standard result must agree within ±15 percent of the non-distilled standard concentration. This mid-level standard shall be prepared at least once for each distillation method used to prepare samples. The mid-level standard shall be analyzed immediately after the first CCV/CCB analysis.

7.2.4 Contract Required Quantitation Limit (CRQL) Verification

7.2.4.1 Verify a CRQL verification standard (CRI) was analyzed to demonstrate that the established calibration is linear and valid near the CRQL.

7.2.4.2 Recalculate and document in worksheets a minimum of 10% of the CRI verification %Rs using the equation below:

$$\text{CRI \%R} = (\text{Found value} / \text{True value}) \times 100$$

7.2.4.3 There are no EPA-established limits for CRA in SOW ILM04.X. Use the Region 9 advisory limits of 65-135%R. (Region 9 Modification, see Appendix A.2) Compare to criteria in Appendix B.

- Verify that the CRA is analyzed after the ICV and at the end of the run or a minimum of 2 times per 8 hour shift, whichever is more frequent. If the frequency is not met, note in CLP PO ATTENTION but do not qualify any results.

- The CRA concentration should be two times the CRDL specified for the target analyte, or two times the instrument detection limit (IDL), whichever is greater.

7.2.4.4 There are EPA established limits for CRI in SOW ILM05.X. Compare CRI results to criteria and actions in Appendix B.

- Verify that a CRI is analyzed immediately after the first CCV/CCB analysis, every 20 analytical samples, and at the end of the run.

- If calibration criteria are grossly exceeded, include a comment in the CLP PO ACTION section of the report.

7.3 BLANKS

The objective of this section is to assess the results of blank analyses to determine the existence and magnitude of contamination from laboratory or field activities. The criteria for evaluation of blanks apply to any blank associated with the samples. Initial calibration blank (ICB), continuing calibration blank (CCB), preparation blank (PB), field blank (FB), and equipment blank (EB) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.17;
- Appendix B2 SOW ILM05.X Table 5.29.

7.3.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form III-IN, Form XIII-IN, Form XIV-IN, Field QA/QC Summary Form, sample preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, Field QA/QC Summary Form, sample preparation logs, instrument logs, instrument printouts, and raw data.

7.3.2 No contaminants should be found in any blank. If problems exist with any blank, evaluate all sample data associated with the blank to determine if the problem is system wide or an isolated occurrence.

7.3.3 Calibration Blanks

7.3.3.1 Verify that an ICB is analyzed after the ICV. Verify that if the absolute value of an analyte in an ICB is greater than the CRQL, the analysis is terminated, the problem corrected, and the associated samples reanalyzed. If the ICB does not meet criteria, apply action to all samples in the analytical run. If the ICB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

7.3.3.2 Verify that a CCB is analyzed after every CCV. If the absolute value of an analyte in a CCB is greater than the CRQL, verify that the analysis is terminated, the problem corrected, and the associated samples reanalyzed. Verify that the CCB is analyzed at a frequency of every ten samples or every two hours during the run, whichever is more frequent. If CCBs do not meet criteria, apply actions to all samples associated with the CCBs out of criteria. If the CCB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the

associated sample data and include a comment in the CLP PO ACTION section of the report.

7.3.4 Preparation Blank

7.3.4.1 Verify that a PB was distilled and analyzed for each matrix per SDG or batch of prepared samples, whichever is more frequent. If the absolute value of any analyte in the PB is less than or equal to the CRQL, no action is required. If the PB does not meet criteria, apply action to all samples prepared in the same preparation batch. If the PB was not analyzed at the correct frequency, use professional judgment to determine whether to comment or estimate the associated sample data and include a comment in the CLP PO ACTION section of the report.

7.3.4.2 Verify that analyte concentrations in the PB are less than the CRQL. Confirm that all samples associated with a contaminated PB contain the contaminated analyte at a concentration greater than 5 times the PB concentration. Verify that the all samples were re-digested and reanalyzed (except for identified field blanks). Note that the laboratory is not to correct any sample concentration by subtracting the blank value. Include a comment in the CLP PO ACTION section of the report if the laboratory failed to re-digest and reanalyze the affected samples.

- Region 9 Modification: For PB contamination, qualify associated sample results greater than the IDL but less than 5 times the amount in the blank as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.3).

7.3.4.3 If the analyte concentration in the PB is less than the negative CRQL value, all samples associated with the contaminated PB with contaminating analyte concentrations less than 5 times the CRQL should have been re-digested and reanalyzed. Include a comment in the CLP PO ACTION section of the report if the laboratory failed to re-digest and reanalyze the affected samples.

7.3.5 Field Blanks

7.3.5.1 Verify all field QC blank samples listed on the Field QA/QC Summary Form and identified on the spreadsheet. Check the Field QA/QC Summary Form to verify that all field QC samples have been properly identified in the report, and on the spreadsheet. Use professional judgment to determine whether to comment or estimate the associated sample data.

7.3.6 If the absolute value of the concentration of an analyte in a blank is greater than the CRQL, apply the following guidelines.

7.3.6.1 For PB contamination, qualify associated sample results greater than the MDL but less than 5 times the amount in the blank as non-detected and estimated (UJ). Include a comment under CLP PO ACTION (Region 9 Modification, see Appendix A.)

7.3.6.2 For ICB or CCB contamination, determine all sample numbers whose results were used from the analytical run between the last acceptable ICB or CCB and the next acceptable CCB. Associated sample results greater than the MDL but less than 5 times the amount in the blank are qualified as non-detected and estimated (UJ). Include a comment under CLP PO ACTION. (Region 9 Modification, see Appendix A.)

7.3.6.3 For any laboratory blank (PB, ICB, CCB) with a negative result whose absolute value is greater than the CRQL, use professional judgment to determine the effect on data quality, i.e., low bias.

7.3.6.4 For FB or EB contamination, identify associated samples based on the same date of collection as the field QC. Associated sample results greater than the MDL but less than 5 times the amount in the blank are qualified as non-detected and estimated (UJ). Include a comment under SAMPLING ISSUES (Region 9 Modification, see Appendix A.)

7.3.7 For sample results less than the CRQL and affected by blank contamination, apply the following guidelines.

7.3.7.1 For undiluted samples, increase associated sample results less than the CRQL to the CRQL and qualify as non-detected (UJ).

7.3.7.2 For diluted samples, increase associated sample results less than the CRQL to the product of the CRQL multiplied by the sample dilution factor (CRQL x df) and qualify as non-detected (UJ).

7.3.7.3 If an ICB or CCB analyte result is greater than the CRQL, and the analysis was not terminated and the affected samples were not reanalyzed, raise the CRQL of the affected samples to the concentration found in the ICB or CCB. Report non-detects and results greater than or equal to the MDL, but less than or equal to the CRQL as [CRQL]U. Include a comment in the CLP PO ACTION section of the report.

7.4 SPIKE SAMPLE ANALYSIS

The objective of this section is to provide information about the effect of each sample matrix on the sample preparation procedures and the analytical method. Matrix spike (MS) criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.18;
- Appendix B2 SOW ILM05.X Table 5.30.

7.4.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Forms V-IN (Parts A and B), Form XIII-IN, Form XIV- IN, Field QA/QC Summary Form, standard preparation logs, distillation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form I-IN (for QC sample), Form VA-IN (Parts A and B), Form XII-IN, Form XIII-IN, Field QA/QC Summary Form, standard preparation logs, distillation logs, instrument logs, instrument printouts, and raw data.

7.4.2 Examine the raw data and recalculate and document in worksheets the MS %Rs using the equation below:

$$\text{Matrix spike \%R} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spiked sample result from the original sample.

SR = Sample result. Use SR = 0 when sample concentration is less than the MDL.

SA = Spike added.

7.4.3 Use the results of the sample designated as the "original sample" to perform re-calculations of the matrix spike. Do not use the average of the "original sample" and the laboratory duplicate sample results for the purpose of determining the matrix spike %R.

7.4.4 Use the post-distillation spike analysis result in conjunction with the pre-distillation spike analysis result to qualify sample results. Comment about post-distillation spike results in the matrix spike section of the inorganic report.

7.4.5 Do not evaluate matrix spike recovery in instances where a sample result exceeds four times the spiking concentration.

7.4.6 Verify that at least one matrix spike sample analysis has been performed on each group of samples of a similar matrix type and concentration, or for each SDG.

7.4.7 Check that the QC sample designated on the chain of custody is used for matrix spike analysis. Comment in "Additional Comments" if a different sample than the one designated on the COC is used

7.4.8 Verify that the spike was added before sample distillation, and that the correct spike concentrations were used in spiking the matrix spike sample by examining the distillation logs and referring to the SOW.

7.4.9 Qualify samples designated as background samples for MS outliers since they reflect the matrix of the environmental samples.

7.4.10 Do not qualify field blank, equipment blank, or performance evaluation (PE) sample data due to MS outliers since these matrices are not representative of environmental samples. If an improper sample, i.e., field or equipment blank, was used for matrix spike analysis and there was no QC sample listed on the COC, comment in SAMPLING and include a comment in Section III, Validity and Comments, indicating that accuracy and precision parameters could not be evaluated due to improperly performed QC.

7.4.11 If inadequate QC has been performed (i.e., due to insufficient sample, etc.), use professional judgment to determine the effect on the data quality, and include a comment in Section III, Validity and Comments.

7.4.12 If incorrect concentrations were used, use professional judgment to determine the effect on the data quality, and include a comment in Section III, Validity and Comments.

7.4.13 If cyanide did not meet matrix spike recovery criterion, check the post-distillation spike sample %Rs on Form 5B. Whether the cyanide results do or do not achieve 75-125 %R, comment on the post-distillation spike recovery results in the matrix spike section of the inorganic template.

7.4.14 If incorrect concentrations were used, use professional judgment to determine the effect on the data quality, and include a comment in CLP PO ACTION section.

7.4.15 A post-distillation spike must be performed if the cyanide result does not meet the specified MS %R criterion. An aliquot of the remaining unspiked sample shall be spiked at two times (2x) the indigenous level or 2x the CRQL, whichever is greater. If a required post-distillation spike was not performed, comment in the CLP PO ACTION section of the report.

7.5 DUPLICATE SAMPLE ANALYSIS

The objective of this section is to verify that the laboratory demonstrated acceptable method precision at the time of sample analysis. Duplicate sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.19;
- Appendix B2 SOW ILM05.X Table 5.31.

7.5.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form VI-IN, Form XIII-IN, Form XIV-IN, QA/QC Summary Form, instrument logs, instrument printouts, and raw data.

  If inadequate QC has been performed, use professional judgment to determine the effect on the data quality, and include a comment for CLP PO ATTENTION and in Section III, Validity and Comments.

- *SOW ILM05.X*: Form I-IN, Form VI-IN, Form XII-IN, Form XIII-IN, QA/QC Summary Form, instrument logs, instrument printouts, and raw data.

  If inadequate QC has been performed, use professional judgment to determine the effect on the data quality, and include a comment in the CLP PO ACTION section of the report.

7.5.2 Examine the raw data and recalculate and document in worksheets 10% of the RPDs using the equation below:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

S = Sample result (original).

D = Duplicate result.

7.5.3 Do not report an RPD of 200 when one result is non-detected and the other result is detected greater than or equal to the CRQL. State in the duplicate sample comment that the analyte was reported at a given concentration in one duplicate analysis, but not detected in the other. (Region 9 Modification, see Appendix A.)

7.5.4 Verify that duplicate sample analyses are performed for Percent Solids.

7.5.5 Verify that at least one duplicate sample analysis has been performed on each group of samples of a similar matrix type and concentration, or for each SDG. Check to make sure that the QC sample designated on the chain of custody was used. Duplicate analysis does not need to be performed on samples labeled as rinsates in an SDG of soil samples.

7.5.6 Qualify Background samples for duplicate outliers since the background sample matrix reflects the matrix of the environmental samples.

7.5.7 Do not qualify results for field blank, equipment blank, or PE sample data for duplicate outliers since the matrix of the blanks is not similar to the environmental samples. If a field blank was used for duplicate sample analysis, note such use of a field blank in CLP PO ACTION and include a statement in Section III, Validity and Comments, indicating that this precision parameter could not be evaluated due to improperly performed QC.

7.6 LABORATORY CONTROL SAMPLE (LCS)

The objective of this section is to monitor the laboratory's overall performance at each step of the analysis, including sample preparation. Laboratory control sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.20;
- Appendix B2 SOW ILM05.X Table 5.32.

7.6.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form VII-IN, Form XIII-IN, Form XIV-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

- *SOW ILM05.X*: Form VII-IN, Form XII-IN, Form XIII-IN, standard preparation logs, instrument logs, instrument printouts, and raw data.

7.6.2 For soils sample analysis, verify that one solid LCS was prepared and analyzed for every group of soil/sediment samples in a SDG, or with each batch of soil samples distilled, whichever is more frequent. The solid LCS must be prepared and analyzed using the identical preparation and analytical methods used for the soil/sediment samples. The solid LCS should be obtained from the USEPA, if available. If the LCS is unavailable from the USEPA, other USEPA QA samples or certified materials may be used.

7.6.3 Solid LCS results must be within the control limits reported on Form VII-IN. If a result is outside the control limit, the laboratory is required to stop, correct the problem, recalibrate, and reanalyze the affected samples. If this is not done, include a comment in CLP PO ACTION section of the report. Use professional judgment to assess the data.

Note: The percent solids determination is not required for the solid LCS.

7.6.4 Examine the raw data and recalculate and document in worksheets a minimum of 10% of the %Rs using the equation below:

LCS %R = (LCS *Found* / LCS *True* Value) X 100

LCS *Found* = concentration of analyte measured in LCS sample.

LCS *True* Value = certified concentration of analyte in LCS source.

7.6.5 Verify that at least one LCS has been analyzed for each group of samples of a similar matrix type (soil) and concentration, or for each SDG.

7.7 FIELD DUPLICATES

The objective of this section is to evaluate overall precision, including both field and laboratory precision. The results may have more variability than laboratory duplicates. Duplicate sample criteria are contained in the appendices as follows:

- Appendix B1 SOW ILM04.X Table 4.19;
- Appendix B2 SOW ILM05.X Table 5.31.

7.7.1 Inspect Data Package to verify the presence of the following review items:

- *SOW ILM04.X*: Form I-IN, Form XIV-IN, Field QA/QC Summary Form, instrument printouts, and raw data.
- *SOW ILM05.X*: Form I-IN, Form XIII-IN, Field QA/QC Summary Form, instrument printouts, and raw data.

7.7.2 Aqueous field sample duplicate analyses have control limits of ±20 RPD (or ±CRQL for sample results less than 5 times the CRQL).

7.7.3 Solid field sample duplicate analyses have Region 9 control limits of ±35 RPD (or ±2 times the CRQL for sample results less than 5x the CRQL). (Region 9 Modification, see Appendix A).

7.7.4 Examine the raw data and recalculate and document in worksheets all of the Field Duplicate RPDs using the equation below:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

S = Field sample result.

D = Field duplicate result.

7.7.5 Verify that all field duplicate QC samples have been properly identified in the report and on the spreadsheet by comparing with the Field QA/QC Summary Form. Label the first duplicate pair as "D1", the second pair as "D2", etc., on Table 1A next to the sample identification.

7.7.6 Do not qualify as estimated or rejected any analyte that exceeds the control limits for laboratory duplicates. Comment on the outlier and provide the RPD in the comment.

7.8 OVERALL ASSESSMENT

Review the entire data package and the data review results and use professional judgment to identify any inconsistencies, anomalies, additive effects of technical problems, impacts on data quality, or other concerns which should be brought to the attention of the data user. Determine whether there is any need to qualify data that were not qualified based on the criteria previously assessed. (Region 9 Modification, see Appendix A.5).

7.8.1 Inspect Data package to verify the presence of the following review items:

- Complete data package, Field QA/QC Summary Form, preparation logs, calibration standard and spiking standard logs, instrument logs, instrument printouts, and raw data.

7.8.2 Sample Result Verification

Examine the raw data and recalculate and document in worksheets a minimum of 10% of the sample results using the equations below:

Note: There are several cyanide analysis modes: manual, semi-automatic, and Midi distillation. Equations for the most common mode of analysis are presented below. Equations for the other modes may be found in the National Functional Guidelines (NFG).

7.8.2.1 Water samples by Midi distillation:

$$\text{Concentration } \mu\text{g/L} = \frac{A \times D \times F}{B}$$

A = μg/L cyanide in sample determined from regression curve.

B = Volume of sample for distillation (0.050 L).

D = Dilution factor, if required.

F = Sample receiving solution volume (0.050 L).

7.8.2.2 Soil samples by Midi distillation:

$$\text{Concentration (dry weight) mg/kg} = \frac{A \times D \times F}{B \times E}$$

A = μg/L cyanide in sample determined from regression curve.

B = Wet weight of original sample.

D = Dilution factor, if required.

E = Percent solids result/100.

F = Sample receiving solution volume (0.050 L).

7.8.2.3 Percent solids calculation for soil samples:

Percent Solids = (Sample *dry* weight / Sample *wet* weight) X 100

7.8.3 Verify that all results on the spreadsheet (Table 1A) were transcribed correctly from the Form 1s. Be sure all "U" and "B" qualifiers have been transposed to the spreadsheet correctly and that "B" flags have been changed to "L" flags.

7.8.4 Verify that preparation blank values from Form 3 are reported on Table 1A.

7.8.5 Verify that every sample has been analyzed, and that results that exceed the calibration range have been diluted (but not over-diluted) and reanalyzed. If not, qualify such results as estimated and comment in CLP PO ACTION.

7.8.6 If any values have been incorrectly recorded, especially those that will cause the qualification of data, notify the laboratory to have the appropriate forms regenerated and resubmitted.

7.8.7 Do not make any corrections on any forms without first checking with the laboratory. Document all communication on a TRL. In general, have the laboratory resubmit any form or page of raw data that requires correction.

7.8.8 When corrected forms and raw data are received by the reviewer, strike each page to be replaced with a single line, mark the page "Replaced," and initial and date. Also, date and initial the replacement page.

7.8.9 Verify the values for all MDLs and CRQLs are transferred from the appropriate Forms to the Table 1A. Form 9 (Form 10 for ILM04.X) to the spreadsheet.

7.8.9.1 Verify that all non-detected results listed on Form 1s and Table 1A have the same value as the MDL, and CRQL values listed on the appropriate forms.

- If the listed non-detected results (U) Form 1s do not agree with the values listed on Form 9, contact the laboratory via TRL to explain the discrepancy.

- Do not make any corrections on Form 1s without first contacting the laboratory via TRL.

7.8.9.2 Examine the dates on the Form 9s to be sure they are current. MDL studies are to be performed quarterly, and if they are not, this should be noted for CLP PO ACTION. Use professional judgment to determine the effect on day quality.

7.8.10 Examine Form 12 (Form 13 for ILM04.X) and the raw data to determine whether the correct volumes for waters or weights for soils have been used. Refer to the SOW (Exhibit D) for guidance. Note any irregularities in ADDITIONAL COMMENTS or CLP PO ACTION.

7.8.11 Examine Form 13 (Form 14 for ILM04.X) to see if the following SOW protocols have been followed (refer to the SOW for further details).

7.8.11.1 Verify that the correct number of calibration standards and calibration verification standards have been analyzed before the environmental samples: these include the calibration standards, ICV/ICB, CRI, ICSA/ICSAB, and the first CCV and CCB. The LCS and PB are considered samples, and must be run after the first CCV/CCB. If this sample order sequence is not followed, include a comment under CLP PO ACTION.

7.8.11.2 Verify that the CCVs/CCBs were analyzed at the proper frequency during each analytical run. Use professional judgment to determine whether to comment or estimate the associated sample data.

7.8.11.3 Verify that a mid-level calibration standard, a PB, a matrix spike, and laboratory duplicate were distilled and analyzed for each matrix (LCS for solids only). Verify that all analyses of preparation blanks and LCS are reported on the appropriate forms. Use professional judgment to determine whether to comment or estimate the associated sample data.

7.8.11.4 Verify the dates and times of analysis on the Run Logs against the raw data. Use professional judgment to determine whether to comment or estimate the associated sample data.

7.8.12 If solid samples are included in the SDG, verify calculations for percent solids in the raw data.

7.8.12.1 Verify that the samples were dried for a minimum of 12 hours, but not more than 24 hours in a drying oven maintained at 103-105°C or that there is documentation showing that constant weight was attained.

7.8.12.2 Verify that percent solids values have been transcribed correctly onto the Form 1s and the spreadsheet.

7.8.12.3 If any errors have occurred, contact the laboratory via TRL to confirm the error and to have all relevant forms regenerated.

7.8.12.4 Examine the raw data for sample, standards, and spike preparation and digestion to verify that the correct weights and volumes were used and transcribed to Form 12 (Form 13 for ILM04.X); and that spike levels and calculations are correct. Check for current stock standard true value and traceability certificate. (Region 9 Modification, see Section X.0).

7.8.12.5 Examine the chain of custody forms to verify that the samples were received intact, and to verify sample type, sample preservation, sample location, laboratory QC sample, dates of sample collection and receipt by the laboratory, and sampler and laboratory receipt signatures. If any of the information is incorrect or missing, including signatures, comment that the effect on the legal defensibility of the data is unknown in ADDITIONAL COMMENTS.

APPENDIX A
REGIONAL MODIFICATIONS TO THE FUNCTIONAL GUIDELINES

1. Region 9 criterion for holding time rejection (R) for metals is two times the technical holding time for unpreserved samples. The Functional Guidelines specify using professional judgment.

2. Region 9 requires a comment in the Sampling Issues section when sample temperature for a water sample is >6°C but ≤20°C and a pH of < 2 for metals analyses (or a pH of > 12 for cyanide analysis). The National Functional Guidelines do not include this criterion.

3. Region 9 requires estimation (J or UJ) of sample results when the temperature for a water sample is >20°C and a pH of < 2 for metals analyses (pH > 12 for cyanide analysis) and comment in the Sampling Issues section. The National Functional Guidelines do not include this criterion.

4. Region 9 requires rejection (R) of non-detected results and estimation (J-) of detected results when the temperature for a water sample is >6°C and ≤20°C and a pH of ≥ 2 for metals analyses (pH ≤ 12 for cyanide analysis) and comment in the Sampling Issues section. The National Functional Guidelines do not include this criterion.

5. Region 9 requires rejection (R) of results for water samples with temperatures >20°C and a pH of ≥ 2 for metals analyses (pH ≤ 12 for cyanide) and a comment in the Sampling Issues section. This differs from the National Functional Guidelines criteria of using professional judgment.

6. Region 9 limits for the CRDL standard are 65-135 %R. The Functional Guidelines do not specify acceptance limits for the CRDL standard. (SOW ILM04.X only.)

7. Region 9 acceptance threshold for preparation, field, and equipment blank contamination is five times the blank contamination result. The Functional Guidelines specifies an acceptance threshold of ten times the blank contamination result.

8. Region 9 frequency for the examination of the %R for post-digest analytical spike samples is for a minimum of 10% of the results to be recalculated and documented in worksheets. The Functional Guidelines requires that the %R of each post digest analytical spike be examined.

9. Region 9 does not require reporting of a 200 RPD result for duplicate sample results where the sample result is greater than or equal to the CRQL and the duplicate sample result is non-detected. Region 9 requires stating that one sample was detected at or above the CRQL and not detected in the duplicate sample. The National Functional Guidelines do not include this criterion.

10. Region 9 does not require an Inorganic Regional Data Assessment (IRDA) form to be included in the data validation report. The Functional Guidelines specifies the inclusion of an IRDA in a data validation reports.

11. Region 9 does not require an Overall Assessment section as part of the data validation report. This differs from the Functional Guidelines requirement that a brief narrative describing concerns found during the overall assessment be included

APPENDIX B1
SOW ILM04 X DATA REVIEW CRITERIA TABLES

Table 4.1
ILM04.X PRESERVATION & HOLDING TIME ACTIONS FOR ICP-AES ANALYSIS

Soil Preservation Requirement: 4°C (±2°C)

Water Preservation Requirements: 4°C (±2°C) and pH less than 2

Sample Holding Time: 180 days (maximum)

Preservation and Holding Time Result	Action for Samples
Water samples received at pH = 2 (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at pH > 2 (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ). Comment in Sampling Issues.
Water samples received at >6.0°but ≤20°C with pH<2 (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at >20°C with pH<2 (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ). Comment in Sampling Issues.
Water samples received at >6.0°but ≤20°C and pH>2 (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ). Comment in Sampling Issues.
Water samples received at >20°C and pH >2 (Region 9 Advisory Limit)	Qualify all associated results as rejected (R). Comment in Sampling Issues.
Soil samples received at >6.0°but ≤20°C (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Soil samples received at >20°C(Region 9 Advisory Limit)	Qualify results ≥MDL as estimated (J). Qualify results <MDL as estimated (UJ). Comment in Sampling Issues.
Technical Holding Time for Soil or Water >180 days but ≤360	Qualify results ≥MDL as estimated (J). Qualify results <MDL as estimated (UJ).
Technical Holding Time for Soil or Water >360 days (Region 9 Advisory Limit)	Qualify all associated results as rejected (R). Comment in CLP PO Action section.

Table 4.2
ILM04.X CALIBRATION ACTIONS FOR ICP-AES ANALYSIS

Instrument Calibration Requirement: instrument calibrated once every 24 hours using a calibration blank and one calibration standard (minimum)

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Percent Recovery Control Limit: 90 – 110%

Contract Required Detection Limit (CRDL) ICP Standard Solution (CRI) Percent Recovery Control Limit: 65 – 135% (Region 9 advisory limit)

Calibration Result	Action for Samples
Calibration not performed every 24 hours	Qualify all results as unusable (R).
If the minimum number of standards were not used to establish instrument standard curve	Qualify all results as unusable (R)
ICV/CCV %R < 75%	Qualify all associated results as rejected (R).
ICV/CCV %R between 75-89%	Qualify results > IDL estimated (J). Qualify non-detects estimated (UJ). Indicate possible low bias in comment.
ICV/CCV %R between 111-125%	Qualify results > IDL estimated (J) and indicate possible high bias in comment. Non-detects not qualified.
ICV/CCV %R > 125%	Qualify > IDL as rejected (R). Non-detects not qualified.
CRI %R < 65% or > 135% and sample results near CRDL	Qualify results near CRDL as estimated (J). Comment in Validity and Comments section.
CRI %R < 65% or > 135% and sample results > 2 x CRDL	Comment in Additional Comments section only.

Table 4.3
ILM04.X BLANK ACTIONS FOR ICP-AES ANALYSIS

Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank (PB), Equipment Blank (EB), and Field Blank (FB) Requirements: analyte concentrations must not exceed CRDL values

ICB and CCB Requirement: ICB and CCB must be analyzed after each ICV and CCV

PB Requirement: one PB must be prepared and analyzed for each SDG (minimum)

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	>IDL but ≤CRDL	≥IDL but ≤CRDL	Report CRDL value with a (U)
ICB/CCB	>CRDL	≥IDL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action
ICB/CCB	|-blank result| >CRDL	≥IDL but <5x blank result absolute value	Qualify affected results as estimated (UJ) and comment for CLP PO action
PB	>IDL but ≤CRDL	≥IDL but ≤CRDL	Report CRDL value with a (U)
PB	>CRDL	>CRDL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action
PB	|-blank result| >CRDL	≥IDL but <5x blank result absolute value	Qualify affected results as estimated (UJ) and comment for CLP PO action
FB / EB	>CRDL	≥IDL but ≤CRDL and <5x blank result	Estimate and report at FB/EB value with a (UJ) and comment in Additional Comments section
		>CRDL but <5x blank result	Qualify results as estimated (UJ) and comment in Additional Comments section

Table 4.4
ILM04.X INTERFERENCE CHECK SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Interference Check Sample AB (ICSAB) Percent Recovery Control Limit: 80 – 120%

Interference Check Sample Result	Action for Samples
ICSAB %R <50%	Qualify results as rejected (R).
ICSAB %R ≥50% but ≤79%	Qualify results >IDL (or MDL) estimated (J). Qualify results <IDL (or MDL) estimated (UJ). Indicate a possible low bias in comment.
ICSAB %R >120%	Qualify results >IDL (or MDL) as estimated (J). Indicate a possible high bias in comment. Non-detects not qualified.

Table 4.5
ILM04.X MATRIX SPIKE SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Matrix Spike Sample Percent Recovery Control Limit: 75 – 125%

Spike Sample Result	Action for Samples
Matrix Spike %R <30%	Qualify non-detect results as rejected (R).
Matrix Spike %R between 30% - 74%	Qualify non-detect results as estimated (UJ).
Matrix Spike %R <75%	Qualify sample results >IDL (or MDL) as estimated (J). Indicate possible low bias in comment.
Matrix Spike %R >125%	Qualify sample results >IDL (or MDL) as estimated (J). Indicate a possible high bias in comment. Non-detects not qualified.

Note: If the original sample result exceeds four times (4x) the spiking concentration, no action is required

Table 4.6
ILM04.X DUPLICATE SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Duplicate Sample Relative Percent Difference (RPD) Control Limits:

Water: ±20 RPD or ±CRDL

Soil: ±35 RPD or ±2x CRDL (Region 9 limit)

Duplicate Sample Result	Action for Samples
Water: Both original and duplicate sample results ≥5x CRDL and RPD > ±20	Qualify results as estimated (J) or (UJ).
Water: If original or duplicate sample results <5x CRDL and absolute difference between sample and duplicate result > ±CRDL	Qualify results as estimated (J) or (UJ).
Soil: Both original and duplicate sample results ≥5x CRDL and RPD > ±35	Qualify results as estimated (J) or (UJ).
Soil: Both original and duplicate sample results <5x CRDL and absolute difference between sample and duplicate result > ±2x CRDL	Qualify results as estimated (J) or (UJ).

Note: When a result is detected at greater than or equal to the CRDL in one sample but non-detected in the duplicate sample, do not report a RPD of 200. Instead, indicate in the duplicate sample comment in the report that the analyte was reported at a given concentration in one sample, but not detected in the duplicate sample.

For field duplicate samples only: provide a comment for the field duplicate pair involved, but do not qualify any data.

Table 4.7
ILM04.X LABORATORY CONTROL SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Laboratory Control Sample (LCS) Percent Recovery Control Limits:

Aqueous: 80 – 120% (Aqueous LCS results for antimony and silver have no fixed control limits)

Soil: within limits established by supplier

LCS Result	Action for Samples
Aqueous LCS % R between <50%	Qualify sample results as rejected (R)
Aqueous LCS % R between 50% - 79%	Qualify sample results >IDL (or MDL) as estimated (J). Qualify sample results <IDL (or MDL) as estimated (UJ). Indicate possible low bias in comment.
Aqueous LCS %R >125%	Qualify sample results >IDL (or MDL) as estimated (J). Indicate a possible high bias in comment. Non-detects are not qualified.
Soil LCS recovery higher than control limits	Qualify sample results >IDL (or MDL) as estimated (J). Indicate a possible high bias in comment. Non-detects are not qualified.
Soil LCS recovery lower than control limits	Qualify sample results >IDL (or MDL) as estimated (J). Qualify sample results <IDL (or MDL) as estimated (UJ). Indicate a possible low bias in comment.
Soil LCS recovery <50% and no control limits are provided (Region 9 advisory limit)	Qualify sample results as rejected (R).

Table 4.8
ILM04.X SERIAL DILUTION SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Serial Dilution Percent Difference (%D) Control Limit: 10 %

Serial Dilution Result	Action for Samples
Sample result >50x IDL (or MDL) and %D > +10%	Qualify affected results as estimated (J) or (UJ). Indicate possible high bias in comment.
Sample result >50x IDL (or MDL) and %D < -10%	Qualify affected results as estimated (J) or (UJ). Indicate possible low bias in comment.

Table 4.9
ILM04.X PRESERVATION AND HOLDING TIME ACTIONS FOR MERCURY ANALYSIS

Soil Preservation Requirement: 4°C (±2°C)

Water Preservation Requirements: 4°C (±2°C) and pH less than 2

Sample Holding Time: 28 days (maximum) for water samples

Preservation and Holding Time Result	Action for Samples
Water samples received at pH = 2 (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at pH > 2 (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ). Comment in Sampling Issues.
Water samples received at >6.0°but ≤20°C (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at >20°C (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ). Comment in Sampling Issues.
Water samples received at >6.0°but ≤20°C and pH>2 (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ). Comment in Sampling Issues.
Water samples received at >20°C and pH >2 (Region 9 Advisory Limit)	Qualify all associated results as rejected (R). Comment in Sampling Issues.
Soil samples received at >6.0°but ≤20°C (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Soil samples received at >20°C(Region 9 Advisory Limit)	Qualify results ≥MDL as estimated (J). Qualify results <MDL as estimated (UJ). Comment in Sampling Issues.
Technical Holding Time for Water 28 days but ≤56	Qualify results ≥MDL as estimated (J). Qualify results <MDL as estimated (UJ).
Technical Holding Time for Water >56 days (Region 9 Advisory Limit)	Qualify all associated results as rejected (R). Comment in CLP PO Action section.

Table 4.10
ILM04.X CALIBRATION ACTIONS FOR MERCURY ANALYSIS

Instrument Calibration Requirement: instrument calibrated once every 24 hours using a calibration blank and four calibration standards (minimum)

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Percent Recovery Control Limit: 80 – 120%

Contract Required Detection Limit (CRDL) Standard Solution (CRI) Percent Recovery Control Limit: 65 – 135% (Region 9 advisory limit)

Calibration Result	Action for Samples
Calibration not performed every 24 hours	Qualify all results as rejected (R).
Calibration incomplete - minimum number of calibration standards not used to establish instrument standard curve	Qualify all results as rejected (R).
Correlation Coefficient less than 0.995	Qualify all associated results > IDL as estimated (J) and results < IDL as estimated (UJ)
ICV/CCV %R < 65%	Qualify all associated results as rejected (R)
ICV/CCV %R between 65-79%	Qualify all associated results > IDL as estimated (J) and results <IDL as estimated (UJ) and indicate possible low bias
ICV/CCV %R between 121-135%	Qualify all associated results > IDL as estimated (J) and indicate possible high bias
ICV/CCV %R > 135%	Qualify all associated results > IDL as rejected (R)
CRI %R < 65% or > 135% and sample results near CRDL	Qualify results near CRDL as estimated (J) and comment in Validity and Comments section
CRI %R < 65% or > 135% and sample results > 2 x CRDL	Comment in Additional Comments section only

Table 4.11
ILM04.X BLANK ACTIONS FOR MERCURY ANALYSIS

Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank (PB), Equipment Blank (EB), and Field Blank (FB) Requirements: analyte concentrations must not exceed CRDL values

ICB and CCB Requirement: ICB and CCB must be analyzed after each ICV and CCV

PB Requirement: one PB must be prepared and analyzed for each SDG (minimum)

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	>IDL but ≤CRDL	≥IDL but ≤CRDL	Report CRDL value with a (U)
ICB/CCB	>CRDL	≥IDL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action
ICB/CCB	|-blank result| >CRDL	≥IDL but <5x blank result absolute value	Qualify affected results as estimated (UJ) and comment for CLP PO action
PB	>IDL but ≤CRDL	≥IDL but ≤CRDL	Report CRDL value with a (U)
PB	>CRDL	>CRDL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action
PB	|-blank result| >CRDL	≥IDL but <5x blank result absolute value	Qualify affected results as estimated (UJ) and comment for CLP PO action
FB / EB	>CRDL	≥IDL but ≤CRDL and <5x blank result	Estimate and report at FB/EB value with a (UJ) and comment in Additional Comments section
		>CRDL but <5x blank result	Qualify results as estimated (UJ) and comment in Additional Comments section

Table 4.12
ILM04.X MATRIX SPIKE SAMPLE ACTIONS FOR MERCURY ANALYSIS

Matrix Spike Sample Percent Recovery Control Limit: 75 – 125%

Spike Sample Result	Action for Samples
Matrix Spike %R <30%	Qualify sample results <IDL (or MDL) as rejected (R). Qualify sample results >IDL (or MDL) as estimated (J) with possible low bias.
Matrix Spike %R between 30% - 74%	Qualify sample results <IDL (or MDL) as estimated (UJ) with possible low bias. Qualify sample results >IDL (or MDL) as estimated (J) with possible low bias.
Matrix Spike %R >125%	Qualify sample results >IDL (or MDL) as estimated (J) with possible high bias.

Note: If the original sample result exceeds four times (4x) the spiking concentration, no action is required

Table 4.13
ILM04.X DUPLICATE SAMPLE ACTIONS FOR MERCURY ANALYSIS

Duplicate Sample Relative Percent Difference (RPD) Control Limits:

Water: ±20 RPD or ±CRDL

Soil: ±35 RPD or ±2x CRDL (Region 9 limit)

Duplicate Sample Result	Action for Samples
Water results >5x CRDL: Duplicate control limit ±20 RPD	Qualify sample results as estimated (J) and non-detected results as estimated (UJ).
Water results <5x CRDL: Duplicate control limit ±CRDL	Qualify sample results as estimated (J) and non-detected results as estimated (UJ).
Soil results >5x CRDL: Duplicate control limit ±35 RPD	Qualify sample results as estimated (J) and non-detected results as estimated (UJ).
Soil results <5x CRDL: Duplicate control limit ±2 x CRDL	Qualify sample results as estimated (J) and non-detected results as estimated (UJ).

Note: When a result is detected at greater than or equal to the CRDL in one sample but non-detected in the duplicate sample, do not report a RPD of 200. Instead, indicate in the duplicate sample comment in the report that the analyte was reported at a given concentration in one sample, but not detected in the duplicate sample.

For field duplicate samples only: provide a comment for the field duplicate pair involved, but do not qualify any data.

Table 4.14
ILM04.X LABORATORY CONTROL SAMPLE ACTIONS FOR MERCURY ANALYSIS

Laboratory Control Sample (LCS) Percent Recovery Control Limits:

Aqueous: Aqueous LCS analysis not required by SOW

Soil: within limits established by supplier

LCS Result	Action for Samples
Soil LCS recovery greater than upper control limit	Qualify sample results >IDL (or MDL) as estimated (J) with a possible high bias.
Soil LCS recovery less than lower control limit	Qualify sample results >IDL (or MDL) as estimated (J) with a possible low bias. Qualify sample results <IDL (or MDL) as estimated (UJ) with a possible low bias.
Soil LCS recovery <50% and no control limits are provided (Region 9 advisory limit)	Qualify sample results as rejected (R).

Table 4.15
ILM04.X PRESERVATION AND HOLDING TIME ACTIONS FOR CYANIDE ANALYSIS

Soil Preservation Requirement: 4°C (±2°C)

Water Preservation Requirements: 4°C (±2°C) and pH greater than 12

Sample Holding Time: 14 days (maximum) for water samples

Preservation and Holding Time Result	Action for Samples
Water samples received at pH = 12 (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at pH < 2 (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ) and Comment in Sampling Issues.
Water samples received at >6.0°but ≤20°C (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at >20°C (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ) and Comment in Sampling Issues.
Water samples received at >6.0°but ≤20°C and pH< 12 (Region 9 Advisory Limit)	Qualify results ≥IDL as estimated (J). Qualify results <IDL as estimated (UJ) and Comment in Sampling Issues.
Water samples received at >20°C and pH < 12 (Region 9 Advisory Limit)	Qualify all associated results as rejected (R) and Comment in Sampling Issues.
Soil samples received at >6.0°but ≤20°C (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Soil samples received at >20°C (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated (J). Qualify results <MDL as estimated (UJ) and Comment in Sampling Issues.
Technical Holding Time for Water 14 days but ≤28	Qualify results ≥MDL as estimated (J). Qualify results <MDL as estimated (UJ).
Technical Holding Time for Water >28 days (Region 9 Advisory Limit)	Qualify all associated results as rejected (R) and Comment in CLP PO Action section.

Table 4.16
ILM04.X CALIBRATION ACTIONS FOR CYANIDE ANALYSIS

Instrument Calibration Requirement: instrument calibrated once every 24 hours using a calibration blank and one calibration standard (minimum)

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Percent Recovery Control Limit: 80 – 120%

Contract Required Detection Limit (CRDL) ICP Standard Solution (CRI) Percent Recovery Control Limit: 65 – 135% (Region 9 advisory limit)

Calibration Result	Action for Samples
Calibration not performed every 24 hours	Qualify all results as rejected (R).
Calibration incomplete - minimum number of calibration standards not used to establish instrument standard curve	Qualify all results as rejected (R).
Correlation Coefficient less than 0.995	Qualify all associated results > IDL as estimated (J) and results < IDL as estimated (UJ).
ICV/CCV %R < 65%	Qualify all associated results as rejected (R).
ICV/CCV %R between 65-79%	Qualify all associated results > IDL as estimated (J) and results <IDL as estimated (UJ) and indicate possible low bias.
ICV/CCV %R between 121-135%	Qualify all associated results > IDL as estimated (J) and indicate possible high bias.
ICV/CCV %R > 135%	Qualify all associated results > IDL as rejected (R).
CRI %R < 65% or > 135% and sample results near CRDL	Qualify results near CRDL as estimated (J) and comment in Validity and Comments section.
CRI %R < 65% or > 135% and sample results > 2 x CRDL	Comment in Additional Comments section only.

Table 4.17
ILM04.X BLANK ACTIONS FOR CYANIDE ANALYSIS

Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank (PB), Equipment Blank (EB), and Field Blank (FB) Requirements: analyte concentrations must not exceed CRDL values

ICB and CCB Requirement: ICB and CCB must be analyzed after each ICV and CCV

PB Requirement: one PB must be prepared and analyzed for each SDG (minimum)

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	>IDL but ≤CRDL	≥IDL but ≤CRDL	Report CRDL value with a (U)
ICB/CCB	>CRDL	≥IDL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action
ICB/CCB	|-blank result| >CRDL	≥IDL but <5x blank result absolute value	Qualify affected results as estimated (UJ) and comment for CLP PO action
PB	>IDL but ≤CRDL	≥IDL but ≤CRDL	Report CRDL value with a (U)
PB	>CRDL	>CRDL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action
PB	|-blank result| >CRDL	≥IDL but <5x blank result absolute value	Qualify affected results as estimated (UJ) and comment for CLP PO action
FB / EB	>CRDL	≥IDL but ≤CRDL and <5x blank result	Estimate and report at FB/EB value with a (UJ) and comment in Additional Comments section
		>CRDL but <5x blank result	Qualify results as estimated (UJ) and comment in Additional Comments section

Table 4.18
ILM04.X MATRIX SPIKE SAMPLE ACTIONS FOR CYANIDE ANALYSIS

Matrix Spike Sample Percent Recovery Control Limit: 75 – 125%

Spike Sample Result	Action for Samples
Matrix Spike %R <30%	Qualify sample results <IDL (or MDL) as rejected (R)
Matrix Spike %R between 30% - 74%	Qualify sample results <IDL (or MDL) as estimated (UJ) with possible low bias.
Matrix Spike %R <75%	Qualify sample results >IDL (or MDL) as estimated (J) with possible low bias.
Matrix Spike %R >125%	Sample results <IDL (or MDL) – No action required.
Matrix Spike %R >125%	Qualify sample results >IDL (or MDL) as estimated (J) with possible high bias.

Note: If the original sample result exceeds four times (4x) the spiking concentration, no action is required

Table 4.19
ILM04.X DUPLICATE SAMPLE ACTIONS FOR CYANIDE ANALYSIS

Duplicate Sample Relative Percent Difference (RPD) Control Limits:

Water: ±20 RPD or ±CRDL

Soil: ±35 RPD or ±2x CRDL (Region 9 limit)

Duplicate Sample Result	Action for Samples
Water results >5x CRDL: Duplicate control limit ±20 RPD	Qualify sample results as estimated (J) and non-detected results as estimated (UJ).
Water results <5x CRDL: Duplicate control limit ±CRDL	Qualify sample results as estimated (J) and non-detected results as estimated (UJ).
Soil results >5x CRDL: Duplicate control limit ±35 RPD	Qualify sample results as estimated (J) and non-detected results as estimated (UJ).
Soil results <5x CRDL: Duplicate control limit ±2 x CRDL	Qualify sample results as estimated (J) and non-detected results as estimated (UJ).

Note: When a result is detected at greater than or equal to the CRDL in one sample but non-detected in the duplicate sample, do not report a RPD of 200. Instead, indicate in the duplicate sample comment in the report that the analyte was reported at a given concentration in one sample, but not detected in the duplicate sample.

For field duplicate samples only: provide a comment for the field duplicate pair involved, but do not qualify any data.

Table 4.20
ILM04.X LABORATORY CONTROL SAMPLE ACTIONS FOR CYANIDE ANALYSIS

Laboratory Control Sample (LCS) Percent Recovery Control Limits:

Aqueous: Aqueous LCS analysis not required by SOW

Soil: within limits established by supplier

LCS Result	Action for Samples
Soil LCS recovery greater than upper control limit	Qualify sample results >IDL (or MDL) as estimated (J) with a possible high bias.
Soil LCS recovery less than lower control limit	Qualify sample results >IDL (or MDL) as estimated (J) with a possible low bias. Qualify sample results <IDL (or MDL) as estimated (UJ) with a possible low bias.
Soil LCS recovery <50% and no control limits are provided (Region 9 advisory limit)	Qualify sample results as rejected (R).

APPENDIX B2
SOW ILM05 X DATA REVIEW CRITERIA TABLES

Table 5.1
ILM05.3 PRESERVATION AND HOLDING TIME ACTIONS FOR ICP-AES ANALYSIS

Soil Preservation Requirement: 4°C ±2°C

Water Preservation Requirements: 4°C ±2°C and pH less than 2

Sample Holding Time: 180 days (maximum)

Preservation and Holding Time Result	Action for Samples
Water samples received at pH ≥ 2	Use professional judgment to: Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received at >6.0°C but ≤20°C with pH<2 (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at >20°C with pH<2 (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated (J). Qualify non-detects as estimated (UJ). Comment in Sampling Issues.
Water samples received at >6.0°C but ≤20°C and pH≥2 (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received at >20°C and pH ≥2 (Region 9 Advisory Limit)	Qualify all associated results as rejected (R). Comment in Sampling Issues.
Soil samples received at >6.0°C but ≤20°C (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Soil samples received at >20°C (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ). Comment in Sampling Issues.
Technical Holding Time for Water >180 days (Region 9 Advisory Limit)	Use professional judgment to: Qualify results ≥MDL as estimated (J-). Qualify non-detects as rejected (R). Comment in CLP PO Action section.
Technical Holding Time for Soil	Use professional judgment.

Table 5.2
ILM05.3 CALIBRATION ACTIONS FOR ICP-AES ANALYSIS

Instrument Calibration Requirement: instrument calibrated once every 24 hours using a calibration blank and one calibration standard (minimum)

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Percent Recovery Control Limit: 90 – 110%

Contract Required Quantitation Limit (CRQL) ICP Standard Solution (CRI) Percent Recovery Control Limit: 70 – 130% recovery (50-150% for Sb, Pb, and Tl)

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as rejected (R).
Calibration incomplete	Use professional judgment to: Qualify results ≥MDL as estimated (J) or rejected (R). Qualify non-detects as estimated (UJ) or rejected (R).
ICV/CCV %R < 75%	Use professional judgment to: Qualify results ≥MDL as estimated low (J-) or rejected (R). Qualify non-detects as rejected (R).
ICV/CCV %R between 75 - 89%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
ICV/CCV %R between 111-125%	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
ICV/CCV %R > 125%	Use professional judgment to: Qualify results ≥MDL as estimated high (J+) or rejected (R). Non-detects are not qualified.
ICV/CCV %R > 160%	Qualify results ≥MDL as rejected (R). Non-detects are not qualified.
CRI %R < 50% (<30% for Sb, Pb, and Tl)	Qualify results ≥MDL but <2x CRQL and non-detects as rejected (R). Qualify results ≥2x CRQL as estimated (J).
CRI %R between 50 - 69% (30 - 49% for Sb, Pb, and Tl)	Qualify results ≥MDL but <2x CRQL as estimated low (J-) and qualify non-detects as estimated (UJ). Results ≥2x CRQL are not qualified.
CRI %R > 130% but ≤ 180% (>150 but ≤200% for Sb, Pb, and Tl)	Qualify results ≥MDL but <2x CRQL as estimated high (J+). Non-detects and results ≥2x CRQL are not qualified.
CRI %R > 180% (>200% for Sb, Pb, and Tl)	Qualify results ≥MDL as rejected (R). Non-detects are not qualified.

Table 5.3
ILM05.3 BLANK ACTIONS FOR ICP-AES ANALYSIS

Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank (PB), Equipment Blank (EB), and Field Blank (FB) Requirements: analyte concentrations must not exceed CRQL values

ICB and CCB Requirement: ICB and CCB must be analyzed after each ICV and CCV

PB Requirement: one PB must be prepared and analyzed for each SDG (minimum)

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥MDL but ≤CRQL	Non-detect	No action.
		≥MDL but ≤CRQL	Report CRQL value with a "U".
		>CRQL	Use professional judgment.
ICB/CCB	>CRQL	≥MDL but ≤ CRQL	Report CRQL value with a "U".
		≥MDL but <5x blank result	Use professional judgment to: Report at level of blank result with a "U" or qualify affected results as rejected (R).
		> Blank Result	Use professional judgment.
ICB/CCB	≤(-MDL) but ≥ (-CRQL)	≥MDL, or non-detect	Use professional judgment.
ICB/CCB	< (-CRQL)	<5x CRQL	Qualify results ≥CRQL as estimated low (J-). Qualify non-detects as estimated (UJ).
PB	≥MDL but ≤CRQL	Non-detect	No action.
		≥MDL but ≤CRQL	Report CRQL value with a "U".
		>CRQL	Use professional judgment.
PB	>CRQL	≥MDL but ≤CRQL	Report CRQL value with a (U).
		>CRQL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action.
		≥5x blank result	No action.
PB	<(-CRQL)	>CRQL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action.
FB / EB	>CRQL	≥MDL but <5x blank result absolute value	Estimate and report at FB/EB value with a (UJ) and comment in Additional Comments section.
		>CRQL and <5x blank result	Qualify results as estimated (UJ) and comment in Additional Comments section

Table 5.4
ILM05.3 INTERFERENCE CHECK SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Interference Check Sample A (ICSA) and Interference Check Sample AB (ICSAB) Percent Recovery Control Limits: 80 – 120% or ±2x CRQL (whichever is greater)

Interference Check Sample Result	Action for Samples
Analyte or interferent in ICSAB %R <50%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R).
ICSA / ICSAB %R between 50 - 79% (or < true value - 2x CRQL)	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
ICSA / ICSAB %R >120% (or > true value + 2x CRQL)	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Potential false positives in field samples with interferents	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Potential false negatives in field samples with interferents	Qualify results ≥MDL but < 10x |negative value| as estimated low (J-). Qualify non-detects as estimated (UJ).

Table 5.5
ILM05.3 MATRIX SPIKE SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Matrix Spike and Post-Digestion Spike Percent Recovery Control Limit: 75 – 125%
Post-Digestion Spiking Concentration: 2xCRQL or 2x indigenous level, whichever is greater

Spike Sample Result	Action for Samples
Matrix Spike %R <30% Post-Digestion Spike %R <75%	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as rejected (R).
Matrix Spike %R <30% Post-Digestion Spike %R ≥75%	Qualify affected results ≥MDL as estimated (J). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R between 30% - 74% Post-Digestion Spike %R <75%	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R between 30% - 74% Post-Digestion Spike %R ≥75%	Qualify affected results ≥MDL as estimated (J). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R >125% Post-Digestion Spike %R >125%	Qualify affected results ≥MDL as estimated high (J+). Non-detects are not qualified.
Matrix Spike %R >125% Post-Digestion Spike %R ≤125%	Qualify affected results ≥MDL as estimated (J). Non-detects are not qualified.
Matrix Spike %R between <30% No Post-Digestion Spike Performed (not required for Ag)	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as rejected (R).
Matrix Spike %R between 30% - 74% No Post-Digestion Spike Performed (not required for Ag)	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R >125% No Post-Digestion Spike Performed (not required for Ag)	Qualify affected results ≥MDL as estimated high (J+). Non-detects are not qualified.

Note: If the original sample result exceeds four times (4x) the spiking concentration, no action is required.

Table 5.6
ILM05.3 DUPLICATE SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Duplicate Sample Relative Percent Difference (RPD) Control Limits:

Water: ±20 RPD or ±CRQL

Soil: ±35 RPD or ±2x CRQL (Region 9 Limit)

Duplicate Sample Result	Action for Samples
Water: Both original and duplicate sample results ≥5x CRQL and RPD > 20	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).
Water: Both original and duplicate sample results <5x CRQL (including non-detects) and absolute difference between sample and duplicate result > CRQL	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).
Soil: Both original and duplicate sample results ≥5x CRQL and RPD > 35	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).
Soil: Both original and duplicate sample results ≤5x CRQL (including non-detects) and absolute difference between sample and duplicate result > 2x CRQL	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).

Note: Region 9 requires that when a result is detected at greater than or equal to the CRQL in one sample but non-detected in the duplicate sample, do not report a RPD of 200. Instead, indicate in the duplicate sample comment in the report that the analyte was reported at a given concentration in one sample, but not detected in the duplicate sample.

Table 5.7
ILM05.3 LABORATORY CCONTROL SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Laboratory Control Sample (LCS) Percent Recovery Control Limits:

Aqueous: 80 – 120% (results for Sb and Ag have no fixed control limits)

Soil: within limits established by supplier and reported on Form VII-IN

LCS Result	Action for Samples
Aqueous LCS % R <50%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R).
Aqueous LCS % R between 50% - 79%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
Aqueous LCS %R >120%	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Aqueous LCS %R >150%	Qualify all results as rejected (R).
Soil LCS recovery greater than reported limits	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Soil LCS recovery less than reported limits	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
Soil LCS recovery <50% and no control limits are provided (Region 9 limit)	Qualify sample results as rejected (R).

Table 5.8
ILM05.3 SERIAL DILUTION SAMPLE ACTIONS FOR ICP-AES ANALYSIS

Serial Dilution Percent Difference (%D) Control Limit: 10 %

Serial Dilution Result	Action for Samples
Sample result >50x MDL and %D > 10%	Qualify results ≥MDL as estimated (J). Qualify non-detects as estimated (UJ).
Interferences present	Use professional judgment.

Table 5.9
ILM05.3 PRESERVATION AND HOLDING TIME ACTIONS FOR ICP-MS ANALYSIS

Soil Preservation Requirement: [None at this time]

Water Preservation Requirements: 4°C ±2°C and pH less than 2

Sample Holding Time: 180 days (maximum)

Preservation and Holding Time Result	Action for Samples
Water samples received at pH ≥ 2	Use professional judgment to: Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received at >6.0°C but ≤20°C with pH<2 (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at >20°C with pH<2 (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated (J). Qualify non-detects as estimated (UJ). Comment in Sampling Issues.
Water samples received at >6.0°C but ≤20°C and pH≥2 (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received at >20°C and pH ≥2 (Region 9 Advisory Limit)	Qualify all associated results as rejected (R) and Comment in Sampling Issues.
Technical Holding Time for Water >180 days (Region 9 Advisory Limit)	Use professional judgment to: Qualify results ≥MDL as estimated (J-). Qualify non-detects as rejected (R). Comment in CLP PO Action section.

Table 5.10
ILM05.3 ICP-MS TUNE ACTIONS FOR ICP-MS ANALYSIS

ICP-MS Tune Result	Action for Samples
Tune not performed	Qualify all results as rejected (R).
Tune not performed properly	Use professional judgment.
Mass calibration not within 0.1 amu	Qualify results ≥MDL as estimated (J). Qualify non-detects as estimated (UJ).
%RSD > 5%	Qualify results ≥MDL as estimated (J). Qualify non-detects as estimated (UJ).

Table 5.11
ILM05.3 CALIBRATION ACTIONS FOR ICP-MS ANALYSIS

Instrument Calibration Requirement: instrument calibrated once every 24 hours using a calibration blank and one calibration standard (minimum)

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Percent Recovery Control Limit: 90 – 110%

Contract Required Quantitation Limit (CRQL) ICP Standard Solution (CRI) Percent Recovery Control Limit: 70 – 130% recovery (50-150% for Co, Mn, and Zn)

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as rejected (R).
Calibration incomplete	Use professional judgment to: Qualify results ≥MDL as estimated (J) or rejected (R). Qualify non-detects as estimated (UJ) or rejected (R).
ICV/CCV %R < 75%	Use professional judgment to: Qualify results ≥MDL as estimated low (J-) or rejected (R). Qualify non-detects as rejected (R).
ICV/CCV %R between 75 - 89%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
ICV/CCV %R between 111-125%	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
ICV/CCV %R > 125%	Use professional judgment to: Qualify results ≥MDL as estimated high (J+) or rejected (R). Non-detects are not qualified.
ICV/CCV %R > 160%	Qualify results ≥MDL as rejected (R). Non-detects are not qualified.
CRI %R < 50% (<30% for Co, Mn, and Zn)	Qualify results ≥MDL but <2x CRQL and non-detects as rejected (R). Qualify results ≥2x CRQL as estimated (J).
CRI %R between 50 - 69% (30 - 49% for Co, Mn, and Zn)	Qualify results ≥MDL but <2x CRQL as estimated low (J-) and qualify non-detects as estimated (UJ). Results ≥2x CRQL are not qualified.
CRI %R > 130% but ≤ 180% (>150 but ≤200% for Co, Mn, and Zn)	Qualify results ≥MDL but <2x CRQL as estimated high (J+). Non-detects and results ≥2x CRQL are not qualified.
CRI %R > 180% (>200% for Co, Mn, and Zn)	Qualify results ≥MDL as rejected (R). Non-detects are not qualified.

Table 5.12
ILM05.3 BLANK ACTIONS FOR ICP-MS ANALYSIS

Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank (PB), Equipment Blank (EB), and Field Blank (FB) Requirements: analyte concentrations must not exceed CRQL values

ICB and CCB Requirement: ICB and CCB must be analyzed after each ICV and CCV

PB Requirement: one PB must be prepared and analyzed for each SDG (minimum)

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥MDL but ≤CRQL	Non-detect	No action.
		≥MDL but ≤CRQL	Report CRQL value with a "U".
		>CRQL	Use professional judgment.
ICB/CCB	>CRQL	≥MDL but ≤ CRQL	Report CRQL value with a "U".
		≥MDL but <5x blank result	Use professional judgment to: Report at level of blank result with a "U" or qualify affected results as rejected (R).
		> Blank Result	Use professional judgment.
ICB/CCB	≤(-MDL) but ≥ (-CRQL)	≥MDL, or non-detect	Use professional judgment.
ICB/CCB	< (-CRQL)	<5x CRQL	Qualify results ≥CRQL as estimated low (J-) and qualify non-detects as estimated (UJ).
PB	≥MDL but ≤CRQL	Non-detect	No action.
		≥MDL but ≤CRQL	Report CRQL value with a "U".
		>CRQL	Use professional judgment.
PB	>CRQL	≥MDL but ≤CRQL	Report CRQL value with a (U).
		>CRQL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action.
		≥5x blank result	No action.
PB	<(-CRQL)	>CRQL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action
FB / EB	>CRQL	≥MDL but <5x blank result absolute value	Estimate and report at FB/EB value with a (UJ) and comment in Additional Comments section
		>CRQL and <5x blank result	Qualify results as estimated (UJ) and comment in Additional Comments section

Table 5.13
ILM05.3 INTERFERENCE CHECK SAMPLE ACTIONS FOR ICP-MS ANALYSIS

Interference Check Sample A (ICSA) and Interference Check Sample AB (ICSAB) Percent Recovery Control Limits: 80 – 120% or ±3x CRQL (whichever is greater)

Interference Check Sample Result	Action for Samples
Analyte or interferent in ICSAB %R <50%	Qualify all sample results as rejected (R).
ICSA and/or ICSAB %R between 50 - 79% (or < true value - 3x CRQL)	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
ICSA and/or ICSAB %R >120% (or > true value + 3x CRQL)	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Potential false positives in field samples with interferents	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Potential false negatives in field samples with interferents	Qualify results ≥MDL but < 10x |negative value| as estimated low (J-). Qualify non-detects as estimated (UJ).

Table 5.14
ILM05.3 LABORATORY CCONTROL SAMPLE ACTIONS FOR ICP-MS ANALYSIS

Laboratory Control Sample (LCS) Percent Recovery Control Limits:

Aqueous: 80 – 120%

Soil: [None at this time]

LCS Result	Action for Samples
Aqueous LCS % R <50%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R).
Aqueous LCS % R between 50% - 79%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
Aqueous LCS %R >120%	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Aqueous LCS %R >150%	Qualify all results as rejected (R).

Table 5.15
ILM05.3 DUPLICATE SAMPLE ACTIONS FOR ICP-MS ANALYSIS

Duplicate Sample Relative Percent Difference (RPD) Control Limits:

Water: ±20 RPD or ±CRQL

Soil: [None at this time]

Duplicate Sample Result	Action for Samples
Water: Both original and duplicate sample results ≥5x CRQL and RPD > 20	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).
Water: Both original and duplicate sample results <5x CRQL (including non-detects) and absolute difference between sample and duplicate result > CRQL	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).

Note: Region 9 requires that when a result is detected at greater than or equal to the CRQL in one sample but non-detected in the duplicate sample, do not report a RPD of 200. Instead, indicate in the duplicate sample comment in the report that the analyte was reported at a given concentration in one sample, but not detected in the duplicate sample.

Table 5.16
ILM05.3 MATRIX SPIKE SAMPLE ACTIONS FOR ICP-MS ANALYSIS

Matrix Spike and Post-Digestion Spike Percent Recovery Control Limit: 75 – 125%
Post-Digestion Spiking Concentration: 2xCRQL or 2x indigenous level, whichever is greater.

Spike Sample Result	Action for Samples
Matrix Spike %R <30% Post-Digestion Spike %R <75%	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as rejected (R).
Matrix Spike %R <30% Post-Digestion Spike %R ≥75%	Qualify affected results ≥MDL as estimated (J). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R between 30% - 74% Post-Digestion Spike %R <75%	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R between 30% - 74% Post-Digestion Spike %R ≥75%	Qualify affected results ≥MDL as estimated (J). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R >125% Post-Digestion Spike %R >125%	Qualify affected results ≥MDL as estimated high (J+). Non-detects are not qualified.
Matrix Spike %R >125% Post-Digestion Spike %R ≤125%	Qualify affected results ≥MDL as estimated (J). Non-detects are not qualified.
Matrix Spike %R between <30% No Post-Digestion Spike Performed	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as rejected (R).
Matrix Spike %R between 30% - 74% No Post-Digestion Spike Performed	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R >125% No Post-Digestion Spike Performed	Qualify affected results ≥MDL as estimated high (J+). Non-detects are not qualified.

Note: If the original sample result exceeds four times (4x) the spiking concentration, no action is required.

Table 5.17
ILM05.3 SERIAL DILUTION SAMPLE ACTIONS FOR ICP-MS ANALYSIS

Serial Dilution Percent Difference (%D) Control Limit: 10 %

Serial Dilution Result	Action for Samples
Sample result >50x MDL and %D > 10%	Qualify results ≥MDL as estimated (J) and non-detects as estimated (UJ).
Interferences present	Use professional judgment.

Table 5.18
ILM05.3 INTERNAL STANDARD ACTIONS FOR ICP-MS ANALYSIS

Internal Standard Percent Relative Intensity (%RI) Control Limit: 60-125 % of the calibration blank

Internal Standard Results	Action for Samples
No internal standards	Qualify all results as rejected (R) and comment in CLP PO Action Section.
<5 of the permitted internal standards used	Qualify all analyte results not bracketed by internal standard masses as rejected (R) and comment in CLP PO Action Section.
Internal standard masses do not bracket target analyte massed	Qualify all analyte results not bracketed by internal standard masses as rejected (R) and comment in CLP PO Action Section.
%RI <60% or >125%, and original sample reanalyzed at 2 time dilution	If diluted sample %RI is within 60-125%, do not qualify the data.
	If diluted sample %RI is outside the 60-125% limits, report results from undiluted analysis and qualify results ≥MDL as estimated (J) and qualify non-detects as estimated (UJ).
Original sample not reanalyzed at 2 time dilution	Use professional judgment to: Qualify sample results as estimated (J) or rejected (R).

ILM05.3 permitted internal standards: lithium (Li^6), scandium (Sc), Yttrium (Y), rhodium (Rh), indium (In^{115}), terbium (Tb), holmium (Ho), lutetium (Lu), bismuth (Bi).

Table 5.19
ILM05.3 PRESERVATION AND HOLDING TIME ACTIONS FOR MERCURY ANALYSIS

Soil Preservation Requirement: 4°C ±2°C

Water Preservation Requirements: 4°C ±2°C and pH less than 2

Sample Holding Time: 28 days (maximum) for water samples

Preservation and Holding Time Result	Action for Samples
Water samples received at pH ≥ 2	Use professional judgment to: Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received at >6.0°C but ≤20°C with pH <2 (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at >20°C with pH <2 (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated (J). Qualify non-detects as estimated (UJ). Comment in Sampling Issues.
Water samples received at >6.0°C but ≤20°C and pH ≥2 (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received at >20°C and pH ≥2 (Region 9 Advisory Limit)	Qualify all associated results as rejected (R). Comment in Sampling Issues.
Soil samples received at >6.0°C but ≤20°C (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Soil samples received at >20°C (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ). Comment in Sampling Issues.
Technical Holding Time for Water >56 days (Region 9 Advisory Limit)	Use professional judgment to: Qualify results ≥MDL as estimated (J-). Qualify non-detects as rejected (R). Comment in CLP PO Action section.
Technical Holding Time for Soil	Use professional judgment.

Table 5.20
ILM05.3 CALIBRATION ACTIONS FOR MERCURY ANALYSIS

Instrument Calibration Requirement: instrument calibrated once every 24 hours using a calibration blank and four calibration standards (minimum)

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Percent Recovery Control Limit: 80 – 120%

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as rejected (R).
Calibration incomplete	Use professional judgment to: Qualify results ≥MDL as estimated (J) or rejected (R). Qualify non-detects as estimated (UJ) or rejected (R).
Correlation Coefficient less than 0.995	Use professional judgment to: Qualify results ≥MDL as estimated (J) or rejected (R). Qualify non-detects as estimated (UJ) or rejected (R).
ICV/CCV %R < 65%	Use professional judgment to: Qualify results ≥MDL as estimated low (J-) or rejected (R). Qualify non-detects as rejected (R).
ICV/CCV %R between 65 - 79%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
ICV/CCV %R between 121-135%	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
ICV/CCV %R > 135%	Use professional judgment to: Qualify results ≥MDL as estimated high (J+) or rejected (R). Non-detects are not qualified.
ICV/CCV %R > 170%	Qualify results ≥MDL as rejected (R). Non-detects are not qualified.

Table 5.21
ILM05.3 CALIBRATION ACTIONS FOR MERCURY ANALYSIS

Contract Required Quantitation Limit (CRQL) Standard Solution (CRI) Percent Recovery Control Limit: 70 – 130% recovery

Calibration Result	Action for Samples
CRI %R < 50%	Qualify results ≥MDL but <2x CRQL and non-detects as rejected (R). Qualify results ≥2x CRQL as estimated (J).
CRI %R between 50 - 69%	Qualify results ≥MDL but <2x CRQL as estimated low (J-). Qualify non-detects as estimated (UJ). Results ≥2x CRQL are not qualified.
CRI %R > 130% but ≤ 180%	Qualify results ≥MDL but <2x CRQL as estimated high (J+). Non-detects and results ≥2x CRQL are not qualified.
CRI %R > 180%	Qualify results ≥MDL as rejected (R).

Table 5.22
ILM05.3 BLANK ACTIONS FOR MERCURY ANALYSIS

Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank (PB), Equipment Blank (EB), and Field Blank (FB) Requirements: analyte concentrations must not exceed CRQL values

ICB and CCB Requirement: ICB and CCB must be analyzed after each ICV and CCV

PB Requirement: one PB must be prepared and analyzed for each SDG (minimum)

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥MDL but ≤CRQL	Non-detect	No action.
		≥MDL but ≤CRQL	Report CRQL value with a "U".
		>CRQL	Use professional judgment.
ICB/CCB	>CRQL	≥MDL but ≤ CRQL	Report CRQL value with a "U".
		≥MDL but <5x blank result	Use professional judgment to: Report at level of blank result with a "U" or qualify affected results as rejected (R).
		> Blank Result	Use professional judgment.
ICB/CCB	≤(-MDL) but ≥ (-CRQL)	≥MDL, or non-detect	Use professional judgment.
ICB/CCB	< (-CRQL)	<5x CRQL	Qualify results ≥CRQL as estimated low (J-). Qualify non-detects as estimated (UJ).
PB	≥MDL but ≤CRQL	Non-detect	No action.
		≥MDL but ≤CRQL	Report CRQL value with a "U".
		>CRQL	Use professional judgment.
PB	>CRQL	≥MDL but ≤CRQL	Report CRQL value with a (U).
		>CRQL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action.
		≥5x blank result	No action.
PB	<(-CRQL)	>CRQL but <5x blank result	Qualify affected results as estimated (UJ) and comment for CLP PO action.
FB / EB	>CRQL	≥MDL but <5x blank result absolute value	Estimate and report at FB/EB value with a (UJ) and comment in Additional Comments section.
		>CRQL and <5x blank result	Qualify results as estimated (UJ) and comment in Additional Comments section

Table 5.23
ILM05.3 MATRIX SPIKE SAMPLE ACTIONS FOR MERCURY ANALYSIS

Matrix Spike and Post-Digestion Spike Percent Recovery Control Limit: 75 – 125%

Spike Sample Result	Action for Samples
Matrix Spike %R between <30% (Post-Digestion Spike not required for Hg)	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as rejected (R).
Matrix Spike %R between 30%-74% (Post-Digestion Spike not required for Hg)	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R >125% (Post-Digestion Spike not required for Hg)	Qualify affected results ≥MDL as estimated high (J+). Non-detects are not qualified.

Note: If the original sample result exceeds four times (4x) the spiking concentration, no action is required.

Table 5.24
ILM05.3 DUPLICATE SAMPLE ACTIONS FOR MERCURY ANALYSIS

Duplicate Sample Relative Percent Difference (RPD) Control Limits:

Water: ±20 RPD or ±CRQL

Soil: ±35 RPD or ±2x CRQL (Region 9 limit)

Duplicate Sample Result	Action for Samples
Water: Both original and duplicate sample results ≥5x CRQL and RPD > ±20	Use professional judgment to: Qualify results ≥MDL as estimated (J) and non-detected results as estimated (UJ).
Water: Both original and duplicate sample results ≤5x CRQL (including non-detects) and absolute difference between sample and duplicate result > ±CRQL	Use professional judgment to: Qualify results ≥MDL as estimated (J) and non-detected results as estimated (UJ).
Soil: Both original and duplicate sample results ≥5x CRQL and RPD > ±35	Use professional judgment to: Qualify results ≥MDL as estimated (J) and non-detected results as estimated (UJ).
Soil: Both original and duplicate sample results ≤5x CRQL (including non-detects) and absolute difference between sample and duplicate result > ±2x CRQL	Use professional judgment to: Qualify results ≥MDL as estimated (J) and non-detected results as estimated (UJ).

Note: Region 9 requires that when a result is detected at greater than or equal to the CRQL in one sample but non-detected in the duplicate sample, do not report a RPD of 200. Instead, indicate in the duplicate sample comment in the report that the analyte was reported at a given concentration in one sample, but not detected in the duplicate sample.

For Field Duplicate Samples Only: Provide a comment for the field duplicate pair involved, but do not qualify any data.

Table 5.25
ILM05.3 LABORATORY CCONTROL SAMPLE ACTIONS FOR MERCURY ANALYSIS

Laboratory Control Sample (LCS) Percent Recovery Control Limits:

Aqueous: Aqueous LCS analysis not required by SOW

Soil: within limits established by supplier and reported of FormVII-IN

LCS Result	Action for Samples
Soil LCS recovery greater than reported limits	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Soil LCS recovery less than reported limits	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
Soil LCS recovery <50% and no control limits are provided (Region 9 limit)	Qualify sample results as rejected (R).

Table 5.26
ILM05.3 PRESERVATION AND HOLDING TIME ACTIONS FOR CYANIDE ANALYSIS

Soil Preservation Requirement: 4°C ±2°C

Water Preservation Requirements: 4°C ±2°C and pH greater than 12

Sample Holding Time: 14 days (maximum) for water samples

Preservation and Holding Time Result	Action for Samples
Water samples received at pH ≤12	Use professional judgment to: Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received at >6.0°C but ≤20°C with pH >12 (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Water samples received at >20°C with pH >12 (Region 9 Advisory Limit)	Qualify results >MDL as estimated (J). Qualify non-detects as estimated (UJ). Comment in Sampling Issues.
Water samples received at >6.0°C but ≤20°C and pH ≤12 (Region 9 Advisory Limit)	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received at >20°C and pH ≤12 (Region 9 Advisory Limit)	Qualify all associated results as rejected (R). Comment in Sampling Issues.
Water samples received with oxidizing agents present	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Water samples received with sulfides present and sulfides are not removed	Qualify results ≥MDL as estimated (J). Qualify non-detects as rejected (R). Comment in Sampling Issues.
Soil samples received at >6.0°C but ≤20°C (Region 9 Advisory Limit)	Do not qualify results but comment in Sampling Issues.
Soil samples received at >20°C (Region 9 Advisory Limit)	Qualify results >MDL as estimated (J). Qualify non-detected results as estimated (UJ). Comment in Sampling Issues.
Technical Holding Time for Water >28 days (Region 9 Advisory Limit)	Use professional judgment to: Qualify results >MDL as estimated low (J-). Qualify non-detects as rejected (R). Comment in CLP PO Action section.
Technical Holding Time for Soil	Use professional judgment.

Table 5.27
ILM05.3 CALIBRATION ACTIONS FOR CYANIDE ANALYSIS

Instrument Calibration Requirement: instrument calibrated once every 24 hours using a calibration blank and three calibration standard (minimum)

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Percent Recovery Control Limit: 85 – 115%
ICV and Mid-Range Calibration Standard must be distilled before analysis and results must agree within ±15% of the undistilled standard true value.

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as rejected (R).
Calibration incomplete	Use professional judgment to: Qualify results ≥MDL as estimated (J) or rejected (R). Qualify non-detects as estimated (UJ) or rejected (R).
Correlation Coefficient less than 0.995	Use professional judgment to: Qualify results ≥MDL as estimated (J) or rejected (R). Qualify non-detects as estimated (UJ) or rejected (R).
ICV/mid-range standard not distilled, or distilled standards >±15% but ≤±30% recovery	Qualify results ≥MDL as estimated (J).
Distilled standards >±30% recovery	Qualify results ≥MDL as rejected (R).
ICV/CCV %R < 70%	Use professional judgment to: Qualify results ≥MDL as estimated low (J-) or rejected (R). Qualify non-detects as rejected (R).
ICV/CCV %R between 70 - 84%	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
ICV/CCV %R between 116-130%	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
ICV/CCV %R > 130%	Use professional judgment to: Qualify results ≥MDL as estimated high (J+) or rejected (R). Non-detects are not qualified.
ICV/CCV %R > 165%	Qualify results ≥MDL as rejected (R). Non-detects are not qualified.

Table 5.28
ILM05.3 CALIBRATION ACTIONS FOR CYANIDE ANALYSIS

Contract Required Quantitation Limit (CRQL) Standard Solution (CRI) Percent Recovery
Control Limit: 70 – 130% recovery

Calibration Result	Action for Samples
CRI %R < 50%	Qualify results ≥MDL but <2x CRQL and non-detects as rejected (R). Qualify results ≥2x CRQL as estimated (J).
CRI %R between 50 - 69%	Qualify results ≥MDL but <2x CRQL as estimated low (J-). Qualify non-detects as estimated (UJ). Results ≥2x CRQL are not qualified.
CRI %R > 130% but ≤ 180%	Qualify results ≥MDL but <2x CRQL as estimated high (J+). Non-detects and results ≥2x CRQL are not qualified.
CRI %R > 180%	Qualify results ≥MDL as rejected (R).

Table 5.29
ILM05.3 BLANK ACTIONS FOR CYANIDE ANALYSIS

Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank (PB), Equipment Blank (EB), and Field Blank (FB) Requirements: analyte concentrations must not exceed CRQL values

ICB and CCB Requirement: ICB and CCB must be analyzed after each ICV and CCV

PB Requirement: one PB must be prepared and analyzed for each SDG (minimum)

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥MDL but ≤CRQL	Non-detect	No action.
		≥MDL but ≤CRQL	Report CRQL value with a "U".
		>CRQL	Use professional judgment.
ICB/CCB	>CRQL	≥MDL but ≤ CRQL	Report CRQL value with a "U".
		≥MDL but <5x Blank Result	Use professional judgment to: Report at level of blank result with a "U". Qualify affected results as rejected (R).
		> Blank Result	Use professional judgment and comment in CLP PO Action Section.
ICB/CCB	≤(-MDL) but ≥ (-CRQL)	≥MDL, or non-detect	Use professional judgment.
ICB/CCB	< (-CRQL)	<5x CRQL	Qualify results ≥CRQL as estimated low (J-). Qualify non-detects as estimated (UJ).
PB	≥MDL but ≤CRQL	Non-detect	No action.
		≥MDL but ≤CRQL	Report CRQL value with a "U".
		>CRQL	Use professional judgment.
PB	>CRQL	≥MDL but ≤CRQL	Report CRQL value with a (U).
		>CRQL but <5x Blank Result	Qualify affected results as estimated (UJ) and comment in CLP PO Action.
		≥5x Blank Result	No action.
PB	<(-CRQL)	>CRQL but <5x Blank Result	Qualify affected results as estimated (UJ) and comment in CLP PO Action.
FB / EB	>CRQL	≥MDL but <5x Blank Result absolute value	Estimate and report at FB/EB value with a (UJ) and comment in Additional
		>CRQL and <5x blank	Qualify results as estimated (UJ) and co

Table 5.30
ILM05.3 MATRIX SPIKE SAMPLE ACTIONS FOR CYANIDE ANALYSIS

Matrix Spike and Post-Distillation Spike Percent Recovery Control Limit: 75 – 125%

Spike Sample Result	Action for Samples
Matrix Spike %R <30% Post- Distillation Spike %R <75%	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as rejected (R).
Matrix Spike %R <30% Post- Distillation Spike %R ≥75%	Qualify affected results ≥MDL as estimated (J). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R between 30% -74% Post- Distillation Spike %R <75%	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R between 30% -74% Post- Distillation Spike %R ≥75%	Qualify affected results ≥MDL as estimated (J). Qualify affected non-detects as estimated (UJ).
Matrix Spike %R >125% Post- Distillation Spike %R >125%	Qualify affected results ≥MDL as estimated high (J+). Non-detects are not qualified.
Matrix Spike %R >125% Post- Distillation Spike %R ≤125%	Qualify affected results ≥MDL as estimated (J). Non-detects are not qualified.
Matrix Spike %R between <30% No Post- Distillation Spike Performed	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as rejected (R). Comment in CLP PO Action Section.
Matrix Spike %R between 30% - 74% No Post- Distillation Spike Performed	Qualify affected results ≥MDL as estimated low (J-). Qualify affected non-detects as estimated (UJ). Comment in CLP PO Action Section.
Matrix Spike %R >125% No Post-Distillation Spike Performed	Qualify affected results ≥MDL as estimated high (J+). Non-detects are not qualified. Comment in CLP PO Action Section.

Note: If the original sample result exceeds four times (4x) the spiking concentration, no action is required

Table 5.31
ILM05.3 DUPLICATE SAMPLE ACTIONS FOR CYANIDE ANALYSIS

Duplicate Sample Relative Percent Difference (RPD) Control Limits:

Water: ±20 RPD or ±CRQL

Soil: ±35 RPD or ±2x CRQL (Region 9 limit)

Duplicate Sample Result	Action for Samples
Water: Both original and duplicate sample results ≥5x CRQL and RPD > 20	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).
Water: Both original and duplicate sample results <5x CRDL (including non-detects) and absolute difference between sample and duplicate result > CRQL	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).
Soil: Both original and duplicate sample results ≥5x CRQL and RPD > 35	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).
Soil: Both original and duplicate sample results ≤5x CRQL (including non-detects) and absolute difference between sample and duplicate result > 2x CRQL	Use professional judgment to: Qualify results ≥MDL as estimated (J). Qualify non-detected results as estimated (UJ).

Note: Region 9 requires that when a result is detected at greater than or equal to the CRQL in one sample but non-detected in the duplicate sample, do not report a RPD of 200. Instead, indicate in the duplicate sample comment in the report that the analyte was reported at a given concentration in one sample, but not detected in the duplicate sample.

For Field Duplicate Samples Only: Provide a comment for the field duplicate pair involved, but do not qualify any data.

Table 5.32
ILM05.3 LABORATORY CCONTROL SAMPLE ACTIONS FOR CYANIDE ANALYSIS

Laboratory Control Sample (LCS) Percent Recovery Control Limits:

Aqueous: Aqueous LCS analysis not required by SOW

Soil: within limits established by supplier and reported on Form VII-IN

LCS Result	Action for Samples
Soil LCS recovery greater than established limits	Qualify results ≥MDL as estimated high (J+). Non-detects are not qualified.
Soil LCS recovery less than established limits	Qualify results ≥MDL as estimated low (J-). Qualify non-detects as estimated (UJ).
Soil LCS recovery <50% and no control limit is provided (Region 9 limit)	Qualify sample results as rejected (R).

APPENDIX C1
SOW ILM04.X WORKSHEET
CASE SUMMARY

Case No.:_____ Site Name:_____

SDG No.:_____ EPA/CERCLIS Site I.D.:_____

Date Received:_____ Due Date: _____ SOW#: _____ TDF#:_____

Laboratory:_____ Lab Code:_____

Region No./CLP PO:_____ Analysis:_____

# of Samples & Matrix: _____Concentration: Low/Med/High

EPA Project Officer & Title:_____

Section & Mail Code:_____

Sample Numbers:_____

Field and/or Equipment Blanks:_____

Background:_____

Field Duplicates:_____

Validation Parameters	Qualification Code			
	ICP	GFAA	Hg	Cyanide
1. PRESERVATION AND HOLDING TIMES				
2. CALIBRATION				
3. LABORATORY AND FIELD BLANKS				
4. ICP INTERFERENCE CHECK SAMPLE (ICS)		N	N	N
5. MATRIX SPIKE ANALYSIS				
6. LABORATORY AND FIELD DUPLICATE ANALYSIS				
7. LABORATORY CONTROL SAMPLE (LCS)				
8. METHOD OF STANDARD ADDITION (MSA) & GFAA QC	N		N	N
9. ICP SERIAL DILUTION, ICP LRV, & IDL STUDY				
10. SAMPLE QUANTITATION				
11. SAMPLE VERIFICATION				
12. OVERALL ASSESSMENT				

Qualification Code: A = Acceptable
R = Rejected
J = Estimated (see worksheet)
C = Comment only
N = Not applicable or provided

1.0 PRESERVATION & TECHNICAL HOLDING TIMES

National Functional Guidelines for Inorganic Data Review Technical Holding Time and Preservation Criteria:

	Dates Prepared	Acceptable	Qualification
Metals: 6 months, preserved to pH <2	_____	Yes/No	R/J/None
Mercury: 28 days, preserved to pH <2	_____	Yes/No	R/J/None
Cyanide: 14 days, preserved to pH >12	_____	Yes/No	R/J/None
Percent Solids: Yes/No If Yes,	_____	Yes/No	R/J/None

Concentration: Low/Med/High

Matrix: Soil/Water/Other

Fld QC	Sample Number	Collected/ Received	pH/ ^{o}C	Dates Run/ # of Day Exceeded						
				ICP	As/GFAA	Pb/GFAA	Se/GFAA	Tl/GFAA	Hg	CN

2.0 CALIBRATION

INSTRUMENT CALIBRATION AND CRDL STANDARDS

Were the following parameters met:	Acceptable?
• ICP calibration curve: 1 blank, 1 standard	Yes/No/NA
•. GFAA calibration curve: 1 blank, 3 standards (1 at CRDL)	Yes/No/NA
• Mercury calibration curve: 1 blank, 4 standards (5-8 for auto)	Yes/No/NA
• Cyanide calibration range: 1 blank, 3 standards (1 at CRDL and mid-range standard must be distilled)	Yes/No/NA
• GFAA, Hg, and Cyanide correlation coefficients (r). 0.995	Yes/No/NA
• Cyanide mid-range standard distilled and result ± 15% of undistilled standard	Yes/No/NA
• CRI standard for ICP at 2× CRDL run after the ICV, after 20 analytical samples, and at the end of the run (not required for Al, Ba, Ca, Fe, Mg, Na, and K)	Yes/No/NA
• CRA standard for GFAA and Hg at CRDL or IDL (whichever is greater) run after an ICV	Yes/No/NA
• CRDL calibration standard used for GFAA, Hg, or cyanide calibration [If No, make comment in CLP PO ATTENTION]	Yes/No/NA

Criteria: CRDL Standards = 65-135% of True Value (Region 9 Advisory Limits)

CRDL Standards Acceptable: Yes/No [If No, make comment in Additional Comments]

Analyte	r value	CRA/CRI %R r I	CRA/CRI %R F	Associated Samples	Qualification

Comments/Calculations:

2.1 CALIBRATION

INITIAL CALIBRATION VERIFICATION (ICV) AND CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: Metals (ICP/AA) = 90-110% of True Value: Hg (Cold Vapor) = 80-120% of True Value; Cyanide = 85-115% of True Value [ESAT allows ±1% deviation due to rounding]

Were the following parameters met:	Acceptable?
• ICV prepared from second source and run immediately after a system calibration	Yes/No
• CCVs run at the beginning (after an ICS), every 10 samples or every 2 hours, and at the end of the analysis	Yes/No
• CCV concentration at or near mid-range calibration standard	Yes/No
• ICV, ICB, 1st CCV, and 1st CCB run before 1st samples	Yes/No
• If ICV or CCV %R exceeds control limits, lab stops analysis, recalibrates, and reruns affected samples [If Yes,-make comment in CLP PO ACTION]	Yes/No

ICV/CCVs Acceptable: Yes/No

Analyte	ICV %R	CCV %R	Associated Samples	Qualification

Comments/Calculations:

3.0 LABORATORY AND FIELD BLANKS

ICB = Initial Calibration Blank CCB = Continuing Calibration Blank PB = Preparation Blank
FB = Field Blank EB = Equipment Blank

Criteria: No analyte should be present in any blank associated with an SDG in excess of the CRDL. Samples associated with blanks having concentrations >CRDL must be qualified according to the following:

Sample Concentration	Qualification
>IDL (MDL) and <5× any blank concentration	*Flag as nondetected (UJ)*

Any blank reported with a negative result whose absolute value is >CRDL must be carefully evaluated. Use professional judgment to determine its effect on the sample data.

- For results <CRDL, raise results to CRDL and flag as nondetected (UJ).
- For diluted results <CRDL, raise CRDL by sample dilution factor (CRDL x df) and flag as nondetected (UJ).

Lab Blanks Acceptable: Yes/No/NA
[If No, make comment in CLP PO ACTION]

Field Blanks Acceptable: Yes/No/NA
[If No, make comment in SAMPLING ISSUES]

Analyte	Blank ID	Conc. in Blank	Associated Samples	Qualification

- Were the blank analysis frequency requirements met? Yes/No
- Was at least one preparation blank prepared and analyzed per matrix for each SDG/preparation batch of 20 or fewer samples? Yes/No

Comments/Calculations:

4.0 ICP INTERFERENCE CHECK SAMPLE (ICS)

Solution A (ICSA) = Al, Ca, Fe, and Mg (interferents)
Solution B (ICSAB) = ICP analytes mixed with the interferents

Criteria: Solution A should only contain Al, Ca, Fe, and Mg. In general, qualification should be made for interference when false positives or negatives occur for ICP analytes in samples with concentrations of Al, Ca, Fe, and/or Mg which are comparable to their respective concentrations in ICS solutions A and AB.

Solution AB concentrations should be 80-120% of the true values, or qualification is necessary.

- If ICSAB %R is outside the control limits, the lab stops analysis, recalibrates, and reruns affected samples. If not done, make comment in CLP PO ACTION.
- Were the ICP Interference Check Samples (solutions A and AB) analyzed and reported at the beginning and end of each analytical run or a minimum of twice per 8 hour working shift (whichever is more frequent) but not before the ICV? Yes/No
- Was an interferent concentration in the sample greater than or equal to the concentration in the ICS solution or any interfering analyte concentration in the sample greater than 10 mg/L? Yes/No
- If the absolute value of any analytes **not** present in the ICSA solution are found at a concentration greater than the CRDL **and** interferents concentrations are greater than or equal to those in the ICS, qualify sample results >IDL as estimated (J) and sample results <IDL as estimated (UJ).

Acceptable: Yes/No

Analyte	ICSAB True	ICSAB Found	ICSAB %R	Associated Samples	Qualification

- If an interferent is present at a concentration greater than 120% of the ICS solution concentration, use the following equation to estimate the affected analyte concentration produced by the interferent:

$$[I] \times A_{IEC} = [C_A]$$

Where, [I] = Interferent concentration (Al, Ca, Fe, Mg) or analyte concentration >10 mg/L.
A_{IEC} = Analyte Interelement Correction Factor (from Form 11A and/or 11B).
$[C_A]$ = estimated analyte concentration produced by the interferent.

If $[C_A]$ is > 2x CRDL **AND** > 10% of the reported concentration of the affected analyte, qualify the affected analyte result as estimated (J).

5.0 MATRIX SPIKE ANALYSIS

Criteria: Apply matrix spike (MS) percent recovery (%R) control limits of 75-125%, except when sample concentration exceeds 4× spike concentration.
Note that the post digest spike %R result is **not** used to estimate sample data.

Background sample data is qualified due to MS outliers.
Field and equipment blank and PE sample data are not qualified due to MS outliers.

- Was a matrix spike analysis performed on each group of samples of similar matrix type and concentration or for each SDG? Yes/No
  [If No, make comment in CLP PO ATTENTION.]

- Was correct spike concentration added to sample before the digestion or distillation step? Yes/No
  [If No, make comment in CLP PO ACTION.]

- Was a post-digestion/post-distillation spike performed for ICP (except Ag) and CN? Yes/No/NA
  [Note that a post-digest spike is not required for Hg.]

- Did all required post-digest spikes meet the required criteria? Yes/No

QC Sample Number(s): Acceptable: Yes/No

Analyte	M-Spike %R	A-Spike %R	Associated Samples	Qualification

- Was the QC sample specified on the chain of custody used in the analysis performed? Yes/No

- Was field or equipment blank used as matrix spike sample? Yes/No
  [If Yes, make comment in CLP PO ACTION.]

- Were the spiking levels specified in Table 3 of the SOW used appropriately? Yes/No

Comments/Calculations:

6.0 LABORATORY AND FIELD DUPLICATES

Criteria: Water: ±20 RPD for sample values > 5× CRDL or ±CRDL for sample values < 5× CRDL.

Soil: ±35 RPD for sample values > 5× CRDL or ±2× CRDL for sample values < 5× CRDL

Background sample data is qualified due to laboratory duplicate outliers.
Field blank, equipment blank, and PE sample data are <u>not</u> qualified due to laboratory duplicate outliers.

- Was a field blank or equipment blank used as duplicate sample? Yes/No
  [If Yes, make comment in CLP PO ACTION.]

- If field duplicate results exceed Control Limits, comment on the duplicate pair involved, but do not qualify data.

QC Sample Number:
Lab. Dups. Acceptable: Yes/No Field Dups. Acceptable: Yes/No/NA

Analyte	RPD or CRDL	Field Dup Sample	Associated Samples	Qualification

Comments/Calculations:

7.0 LABORATORY CONTROL SAMPLE (LCS)

Criteria: Water (LCSW): 80-120% of the true value
(except for Sb and Ag which have no control limits)

Solid (LCSS): Must fall within the control limits established by the EPA for each element.

- The analysis of the laboratory control sample is required for each analyte (except Hg and Cyanide in water samples) using the same sample preparation, analytical methods, and QC procedures employed for the other samples in the SDG.

- The LCSW or LCSS results for target analytes shall fall within the specified acceptance limits. Otherwise, all associated samples shall be re-digested and reanalyzed.
[If this was not done, make a comment in CLP PO ACTION.]

Matrix: Soil/Water Acceptable: Yes/No/NA

Analyte	True Value	Found Value	%R (LCSW) / EPA Limits (LCSS)	Associated Samples	Qualification

- If the LCSW or LCSS recovery is <50%, reject associated samples and comment in CLP PO ACTION.

Comments/Calculations:

8.0 MSA and GFAA QC

Refer to the Furnace Atomic Absorption Analysis Scheme (Figure 1) on page E-29 of the Inorganic CLP SOW ILM04.0

- Were duplicate injections and post-digestion analytical spikes at 2× CRDL performed for all samples? Yes/No
  If No, Sample Number:

- Did the duplicate injections agree within 20% RSD or CV for sample concentrations >CRDL? Yes/No
  If No, Sample Number:

- Were the samples with <40% spike recovery diluted and rerun with another spike? Yes/No/NA
  If No, Sample Number:

- Were the post-digestion spike recoveries for samples with concentrations <50% of the spike concentration within 85-115%? Yes/No/NA
  If No, see table below:

- Was an MSA performed on samples with spike recovery <85% or >115% and with sample concentrations 50% of the spike? Yes/No/NA
  If No, Sample Number:

- Were the MSA spike concentrations at approximately 50%, 100%, and 150% of the sample concentration? Yes/No/NA
  If No, Sample Number:

- For samples quantitated by MSA, was the correlation coefficient (r) $\geq$ 0.995? Yes/No/NA
  If No, Sample Number:

- Were all samples analyzed undiluted? Yes/No/NA
  If No, list sample numbers, analytes, and dilution factors:

8.1 GFAA QC WORKSHEET

Sample Number	Arsenic %R/Flag	Lead %R/Flag	Selenium %R/Flag	Thallium %R/Flag	Analyte MSA r<0.995

Comments/Calculations:

9.0 ICP SERIAL DILUTION ANALYSIS, ICP LRV, AND IDL STUDY

Field blank, equipment blank, and PE sample data are not qualified due to ICP serial dilution outliers.

- Was an ICP serial dilution (a five fold dilution) analysis performed on the sample from a group of samples from a similar matrix and concentration or for each SDG, whichever is more frequent? Yes/No

- Was the serial dilution within 10% of the original result after correction for dilution for analytes with concentrations that are greater that 50× the IDL in the original sample? Yes/No/NA
  [If serial dilution sample was **not** analyzed, note in Additional Comments.]

QC Sample Number: Acceptable: Yes/No

Analyte	% Difference	Associated Samples	Qualification

ICP Linear Range Verification (LRV) and Instrument Detection Limit (IDL) Study:

- Was the ICP LRV performed at least quarterly (every 3 calendar months) for each analyte reported? Yes/No

- Was the IDL determined at least quarterly (every 3 calendar months) for each analyte reported on each instrument used? Yes/No

- Were the IDLs correctly reported on Form 10? Yes/No

- Does instrument ID on Forms 10 and 12 match instrument ID used for sample analysis? Yes/No

Comments/Calculations:

10.0 VALIDATION FINDINGS AND RECALCULATIONS

Percent recoveries (%R) for an ICP interference check sample, laboratory control sample, and a matrix spike sample were recalculated using the following equation:

%R = (Found/True) × 100 Where, Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, *Found = SSR (spike sample result) - SR (sample result).*

True = Concentration of each analyte in the source.

Example:

A sample and duplicate relative percent difference (RPD) was recalculated using the following equation:

RPD = .[|S-D| /((S-D)/2)] × 100 Where, S = Original sample concentration
D = Duplicate sample concentration

Example:

An ICP serial dilution percent difference (%D) was recalculated using the following equation:

%D = (|I-SDR|/I) × 100 Where, I = Initial Sample Result (µg/L)
SDR = Serial Dilution Result (µg/L) [Instrument Reading × 5]

Example:

Analysis	Found/S/I (units)	True/D/SDR (units)	Recalculated %R/RPD/%D	Reported %R/RPD/%D	Acceptable (Yes/No)
ICP check					
LCS					
MS					
Duplicate					
ICP Serial					

11.0 SAMPLE RESULT VERIFICATION AND OTHER PROBLEMS/COMMENTS

- Were all the sample results reported correctly on the Form 1s? Yes/No
- Are there any transcription or data reduction errors (e.g., dilutions, percent solids, sample weights, dilutions, percent solids, sample weights, or volumes) on one or more samples? Yes/No
- Were all of the sample results corrected for sample weight, sample volume, and dilutions? Yes/No
- Are all of the results within the linear range of the ICP and within the calibration range for the non-ICP parameters? Yes/No
- Are there method non-compliance issues? If so, note below. Yes/No

METHOD NON-COMPLIANCE ISSUES, OTHER PROBLEMS, AND COMMENTS:

APPENDIX C2
SOW ILM05.X WORKSHEET

CASE SUMMARY

Case No.:_____ Site Name:_____

SDG No.:_____ EPA/CERCLIS Site I.D.:_____

Date Received:_____ Due Date:_____ SOW#:_____ TDF#:_____

Laboratory:_____ Lab Code:_____

Region No./CLP PO:_____ Analysis:_____

# of Samples & Matrix: _____ Concentration: Low/Med/High

EPA Project Officer & Title:_____

Section & Mail Code:_____

Sample Numbers:_____

Field and/or Equipment Blanks:_____

Background:_____

Field Duplicates:_____

Validation Parameters	Qualification Code			
	ICP-AES	ICP-MS	Hg	Cyanide
1. PRESERVATION AND HOLDING TIMES				
2. ICP-MS TUNE ANALYSIS	N		N	N
3 CALIBRATION				
4. LABORATORY AND FIELD BLANKS				
5. ICP INTERFERENCE CHECK SAMPLE (ICS)			N	N
6. LABORATORY CONTROL SAMPLE (LCS)				
7. LABORATORY AND FIELD DUPLICATE ANALYSIS				
8. MATRIX SPIKE ANALYSIS				
9. ICP SERIAL DILUTION, ICP LRV, & IDL STUDY				
10. ICP-MS INTERNAL STANDARDS	N		N	N
11. SAMPLE QUANTITATION				
12. SAMPLE VERIFICATION				
13. OVERALL ASSESSMENT				

Qualification Code: A = Acceptable
R = Rejected
J = Estimated (see worksheet)
C = Comment only
N = Not applicable or provided

1.0 PRESERVATION & TECHNICAL HOLDING TIMES

National Functional Guidelines for Inorganic Data Review Technical Holding Time and Preservation Criteria:

	Dates Prepared	Prep Code	Acceptable	Qualification
Metals: 6 months, preserved to pH <2	_____	_____	Yes/No	R/J/None
Mercury: 28 days, preserved to pH <2	_____	_____	Yes/No	R/J/None
Cyanide: 14 days, preserved to pH >12	_____	_____	Yes/No	R/J/None
Cyanide: water samples tested for sulfides and/or oxidizing agents?			Yes/No	R/J/None
Percent Solids: Yes/No If Yes,	_____		Yes/No	R/J/None
Percent Solids: Oven Temp: _____	Drying Time: _____			

Concentration: Low/Med/High Matrix: Soil/Water/Other

Field QC	Sample Number	Collected/ Received	pH/ ^{O}C	Dates Analyzed / Number of Days Exceeded				
				ICP-AES	ICP-MS	Hg	CN	

2.0 ICP-MS INSTRUMENT TUNE

Criteria: Prior to calibration, ICP-MS tuning solution analyzed at least five times consecutively.
The tuning solution contains 100 µg/L of Be, Mg isotopes, Co, In isotopes, and Pb isotopes.

Were the following tune criteria met:	Acceptable?
• ICP-MS tune solution analyzed 5 times consecutively with the appropriate analytes present and tune masses bracket the masses in the target analyte list [If **No**, make comment in CLP PO ACTION and Validity and Comments section of report]	Yes/No/NA
• ICP-MS peak width at 5% of peak height ≤ 0.75 atomic mass units (amu) (or 0.65-0.80 amu at 10% peak height depending on instrument manufactures' requirements) [If **No**, make comment in CLP PO ACTION and Validity and Comments section of report]	Yes/No/NA
• Mass calibration within 0.1 amu for any tuning solution isotope [If **No**, make comment in CLP PO ACTION and Validity and Comments section of report]	Yes/No/NA
• %RSD for any isotope in tuning solution < 5% [If **No**, make comment in CLP PO ACTION and Validity and Comments section of report]	Yes/No/NA

ICP-MS Tune Solution Acceptable: Yes/No

Tune Isotope	peak width @ ___% peak ht.	Isotope Mass	%RSD	Associated Samples	Qualification

Comments/Calculations:

3.0 CALIBRATION

INSTRUMENT CALIBRATION AND CRQL STANDARDS

Criteria: ICP-AES CRQL Standards = 70-130% of True Value (50-150% for Sb, Pb, and Tl)
ICP-MS CRQL Standards = 70-130% of True Value (50-150% for Co, Mn, and Zn)
Hg and cyanide CRQL Standards = 70-130% of True Value

Were the following parameters met:	Acceptable?
• ICP-AES calibration curve: 1 blank, 1 standard	Yes/No/NA
• ICP-MS calibration curve: 1 blank, 1 standard	Yes/No/NA
• Mercury calibration curve: 1 blank, 4 standards (1 at CRQL)	Yes/No/NA
• Cyanide calibration range: 1 blank, 3 standards (1 at CRQL)	Yes/No/NA
• Hg and/or Cyanide correlation coefficients (r) $\geq$ 0.995	Yes/No/NA
• Cyanide mid-range standard distilled and result ± 15% of un-distilled standard	Yes/No/NA
• CRI standard for ICP-AES at CRQL run after the ICV, after 20 analytical samples, and at the end of the run (not required for Al, Ba, Ca, Fe, Mg, Na, and K)	Yes/No/NA
• CRI standard for ICP-MS at CRQL run after the ICV, after 20 analytical samples, and at the end of the run (every isotope used in the analysis)	Yes/No/NA
• CRI standard for Hg and/or cyanide at CRQL run after an ICV, after 20 analytical samples, and at the end of the run	Yes/No/NA
• CRQL calibration standard used for Hg and/or cyanide calibration [If **No**, make comment in CLP PO ATTENTION]	Yes/No/NA

CRI Standards Acceptable: Yes/No

Analyte	r value	CRI %R	Associated Samples	Qualification

Comments/Calculations:

3.1 CALIBRATION

INITIAL CALIBRATION VERIFICATION (ICV) AND CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: Metals (ICP-AES/MS) = 90-110% of True Value; Hg (Cold Vapor) = 80-120% of True Value; Cyanide = 85-115% of True Value [ESAT allows ±1% deviation due to rounding]

Were the following parameters met:	Acceptable?
• ICV prepared from second source and run immediately after a system calibration	Yes/No
• CCVs run at the beginning (after an ICS), every 10 samples or every 2 hours, and at the end of the analysis	Yes/No
• CCV concentration at or near mid-range calibration standard	Yes/No
• ICV, ICB, 1st CCV, and 1st CCB run before 1st samples	Yes/No
• If ICV or CCV %R exceeds control limits, lab stops analysis, recalibrates, and reruns affected samples [If Yes,-make comment in CLP PO ACTION]	Yes/No

ICV/CCVs Acceptable: Yes/No

Analyte	ICV %R	CCV %R	Associated Samples	Qualification

Comments/Calculations:

4.0 LABORATORY AND FIELD BLANKS

ICB = Initial Calibration Blank CCB = Continuing Calibration Blank PB = Preparation Blank
FB = Field Blank EB = Equipment Blank

Criteria: No analyte should be present in any blank associated with an SDG in excess of the CRDL. Samples associated with blanks having concentrations >CRQL must be qualified according to the following:

Sample Concentration	Qualification
>MDL and <5× any blank concentration	Flag as non-detected (UJ)

Any blank reported with a negative result whose absolute value is >CRQL must be carefully evaluated. Use professional judgment to determine its effect on the sample data.

- For results ≥MDL but ≤CRQL, raise results to CRQL with a (U).
- For diluted results <CRQL, raise CRQL by sample dilution factor (CRQL x df) and flag as non-detected (UJ).

Lab Blanks Acceptable: Yes/No/NA
[If **No**, make comment in CLP PO ACTION]

Field Blanks Acceptable: Yes/No/NA
[If **No**, make comment in SAMPLING ISSUES]

Analyte	Blank ID	Conc. in Blank	Associated Samples	Qualification

- Were the blank analysis frequency requirements met? Yes/No
- Was at least one preparation blank prepared and analyzed per matrix for each SDG/preparation batch of 20 or fewer samples? Yes/No

Comments/Calculations:

5.0 ICP-AES/ICP-MS INTERFERENCE CHECK SAMPLE (ICS)

Solution A (ICSA) = Al, Ca, Fe, and Mg (interferents)
Solution B (ICSAB) = ICP analytes mixed with the interferents

Criteria: ICP-**AES**: Solution A and Solution AB concentrations should be 80-120% of the true values or ±**2x** CRQL (whichever is greater), or qualification is necessary.
ICP-**MS**: Solution A and Solution AB concentrations should be 80-120% of the true values or ±**3x** CRQL (whichever is greater), or qualification is necessary.
Solution A must be analyzed before Solution AB. ICS must be analyzed after the ICV/ICB and *followed by a CCV/CCB.*

Solution A should only contain Al, Ca, Fe, and Mg. In general, qualification should be made for interference when false positives or negatives occur for ICP analytes in samples with concentrations of Al, Ca, Fe, and/or Mg which are greater than or equal to the concentrations in ICS solutions A *and AB.*

- If ICS results are outside the control limits, the lab stops analysis, recalibrates, and reruns affected samples.
  If not done, make comment in CLP PO ACTION and Validity and Comments section of report.
- Were the ICS (solutions A and AB) analyzed and reported at the beginning, the end, and after every 20 analytical samples and followed by a CCV/CCB combination? Yes/No

- Was an interferent concentration in the sample greater than or equal to the concentration in the ICS solution or any interfering analyte concentration in the sample greater than 10 mg/L? Yes/No

- If a result for any analyte **not** present in the ICS solution is found at a concentration ≥CRQL **and** interferent concentrations are ≥those in the ICS and analyte concentrations are approximate to those in the ICS, qualify sample results > CRQL as estimated high (J+).
- If a negative result for any analyte **not** present in the ICS solution is found at a concentration ≥CRQL **and** interferent concentrations are ≥those in the ICS, estimate non-detected results (UJ) and qualify sample results ≥CRQL but <10X the absolute value of the negative result as estimated low (J-).

Acceptable: Yes/No

Analyte	ICSA True	ICSA Found	ICSAB True	ICSAB Found	Associated Samples	Qualification

- If an interferent is present at a concentration greater than 120% of the ICS solution concentration, use *the following equation to estimate the affected analyte concentration produced by the interferent:*
  [I] x A_{IEC} = [C_A] where, [I] = Interferent concentration (Al, Ca, Fe, Mg) or analyte concentration >10 mg/L
  A_{IEC} = Analyte Interelement Correction Factor (from Form 11A and/or 11B)
  [C_A] = estimated analyte concentration produced by the interferent.

If [C_A] is > 2x CRQL **AND** > 10% of the reported concentration of the affected analyte, qualify the affected analyte result as estimated (J).

6.0 LABORATORY CONTROL SAMPLE (LCS)

Criteria: Water (LCSW): 80-120% of the true value (except for ICP-AES Sb and Ag results which have no control limits)

Solid (LCSS): Must fall within the control limits established by the EPA (or supplier) for each element.

- The analysis of the laboratory control sample is required for each analyte (except Hg and Cyanide in water samples) using the same sample preparation, analytical methods, and QC procedures employed for the other samples in the SDG.

- The LCSW or LCSS results for target analytes shall fall within the specified acceptance limits. Otherwise, all associated samples shall be re-digested and reanalyzed.
  [If this was not done, make a comment in CLP PO ACTION.]

Matrix: Soil/Water Acceptable: Yes/No/NA

Analyte	True Value	Found Value	%R (LCSW) / EPA Limits (LCSS)	Associated Samples	Qualification

- If the LCSW or LCSS recovery is <50% and no true values are provided, reject associated samples and comment in CLP PO ACTION.

Comments/Calculations:

7.0 LABORATORY AND FIELD DUPLICATES

Criteria: Water: ±20 RPD for sample values > 5× CRQL or ±CRQL for sample values < 5× CRQL.

Soil: ±35 RPD for sample values > 5× CRQL or ±2× CRQL for sample values < 5× CRQL

Background sample data is qualified due to laboratory duplicate outliers.
Field blank, equipment blank, and PE sample data are not qualified due to laboratory duplicate outliers.

- Was a field blank or equipment blank used as duplicate sample? Yes/No
  [If Yes, make comment in CLP PO ACTION.]
- If field Duplicate Results exceed Control Limits, Comment on the Duplicate Pair Involved, but do **not** qualify data.

QC Sample Number:
Lab. Dups. Acceptable: Yes/No Field Dups. Acceptable: Yes/No/NA

Analyte	RPD or CRQL	Field Dup Sample	Associated Samples	Qualification

Comments/Calculations:

8.0 MATRIX SPIKE ANALYSIS

Criteria: Apply matrix spike (MS) percent recovery (%R) control limits of 75-125%, except when sample concentration exceeds 4× spike concentration. Note that the post digest spike %R result is not used to estimate sample data.

Background sample data is qualified due to MS outliers.
Field and equipment blank and PE sample data are not qualified due to MS outliers.

- Was a matrix spike analysis performed on each group of samples of similar matrix type and concentration or for each SDG? Yes/No
  [If **No**, make comment in CLP PO ACTION.]

- Was correct spike concentration added to sample before the digestion or distillation step? Yes/No
  [If **No**, make comment in CLP PO ACTION.]

- Was a post-digestion/post-distillation spike performed for ICP-AES/ICP-MS and CN? Yes/No/NA
  [Note that a post-digest spike is **not** required for ICP-AES Ag and Hg.]

- Did all required post-digest spikes meet the required criteria? Yes/No

QC Sample Number(s): Acceptable: Yes/No

Analyte	M-Spike %R	A-Spike %R	Associated Samples	Qualification

- Was the QC sample specified on the chain of custody used in the analysis performed? Yes/No

- Was field or equipment blank used as matrix spike sample? Yes/No
  [If Yes, make comment in CLP PO ACTION.]

- Were the ICP spiking levels specified in Table(s) 2 and/or 5 of the SOW followed? Yes/No

- Were the Hg and/or CN spiking levels specified in the SOW followed? Yes/No/NA
  (Levels: Hg: 1 μg/L (water), 0.5 mg/Kg (soil); CN: 100 μg/L (final conc.)

Comments/Calculations:

9.0 ICP SERIAL DILUTION ANALYSIS, ICP LRV, AND MDL STUDY

Field blank, equipment blank, and PE sample data are not qualified due to ICP serial dilution outliers.

- Was an ICP serial dilution (a five fold dilution) analysis performed on the sample from a group of samples from a similar matrix and concentration or for each SDG, whichever is more frequent? Yes/No

- Was the serial dilution within 10% of the original result after correction for dilution for analytes with concentrations that are greater that 50× the IDL in the original sample? Yes/No/NA
  [If serial dilution sample was **not** analyzed, note in Additional Comments.]

QC Sample Number: Acceptable: Yes/No

Analyte	% Difference	Associated Samples	Qualification

ICP Linear Range Verification (LRV) and Method Detection Limit (MDL) Study:

- Was the ICP LRV performed at least quarterly (every 3 calendar months) for each analyte reported? Yes/No

- Was the MDL determined at least annually (every 12 calendar months) for each analyte reported on each instrument used? Yes/No

- Were the MDLs correctly reported on Form 9? Yes/No

- Does instrument ID on Forms 9 through 13 match instrument ID used for sample analysis? Yes/No

Comments/Calculations:

10.0 ICP-MS INTERNAL STANDARDS

Criteria: All samples analyzed during a run, except the ICP-MS tune solution, must contain a minimum of five internal standards. The internal standard response intensity in the samples is compared to the internal standard response intensity in the calibration blank. The percent relative intensity (%RI) on Form 15 for must be within 60-125% of the calibration blank response.

- Were a minimum of 5 specified internal standards added to all samples, except the tune solution? Yes/No
  Specified internal standards: Li^6 (enriched), Sc, Y, Rh, In^{115}, Tb, Ho, Lu, and Bi
  [If **No**, qualify data as rejected (R) and comment in CLP PO ACTION and Validity and Comments section of report.]

- Do the internal standard masses bracket the target analyte masses? Yes/No
  [If **No**, qualify target analyte data not bracketed by internal standards as rejected (R) and comment in CLP PO ACTION and Validity and Comments section of report.]

Internal Standards Acceptable: Yes / No

Internal Standard	% RI	Affected Analyte and Associated Samples	Qualification

Comments/Calculations:

11.0 VALIDATION FINDINGS AND RECALCULATIONS

Percent recoveries (%R) for an ICP interference check sample, laboratory control sample, and a matrix spike sample were recalculated using the following equation:

%R = (Found/True) × 100 Where: Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, Found = SSR *(spike sample result)-SR (sample result).*
True = Concentration of each analyte in the source.

Example:

A sample and duplicate relative percent difference (RPD) was recalculated using the following equation:

RPD = [|S-D| /((S-D)/2)] × 100 Where: S = Original sample concentration
D = Duplicate sample concentration

Example:

An ICP serial dilution percent difference (%D) was recalculated using the following equation:

%D = (|I-SDR|/I) × 100 Where: I = Initial Sample Result (µg/L)
SDR = Serial Dilution Result (µg/L) [Instrument Reading × 5]

Example:

Analysis	Found/S/I (units)	True/D/SDR (units)	Recalculated %R/RPD/%D	Reported %R/RPD/%D	Acceptable (Yes/No)
ICP check					
LCS					
MS					
Duplicate					
ICP Serial					

12.0 SAMPLE RESULT VERIFICATION AND OTHER PROBLEMS/COMMENTS

- Were all the sample results reported correctly on the Form 1s? Yes/No
- Are there any transcription or data reduction errors (e.g., dilutions, percent solids, sample weights, dilutions, percent solids, sample weights, or volumes) on one or more samples? Yes/No
- Were all of the sample results corrected for sample weight, sample volume, and dilutions? Yes/No
- Are all of the results within the linear range of the ICP and within the calibration range for the non-ICP parameters? Yes/No
- Are there method non-compliance issues? If so, note below. Yes/No

METHOD NON-COMPLIANCE ISSUES, OTHER PROBLEMS, AND COMMENTS: