

V. ICP-AES INTERFERENCE CHECK SAMPLE (ICS)

A. OBJECTIVE

Interference Check Samples (ICSs) in ICP-AES are analyzed to verify the instrument's ability to correct for spectral interferences using correction factors that correct for interelement contributions. Interference Check Samples contain known concentrations of interferents that will provide a test of the interelement correction factors. These interelement correction factors are unique to each instrument and its operating conditions and are determined experimentally.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. Interelement correction factors must be determined for each ICP-AES instrument at the method-required frequency and, at a minimum, annually.
2.
 - a. The ICS solutions must contain interferents and analytes specified in the method. Typically, the ICS consists of two solutions. The ICSA solution contains the interferents, and the ICSAB solution contains the analytes mixed with the interferents.
 - b. The ICS solutions should be from a source providing certified solutions, or prepared by the laboratory at interferent and analyte concentrations specified in the method.
 - c. The ICS solutions must be analyzed at the method-required frequency and, minimally, once within each analytical run after the initial calibration verification (ICV) but prior to sample analysis.
3.
 - a. The results of all target analytes in the ICSAB solution must be within 80-120 percent of the ICSAB true value.
 - b. The results of all target analytes in the ICSA solution must be within 80-120 percent of the ICSA true value or within the ICSA true value $\pm 2x$ the MDL, whichever range of control limits is greater.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
<p>1. Verify that interelement correction factors were determined for each ICP-AES instrument at the frequency specified in the method and at least annually.</p>	<p>All potential impacts on the sample data resulting from ICS anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.</p> <p>1. If interelement correction factors were not determined at the proper frequency, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected. The validator should take into consideration the results of the ICS.</p>
<p>2. a. Verify that the ICSA and ICSAB solutions contain the method-required target analytes and interferents. Common interferents aluminum, iron, calcium, and magnesium should be in the ICS solutions. Other interferents may be included in the ICS as required by the method.</p> <p>b. Verify that the ICS solutions were obtained from a source providing certified solutions. If the ICS solutions were prepared by the laboratory, verify that the method-required concentrations of analytes and interferents were used.</p> <p>c. Verify that the ICS solutions were analyzed at the method-required frequency and, minimally, once within each analytical run. The ICS solutions should be analyzed at the beginning of the run after the ICV but before sample analysis.</p>	<p>2. If the ICS solutions were not analyzed for the required analytes and interferents at the correct frequency or were not obtained from a certified source, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected. A discussion of the rationale for data qualification and qualifiers used should be documented in the Data Validation Memorandum.</p>

C. EVALUATION	D. ACTION
<p>3. Evaluate the recoveries of all analytes in the ICSAB solution. Verify that all ICSAB recoveries are within 80-120% of the ICSAB true value.</p>	<p>Note: In general, action regarding unacceptable ICSA and ICSAB results is taken when interferences in the ICS are found in the sample at concentrations greater than 50% of their respective concentrations in the ICS. Professional judgment should be used to apply actions when more than one interferent is present in the sample. Generally, the interferent yielding the greatest interference effect is used as the worst case scenario. Generally, no action is taken if the sample contains interferences at concentrations less than or equal to 50% of their respective concentrations in the ICS.</p> <p>3. a. If any ICSAB percent recovery is greater than 120% but less than or equal to 150%, then the validator should:</p> <ul style="list-style-type: none"> i. Estimate (J) positive detects for the affected analyte in all samples associated with the ICS. ii. Accept non-detects for the affected analyte in all samples associated with the ICS.

C. EVALUATION	D. ACTION
3. Continued from above.	<p>3. b. If any ICSAB percent recovery is greater than 150%, then the validator should reject (R) positive detects and accept non-detects for the affected analyte in all samples associated with the ICS. Professional judgment may be used to estimate (J) positive detects, taking into consideration project DQOs.</p> <p>c. If any ICSAB percent recovery is less than 80% but greater than or equal to 50%, then the validator should:</p> <ul style="list-style-type: none">i. Estimate (J) positive detects for the affected analyte in all samples associated with the ICS.ii. Estimate (UJ) non-detects for the affected analyte in all samples associated with the ICS. <p>d. If any ICSAB percent recovery is less than 50%, then the validator should reject (R) positive detects and non-detects for the affected analyte in all samples associated with the ICS.</p>

C. EVALUATION	D. ACTION
<p>Note: The CLP SOW ILM05.4 ICS method QC acceptance criteria differ from the Region I Functional Guidelines ICS criteria. If data quality objectives allow for greater variability of data, then expanded ICS validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP. If greater variability is allowed, then the validator should ensure that there is sufficient QC data to support the use of the expanded criteria.</p> <p>4. a. Evaluate the results of all analytes in the ICSA solution. Verify that the ICSA result is within 80-120% of the ICSA true value or within the ICSA true value \pm 2x the MDL, whichever range of control limits is greater.</p>	<p>Note: The validator should evaluate the magnitude of the interference effects caused by the levels of interferents in the ICS compared to the potential estimated interference effect which may be caused by the levels of interferents in the sample. In some cases, the validator may determine that the level of interferent present in the sample does not warrant data qualification if the respective estimated interference effect is deemed not to have an impact on sample results. Generally, if the estimated interference is less than 10% of the sample concentration of the affected analyte, then no action is applied and the sample result is accepted without qualification. If the estimated interference is greater than 10% of the reported sample concentration of the affected analyte, then validation actions apply. Certain circumstances may warrant rejection (R) of the data if it cannot be determined whether or not the sample result is due entirely to interferences or if the validator determines that estimated interferences comprise greater than 90% of the sample result. Professional judgment should be used to reject, qualify, or accept sample results. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p> <p>4. a. For any ICSA result, positive or negative, which is not within 80-120% if the ICSA true value or within the ICSA true value \pm 2x the MDL, whichever range is greater, the following actions should be taken:</p> <p>i. If any ICSA result is greater than 120% of the ICSA true value or is greater than the ICSA true value plus 2x the MDL, whichever value is greater, then the validator should:</p> <ul style="list-style-type: none"> • Estimate (J) positive detects for the affected analyte in all samples associated with the ICSA to indicate potential high bias.

C. EVALUATION	D. ACTION
<p>4. c. For any ICSA analyte which does not meet criteria, evaluate whether or not the ICSAB criteria were met.</p>	<p>4. c. If any analyte does not meet acceptance criteria for the ICSA but meets ICSAB criteria, then the validator may use professional judgment to qualify only those sample results in the affected concentration range, taking into consideration the project DQOs. For example, if a false positive/high bias or false negative/low bias is observed in the ICSA, but the ICSAB result for the analyte is within 80-120% recovery criteria, then the validator may use professional judgment to qualify only those sample results less than the ICSAB true value and accept the sample results greater than or equal to the ICSAB true value. A discussion of the rationale for data qualification or data acceptance should be documented in the Data Validation Memorandum.</p> <p>d. The validator should review the sample data to determine if other analytes which may act as potential interferents are present in the sample at greater than 10 mg/L. Possible interference effects, as documented in EPA methods, including the CLP method, and as indicated by the laboratory's reported interelement correction factors for that particular instrument should be reviewed. The analyte concentration equivalents presented in the methods should only be used as estimated values since the exact value of any analytical system is instrument-specific. Professional judgment should be used to qualify sample data if the validator suspects interference effects other than those arising from the interferents in the ICS.</p>

C. EVALUATION	D. ACTION
<p>4. Continued from above.</p>	<p>4. e. Evaluating ICS data is not necessarily straightforward. When multiple ICS analyses exist within an analytical run, the validator may use professional judgment to apply actions only to those samples bracketed by a particular ICS or to apply actions from the worst case scenario to all samples within that run. If multiple ICS analyses within an analytical run exhibit both positive and negative values for a particular analyte, then the validator should use professional judgment in qualifying sample results. It may help to evaluate the results of blanks analyzed within the same run to determine whether the positive or negative ICS values may be due to erratic responses also exhibited in blanks.</p> <p>f. Actions regarding the interpretation and/or the qualification of sample results due to ICS results can be complex. The validator should use professional judgment in determining the need to accept, qualify or reject the associated sample data based on the ICS results and project DQOs. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p>

C. EVALUATION	D. ACTION
<p>*5. a. Check raw data to verify that ICS results are accurately reported on the tabulated forms. Confirm that results equal to or below the negative MDL are also reported on the forms.</p> <p>b. Check and recalculate the ICS percent recovery for at least one analyte per each pair of ICSA/ICSAB. Verify that the recalculated value agrees within $\pm 10\%$ of the reported value.</p>	<p>5. If there are any transcription and/or calculation errors, then the validator should contact the laboratory to obtain corrected raw data and forms. If errors greater than 10% are detected, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p>

* Note: The following subsection is applicable only to a Tier III data validation:

C.5

Table INORG-V-1:

**QUALIFICATION OF ICP-AES ANALYTES BASED ON INTERFERENCE CHECK SAMPLE
ICSAB RECOVERIES**

Sample Results	ICSAB Recoveries				
	%R < 50%	50% ≤ %R < 80%	80% ≤ %R ≤ 120%	120% < R ≤ 150%	%R > 150%
Detects	R	J	A	J	R*
Non-detects	R	UJ	A	A	A

* Professional judgment may be used to estimate (J) positive detects, taking into consideration project DQOs. Note: Generally, action is applied when interferents are present in samples at greater than 50% of their respective levels in the ICS.

Table INORG-V-2:

**QUALIFICATION OF ICP-AES ANALYTES BASED ON INTERFERENCE CHECK SAMPLE
ICSA RESULTS**

Sample Results	ICSA Concentration or % Recovery*		
	%R < 80% or Conc. < TV-(2xMDL)	%R = 80-120% or Conc. = TV±(2xMDL)	%R > 120% or Conc. > TV+(2xMDL)
Detects	J	A	J
Non-detects	UJ	A	A

TV = ICSA True Value

* ICSA criteria are based on either 80-120% of the ICSA true value or the ICSA true value ± 2x the MDL, whichever range of control limits is greater.

Note: Generally, action is applied when interferents are present in samples at greater than 50% of their respective levels in the ICS. Generally, no action is taken when the estimated interference is less than 10% of the sample result of the affected analyte. Professional judgment may be used to reject the data if the estimated interference comprises greater than 90% of the sample result. If ICSAB recovery criteria are met, then professional judgment may be used to qualify only those sample results in the affected concentration range.

E. EXAMPLES

Example #1: (Low ICSAB recovery)

The ICSAB recovery for antimony was 70.5%. All samples associated with the ICSAB contained levels of aluminum, calcium, iron, and magnesium which were less than 50% of their respective levels in the ICSAB, with the exception of sample MAGF02 which contained iron at a concentration greater than 50% of the level detected in the ICSAB solution. Antimony was not detected in sample MAGF02. The validator estimates (UJ) the antimony non-detect on the Data Summary Table to indicate the possibility of a false negative. The validator notes the problem in the Data Validation Memorandum.

Example #2: (Cadmium ICSA TV is 0; ICSA result > ICSA TV \pm 2x the MDL)

The ICSA true value for cadmium is 0. The MDL for cadmium is 2 ug/L. Cadmium was reported in the ICSA at 6 ug/L which exceeds the criteria of the ICSA true value \pm 2x the MDL (0 ± 4 ug/L). All samples associated with the ICSA contained levels of interferents that were less than 50% of their respective levels in the ICSA, with the exception of samples MACB01 and MACB02. Iron concentrations in these two samples were greater than 50% of that in the ICSA.

- Sample MACB01 contains cadmium at 10 ug/L. Since 100,000 ug/L of iron yielded 6 ug/L of cadmium in the ICSA, 70,000 ug/L of iron in sample MACB01 is expected to yield a potential estimated interference of 4 ug/L. The validator estimates (J) the positive cadmium result in sample MACB01 on the Data Summary Table to indicate potential high bias.
- Sample MACB02 contains cadmium at 50 ug/L. The iron concentration of 60,000 ug/L in sample MACB02 is expected to yield a potential estimated interference of 3.6 ug/L which is less than 10% of the reported cadmium concentration in the sample. The validator accepts the positive cadmium result in sample MACB02.

The validator discusses all sample qualifications in the Data Validation Memorandum.

E. EXAMPLES (continued)Example #3: (High lead ICSA result)

The ICSA true value for lead is 15 ug/L. The MDL for lead is 4 ug/L. The validator uses the criteria of the ICSA true value $\pm 2x$ the MDL (7-23 ug/L) rather than 80-120% recovery (12-18 ug/L) in evaluating the ICSA data since $\pm 2x$ the MDL yields wider criteria. Lead was detected in the ICSA at 39 ug/L which exceeds the ICSA true value $\pm 2x$ the MDL criteria. All samples associated with the ICSA contained levels of aluminum, calcium, iron, and magnesium which were less than 50% of their respective levels in the ICSA, with the exception of three samples. Samples MAGF02, MAGF04, and MAGF07 contained concentrations of aluminum which were greater than 50% of that in the ICSA.

- Lead was not detected in sample MAGF02. The validator accepts the lead non-detect in sample MAGF02.
- Sample MAGF04 contains lead at 150 ug/L. Since 250,000 ug/L of aluminum yielded 24 ug/L above the true value of lead in the ICSA, 150,000 ug/L of aluminum in sample MAGF04 is expected to yield a potential estimated interference of approximately 14.4 ug/L. Since the estimated interference comprises less than 10% of the reported sample result of 150 ug/L, no action is taken and the validator accepts the positive lead result on the Data Summary Table.
- Sample MAGF07 contains lead at 20 ug/L. Since 250,000 ug/L of aluminum yielded 24 ug/L above the true value of lead in the ICSA, 200,000 ug/L of aluminum in sample MAGF07 is expected to yield a potential estimated interference of approximately 19.2 ug/L. Therefore, the validator rejects (R) the positive lead results in sample MAGF07 on the Data Summary Table since the result is suspected of being due entirely to interference.

The validator discusses all sample qualifications in the Data Validation Memorandum.

Example #4: (Antimony ICSA TV is 0; Negative antimony ICSA result)

The ICSA true value for antimony is 0. The antimony MDL is 14 ug/L. Antimony was reported in the ICSA at -41 ug/L, outside the criteria of the ICSA true value $\pm 2x$ the MDL (0 ± 28 ug/L). The levels of interferents aluminum, iron, calcium, and magnesium in the samples associated with the ICSA are below 50% of their respective levels in the ICSA solution with the exception of iron in samples MAGF02 and MAGF03, which was present in these samples at levels greater than 50% of that in the ICSA.

- Antimony was reported as a non-detect in sample MAGF02. The validator estimates (UJ) the antimony non-detect in sample MAGF02 on the Data Summary Table due to the possibility of a false negative.
- Sample MAGF03 contains antimony at 65 ug/L. Since 100,000 ug/L iron yielded -41 ug/L of antimony in the ICSA, 80,000 ug/L of iron in sample MAGF03 is expected to yield a potential estimated interference of -33 ug/L. Therefore, the validator estimates (J) the positive antimony result in sample MAGF03 on the Data Summary Table and notes the possibility of low bias.

The validator discusses all sample qualifications in the Data Validation Memorandum.

VI. ICP-MS INTERFERENCE CHECK SAMPLE (ICS)

A. OBJECTIVE

ICP-MS Interference Check Samples (ICSs) contain interfering elements that provide a test of the adequacy of the measurement system to correct for isobaric interferences. Interference Check Samples are analyzed to determine the existence and magnitude of interferences and to verify the instrument's ability to correct for those interferences.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1.
 - a. The ICS solutions must contain interferents and analytes specified in the method. Typically, the ICS consists of two solutions. The ICSA solution contains the interferents, and the ICSAB solution contains the analytes mixed with the interferents.
 - b. The ICS solutions should be from a source providing certified solutions or prepared by the laboratory at interferent and analyte concentrations specified in the method.
 - c. The ICS must be analyzed at the method-required frequency and, minimally, once within each analytical run after the initial calibration verification (ICV) but prior to sample analysis.
2.
 - a. The results of all target analytes in the ICSAB solution must be within 80-120 percent of the ICSAB true value.
 - b. The results of all target analytes in the ICSA solution must be within 80-120 percent of the ICSA true value or within the ICSA true value $\pm 2x$ the MDL, whichever range of control limits is greater.

C. EVALUATION/D. ACTION

C. EVALUATION	D. ACTION
<p>1. a. Verify that the method-required ICSEA and ICSEB solutions were analyzed at the proper frequency and, minimally, once within each analytical run.</p> <p>* b. Verify that the ICS solutions were obtained from a source providing certified solutions. If the ICS solution was prepared by the laboratory, verify that the required concentrations of analytes and interferences were used.</p> <p>Note: The tabulated forms may not contain results for all ICS interferences. It may be necessary to review the raw data (instrument raw data or standards preparation logs) to confirm that the proper components were used to prepare the ICS solutions. Note that monitoring the interference source in ICP-MS analysis does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent.</p>	<p>All potential impacts on the sample data resulting from ICS anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.</p> <p>1. a. If the ICS solutions were not analyzed at the method-required frequency, then the validator should use professional judgment to assess the impact of potential interferences on data quality. A discussion of the rationale for data qualification of the qualifiers used should be documented in the data validation memorandum.</p> <p>b. If the ICS solution was not obtained from a source providing certified solutions or if the required analytes and interferences were not used to prepare the ICS solutions at the required concentrations, then the validator should use professional judgment to determine if sample qualification is required.</p>

C. EVALUATION	D. ACTION
<p>2. Evaluate the recoveries of all analytes in the ICSAB solution. Verify that all ICSAB recoveries are within 80-120% of the ICSAB true value.</p>	<p>2. a. If any ICSAB percent recovery is greater than 120% but less than or equal to 150%, then the validator should:</p> <ul style="list-style-type: none">i. Estimate (J) positive detects for the affected analyte in all samples associated with the ICS.ii. Accept non-detects for the affected analyte in all samples associated with the ICS. <p>b. If any ICSAB percent recovery is greater than 150%, then the validator should use professional judgment to estimate (J) or reject (R) positive detects and accept non-detects for the affected analyte in all samples associated with the ICS.</p> <p>c. If any ICSAB percent recovery is less than 80% but is greater than or equal to 50%, then the validator should:</p> <ul style="list-style-type: none">i. Estimate (J) positive detects for the affected analyte in all samples associated with the ICS.ii. Estimate (UJ) non-detects for the affected analyte in all samples associated with the ICS. <p>d. If any ICSAB percent recovery is less than 50%, then the validator should reject (R) positive detects and non-detects for the affected analyte in all samples associated with the ICS.</p>

C. EVALUATION	D. ACTION
<p>Note: The CLP SOW ILM05.4 ICS method QC acceptance criteria differ from the Region I Functional Guidelines ICS criteria. If data quality objectives allow for greater variability of data, then expanded ICS validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP. If greater ICS recoveries are allowed, then the validator should ensure that there is sufficient QC data to support the use of the expanded criteria.</p> <p>3. a. Evaluate the results of all analytes in the ICSA solution. Verify that the ICSA result is within 80-120% of the ICSA true value or within the ICSA true value \pm 2x the MDL, whichever range of control limits is greater.</p>	<p>3. a. For any ICSA analyte result, positive or negative, which is not within 80-120% of the ICSA true value or within the ICSA true value \pm 2x the MDL, whichever range is greater, the following actions should be taken:</p> <p>i. If any ICSA result is greater than 120% of the ICSA true value or is greater than the ICSA true value plus 2x the MDL, whichever value is greater, then the validator should:</p> <ul style="list-style-type: none"> • Estimate (J) positive detects for the affected analyte in all samples associated with the ICSA to indicate potential high bias. • Accept non-detects for the affected analyte in all samples associated with the ICSA.

C. EVALUATION	D. ACTION
<p>3. a. Continued from above.</p> <p>b. Evaluate the extent of the deviation of the ICSA result from the ICSA true value for the analyte.</p> <p>c. For any ICSA analyte which does not meet criteria, evaluate whether or not the ICSAB criteria were met.</p>	<p>3. a. ii. If any ICSA result (positive or negative) is less than 80% of the ICSA true value or is less than the ICSA true value minus 2x the MDL, whichever value is lower, then the validator should:</p> <ul style="list-style-type: none"> • Estimate (J) positive detects for the affected analyte in all samples associated with the ICSA to indicate potential low bias. • Estimate (UJ) non-detects for the affected analyte in all samples associated with the ICSA. <p>b. Depending upon the extent of the deviation of the ICSA result, the validator may use professional judgment to take further action, taking into consideration the project DQOs. It may be necessary to reject (R) positive detects and/or non-detects for the affected analyte in samples associated with that ICS.</p> <p>c. If any analyte does not meet acceptance criteria for the ICSA but meets ICSAB criteria, then the validator may use professional judgment to qualify only those sample results in the affected concentration range, taking into consideration the project DQOs. For example, if a false positive/high bias or false negative/low bias is observed in the ICSA, but the ICSAB result for the analyte is within recovery criteria, then the validator may use professional judgment to qualify only those sample results less than the ICSAB true value and accept the sample results greater than or equal to the ICSAB true value. A discussion of the rationale for data qualification or data acceptance should be documented in the Data Validation Memorandum.</p>

C. EVALUATION	D. ACTION
3. Continued from above.	3. d. Actions regarding the interpretation and/or the qualification of sample results due to ICS results can be complex. The validator should use professional judgment in determining the need to accept, qualify or reject the associated sample data based on the ICS results and project DQOs. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
<p>*4. a. Check raw data to verify that ICS results are accurately reported on the tabulated forms. Confirm that results equal to or below the negative MDL are also reported on the forms.</p> <p>b. Check and recalculate the ICS percent recovery for at least one analyte per each pair of ICSA/ICSAB. Verify that the calculated value agrees within $\pm 10\%$ of the reported value.</p>	4. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note:** The following subsections are applicable only to a Tier III data validation:

C.1.b, C.4

Table INORG-VI-1:

**QUALIFICATION OF ICP-MS ANALYTES BASED ON INTERFERENCE CHECK SAMPLE
ICSAB RECOVERIES**

Sample Results	ICSAB Recoveries				
	%R < 50%	50% ≤ %R < 80%	80% ≤ %R ≤ 120%	120% < %R ≤ 150%	%R > 150%
Detects	R	J	A	J	R*
Non-detects	R	UJ	A	A	A

* Professional judgment should be used to estimate (J) positive detects, taking into consideration project DQOs.

Table INORG-VI-2:

**QUALIFICATION OF ICP-MS ANALYTES BASED ON INTERFERENCE CHECK SAMPLE
ICSA RESULTS**

Sample Results	ICSA Concentration or % Recovery*		
	%R < 80% or Conc. < TV-(2xMDL)	%R = 80-120% or Conc. = TV±(2xMDL)	%R > 120% or Conc. > TV+(2xMDL)
Detects	J	A	J
Non-detects	UJ	A	A

TV = ICSA True Value

* ICSA criteria are based on either 80-120% of the ICSA true value or the ICSA true value ± 2x the MDL, whichever range of control limits is greater.

Note: Professional judgment may be used to reject (R) positive detects and/or non-detects for the affected analytes.

E. EXAMPLESExample #1: (One low ICSAB recovery)

The validator notes that the ICSAB recovery for cadmium is 70.5%. In the associated samples, cadmium was detected in samples MAFG02 through MAFG05 and was a non-detect in sample MAFG06. Therefore, the validator estimates (J) the positive detects for cadmium and estimates (UJ) the cadmium non-detect in the samples associated with the ICSAB. The validator reports the qualified results on the Data Summary Table and notes that the detected cadmium results are biased low and the cadmium non-detect contains a possible false negative in the Data Validation Memorandum.

Example #2: (One high ICSA result; ICSAB within acceptance limits)

The observed ICSA concentration for manganese is 9.0 ug/L. The ICSA true value for manganese is 7.0 ug/L. The MDL for manganese is 0.3 ug/L. The validator uses the 80-120% R criteria (5.6 to 8.4 ug/L) rather than the ICSA true value $\pm 2x$ the MDL criteria (6.4 to 7.6 ug/L) since the recovery criteria gives wider control limits. The ICSA for manganese recovered at 129%, outside the acceptance limit. In the associated samples, manganese was detected in samples MAGF02 through MAGF04 and was a non-detect in samples MAGF05 through MAGF07. The ICSAB result for manganese was within 80-120% R criteria. Therefore, the validator accepts the manganese non-detects and uses professional judgment to estimate (J) only those positive detects less than the ICSAB true value since manganese was within criteria for the ICSAB.

Example #3: (One negative ICSA result)

The ICSA true value for arsenic is 0 and the arsenic MDL is 0.5 ug/L. The validator notes that arsenic in the ICSA is reported as -2.0 ug/L, which is outside the ICSA true value $\pm 2x$ the MDL criteria (-1.0 to 1.0 ug/L). In the associated samples, arsenic was detected in samples MAFG01 and MAFG02, and was a non-detect in sample MAFG03. The ICSAB result for arsenic was within the 80-120% recovery criteria. Therefore, the validator estimates (UJ) the arsenic non-detect in sample MAFG03, and uses professional judgment to estimate (J) only those positive detects less than the ICSAB true value for samples MAFG01 and MAFG02 since arsenic was within criteria for the ICSAB. The validator reports the qualified data on the Data Summary Table and notes in the Data Validation Memorandum that the qualified positive results are biased low and the arsenic non-detect may be a possible false negative.

VII. ICP-MS INTERNAL STANDARDS

A. OBJECTIVE

Instrument performance and stability, physical/sample matrix interferences, and laboratory precision throughout an analytical sequence are monitored by the addition of internal standard analytes. Internal standards (ISs) are added to every field sample, QC sample, standard and blank at identical levels to determine the existence and magnitude of instrument drift and physical interferences. Evaluation of the behavior of internal standards is not necessarily straightforward. Interfering sample matrix effects are frequently outside of the laboratory's control and may adversely affect the analysis of internal standards.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. The internal standard analytes specified in the method must be added to all samples, QC samples, standards and blanks at the required concentrations. An appropriate internal standard analyte is required for each target analyte determined by ICP-MS. Generally, the atomic mass of each internal standard is within 50 amu of the mass of its associated target analyte.
2. The intensities of the internal standards must be monitored throughout the analytical run and compared to their respective intensities in the blank calibration standard (from the initial calibration). Internal standard intensities must be within the method QC acceptance criteria.
3. Samples must be reanalyzed at the method-required dilutions if internal standard method QC acceptance criteria are not met.

C. EVALUATION/D. ACTION

C. EVALUATION	D. ACTION
<p>1. Verify that the correct internal standard analytes were added to all samples, QC samples, standards and blanks at the method-specified concentrations. (Internal standards are not added to the tuning solution.) Verify that there is an internal standard associated with each target analyte.</p>	<p>All potential impacts on the sample data resulting from internal standard anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.</p> <p>1. If the laboratory did not add the required internal standard analytes to all samples, QC samples, standards and blanks at the correct concentrations, or if there is no internal standard associated with each target analyte, then the validator must use professional judgment to determine how the associated sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p>
<p>2. Verify that all IS percent relative intensities are within the method QC acceptance criteria.</p>	<p>2. a. If the percent relative intensity of an IS is not within the method QC acceptance criteria, then the validator should estimate (J) all positive detects and estimate (UJ) all non-detects for the analytes associated with that IS in the affected sample.</p> <p>b. If internal standard performance exhibits a major deviation from method QC criteria, indicating a severe loss of sensitivity or possible interferences, then the validator may use professional judgment to reject the associated sample data. Other relevant QC information should be taken into account. The rationale for data qualification and the qualifiers used should be discussed in the Data Validation Memorandum.</p>

C. EVALUATION	D. ACTION
<p>3. a. Verify that if any internal standard relative intensity is outside the method QC acceptance criteria, then the method-required reanalysis was performed on the diluted sample with the appropriate amounts of internal standards and/or the method-required recalibration was performed.</p> <p>b. If there are two analyses for a particular sample and analyte, then the validator must determine which data to report. Considerations should include but are not limited to:</p> <ul style="list-style-type: none"> - Magnitude of the IS intensity shift; - Comparison of the values of the target analytes reported in each analysis; - Other relevant QC; - Project DQOs. 	<p>3. a. If a laboratory fails to reanalyze a sample with an internal standard that is outside the method QC acceptance criteria or fails to recalibrate the instrument, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this method deviation/contractual deficiency in the Data Validation Memorandum.</p> <p>b. If a sample has been analyzed and reported more than once, then the validator should use professional judgment when considering which analysis or portion of an analysis to report. The validator must consider all relevant QC information in making a decision. Generally, the following actions should be taken:</p> <ul style="list-style-type: none"> i. If the relative intensity of an internal standard in the diluted sample analysis does not meet the method QC acceptance criteria, then the data from the original undiluted sample analysis should be reported qualified according to the guidelines above. ii. If the relative intensity of an internal standard in the diluted sample analysis meets the method QC acceptance criteria, then the data from the diluted sample analysis should be reported without qualification. iii. The validator should review the project DQOs and determine which sample result better achieves project objectives. The validator should document and discuss all technical decisions made based on professional judgment in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
<p>3. c. Verify that all IS percent relative intensities in the calibration verification solutions (ICV/CCV) and calibration blanks (ICB/CCB) are within 80-120%.</p>	<p>3. c. Evaluate the percent relative intensities of internal standards in the ICV/CCV and ICB/CCB QC samples. If the percent relative intensity of an IS in any of these QC samples is outside 80-120% of the percent relative intensity of the IS in the blank calibration standard, then this may indicate an instrumental problem. Professional judgment should be used to evaluate the impact of the QC sample IS results on the IS results in the field samples. A discussion of the rationale for all data qualifications should be documented in the Data Validation Memorandum.</p>
<p>*4. Check raw data to verify that IS relative intensities are accurately reported on the tabulated forms. Recalculate the internal standard percent relative intensities for each internal standard in at least one sample. Verify that the recalculated value agrees within $\pm 10\%$ of the reported value.</p>	<p>4. If errors greater than 10% are detected in the percent relative intensity calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p>

* Note: The following subsection is applicable only to a Tier III data validation:

C.4

Table INORG-VII-1:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON INTERNAL STANDARD
RELATIVE INTENSITIES**

Sample Results	Internal Standard Relative Intensities (RI)*		
	% RI < LL	LL ≤ % RI ≤ UL	% RI > UL
Detects	J	A	J
Non-detects	UJ	A	UJ

LL = Lower Limit of method QC acceptance criteria

UL = Upper Limit of method QC acceptance criteria

* Professional judgment may be used to reject data for severe loss of sensitivity.

E. EXAMPLES

Example #1: (Instrument drift; Sample IS % RI < LL of method QC acceptance criteria)

The validator reviews the IS % relative intensities for samples analyzed by CLP SOW ILM05.4 and notes that the yttrium response decreases over time and the response in sample MAAB04 is 40%, below the lower method QC acceptance limit of 60% of the response in the blank calibration standard. Upon review of the data, the validator determines that the laboratory did not perform the required reanalysis of the sample at a two-fold dilution. The validator ascertains from the data that arsenic (mass 75), selenium (mass 82), and zinc (mass 66) were associated with the yttrium IS (mass 89). Therefore, on the Data Summary Table, the validator estimates (J) positive detects and estimates (UJ) non-detects in sample MAAB04 for arsenic, selenium, and zinc. The validator discusses the instrument's loss in sensitivity, the sample qualifications, and the laboratory's deviation from the method in the Data Validation Memorandum.

Example #2: (Sample IS % RI > UL of method QC acceptance criteria; Acceptable IS % RI from two-fold dilution)

The validator reviews the IS % relative intensities for samples analyzed by CLP SOW ILM05.4 and notes that the rhodium and indium IS intensities increase over time. In addition, the validator notes that sample MA1GN8 was reported from a two-fold dilution. The QC data indicate that the IS % relative intensities for rhodium and indium for sample MA1GN8 in the original undiluted sample analysis are above the upper method QC acceptance limit of 125% of the IS response in the calibration blank. The laboratory performed the method-required reanalysis of the sample at a two-fold dilution with the rhodium and indium % relative intensities meeting the method QC acceptance criteria. The validator reports the sample results from the two-fold dilution without qualification and with the raised quantitation limits for non-detects on the Data Summary Table and discusses the reason for reporting sample results from the diluted sample analysis in the Data Validation Memorandum.

VIII. MATRIX SPIKES**A. OBJECTIVE**

Data for matrix spikes are generated to determine method bias for specific sample matrices at the time of sample preparation and analysis. Matrix spike data can be used to determine long-term inter-laboratory bias of an analytical method for various matrices and are used in setting quality control acceptance criteria for spiking compounds.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications, or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. In accordance with the SAP, QAPP and/or method, a field sample of each matrix is spiked with known concentrations of specific target analytes to generate a matrix spike sample. Concurrently, the laboratory analyzes an unspiked aliquot and the matrix spike of the field sample.
2.
 - a. Field samples (not equipment or bottle blanks and not PE samples) must be spiked to assess matrix effects.
 - b. Field samples chosen for matrix spike analysis should not contain high levels of matrix spiking analytes prior to spiking. Preferably, field samples chosen for matrix spike analysis should contain low levels of the spiking analytes.
3. Spike recoveries must be within the QC acceptance criteria specified in the method, SAP, or QAPP.
4. If a post-digestion spike analysis is required by the method, SAP, or QAPP for analytes whose matrix spike recoveries are not within method QC acceptance criteria, then an unspiked aliquot of the prepared field sample chosen for matrix spike analysis is spiked with known concentrations of specific target analytes. The concentration and acceptance criteria for the post-digestion spike analysis should be stated in the method, SAP, or QAPP.

C. EVALUATION/D. ACTION

C. EVALUATION	D. ACTION
<p>1. Verify that the correct analytes were added to the sample at the required concentrations, that matrix spike samples were analyzed at the proper frequency, and that matrix spike results are provided for each sample matrix. If more than one analytical method was used to report sample results for an analyte, then verify that matrix spike results for that analyte are provided by each method.</p>	<p>All potential impacts on the sample data resulting from matrix spike sample analysis anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.</p> <p>1. If the laboratory did not use the required analytes at the concentration and frequency specified in the method for each sample matrix and method, then the validator must use professional judgment to determine whether the associated sample data should be qualified.</p>
<p>2. a. Verify that a field sample was chosen for the matrix spike.</p> <p>b. Determine if an inappropriate sample containing high levels of the spiking analytes was chosen for the matrix spike.</p> <p>c. Ascertain if the matrix spike sample required dilutions.</p>	<p>2. a. If an equipment blank, a bottle blank, or a PE sample was used for the matrix spike sample, then the validator should note this information in the Data Validation Memorandum and discuss the impact on assessing method bias, sample matrix effects and, ultimately, data usability.</p> <p>b. If the matrix spike analytes were present in the field sample at high concentrations (e.g., 4x the spike concentration) before spiking, then the validator must use professional judgment in assessing matrix spike recoveries. Generally, spike recovery limits do not apply when the sample concentration exceeds the spike concentration by a factor of 4 or more.</p> <p>c. If no matrix spike data can be reported because of sample dilution, then the validator should note this problem in the Data Validation Memorandum and discuss the impact on assessing data usability in the case where method bias information is absent.</p>

C. EVALUATION	D. ACTION
<p>3. Verify that all spike recoveries are within the QC acceptance criteria specified in the method.</p>	<p>Note: Action applies to the affected analyte in <u>all</u> samples of the same matrix prepared and analyzed by the same method.</p> <p>3. a. If any spike recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should:</p> <ul style="list-style-type: none"> i. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. Accept non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. <p>b. If any recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 30%, then the validator should:</p> <ul style="list-style-type: none"> i. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. Estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. <p>c. If any recovery is less than 30%, then the validator should:</p> <ul style="list-style-type: none"> i. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. ii. Reject (R) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method.

C. EVALUATION	D. ACTION
3. Continued from above.	3. d. If the majority of spike analyte recoveries for a method are outside the method QC acceptance criteria, then the validator may use professional judgment to estimate or reject <u>all</u> positive detects and non-detects in all samples of the same matrix prepared and analyzed by the same method.
4. Verify that a post-digestion spike sample was analyzed at the proper frequency, that the correct analytes were added at the required concentrations, and that post-digestion spike results are provided in accordance with method requirements if matrix spike method QC acceptance criteria are not met.	4. Generally, no action is taken based solely on the post-digestion spike result. However, in some cases, post-digestion spike data may aid in evaluating matrix interferences. The data validator should use professional judgment to qualify sample data based on post-digestion spike results. A discussion of any possible impacts on the data should be included in the Data Validation Memorandum.
*5. Check and recalculate the analytical concentrations and percent recovery for at least one spiked analyte per method. Verify that the recalculated value agrees within $\pm 10\%$ of the reported value.	5. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should contact the laboratory to requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
6. Evaluate the appropriateness of not qualifying the entire data set based on method/matrix bias results.	6. Generally, action based on the matrix spike results is applied to the affected analyte in <u>all</u> samples of the same matrix prepared and analyzed by the same method in a sample delivery group. However, professional judgment may be used to apply the action only to a specific subset of samples of the same matrix or to the matrix spike sample if there is information to support such an action. All justifications for not qualifying data should be documented in the Data Validation Memorandum and the potential impact on data usability in meeting the project DQOs should be discussed.

* Note: The following subsection is applicable only to a Tier III data validation:

C.5

Table INORG-VIII-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON MATRIX SPIKE RECOVERIES*

Sample Results	% Recovery			
	%R < 30%	30% ≤ %R < LL	LL ≤ %R ≤ UL	%R > UL
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL = Lower Limit of method QC acceptance criteria

UL = Upper Limit of method QC acceptance criteria

* Qualification is applied to the affected analyte in all samples of the same matrix analyzed by the same method; however, the validator may use professional judgment to apply actions to all positive detects and non-detects if the majority of spike analyte recoveries are outside method QC acceptance criteria.

E. EXAMPLES

Example #1: (High matrix spike recovery for one analyte)

Aqueous QC sample MADG56MS, analyzed by ICP-AES under CLP SOW ILM05.4, has a high matrix spike recovery result for zinc.

Sample No.	Analyte	MS % Rec.	MS % Rec. Criteria	Post-digestion Spike % Rec.
MADG56	Zinc	137	75-125	132

The validator concludes that the sample matrix causes a positive bias for zinc in all aqueous samples associated with this sample delivery group. The validator estimates (J) positive detects for zinc in all aqueous samples on the Data Summary Table. The validator discusses the high matrix spike recovery in the Data Validation Memorandum and notes that the post-digestion spike recovery for zinc was also high, confirming a matrix interference, and that recoveries for the other matrix spike analytes were acceptable.

Example #2: (Extremely low matrix spike recovery for one analyte)

Soil QC sample MAFG77MS, analyzed by ICP-AES under CLP SOW ILM05.4, has an extremely low antimony matrix spike recovery result.

Sample No.	Analyte	MS % Rec.	MS % Rec. Criteria	Post-digestion Spike % Rec.
MAFG77	Antimony	18	75-125	43

The validator concludes that the sample matrix causes a negative bias for antimony in all soil samples associated with this sample delivery group. The validator estimates (J) positive detects and rejects (R) non-detects for antimony in all soil samples on the Data Summary Table. The validator discusses the low matrix spike recovery in the Data Validation Memorandum and notes that the post-digestion spike recovery for antimony was also low, confirming a matrix interference, and that recoveries for the other matrix spike analytes were acceptable.

E. EXAMPLES (Continued)

Example #3: (One analyte analyzed by two methods; Low matrix spike recovery for one method, acceptable matrix spike recovery for the other method)

Aqueous samples in an SDG were analyzed for lead under CLP SOW ILM05.4. Some lead sample results were reported from the ICP-AES analysis and other lead sample results were reported from the ICP-MS analysis. Aqueous QC sample MAAG23MS has a low matrix spike recovery for lead analyzed by ICP-MS and an acceptable recovery for lead analyzed by ICP-AES.

Sample No.	Analyte	Method	MS % Rec.	MS % Rec. Criteria	Post-digestion Spike % Rec.
MAAG23	Lead	ICP-MS	53	75-125	80
		ICP-AES	87		NA

NA = Not Applicable

The validator concludes that the sample matrix causes a negative bias for lead in all aqueous samples analyzed by ICP-MS in this sample delivery group. The validator estimates (J) positive detects and estimates (UJ) non-detects for lead in all aqueous samples reported from the ICP-MS analysis. The validator accepts all lead results in aqueous samples analyzed by ICP-AES. The validator discusses the low matrix spike recovery and the acceptable post-digestion spike recovery for lead analyzed by ICP-MS in the Data Validation Memorandum and notes that recoveries for lead in aqueous samples analyzed by ICP-AES and the other matrix spike analytes were acceptable.

IX. LABORATORY DUPLICATE SAMPLES**A. OBJECTIVE**

Data for laboratory duplicate sample analyses are generated to determine laboratory precision for specific sample matrices at the time of sample preparation and analysis. Duplicate sample analysis data can be used to determine long-term interlaboratory precision of an analytical method for various matrices.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Inorganic data. The CLP-Inorganic method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the Inorganic analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications, or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA-approved QAPP/SAP or amendment to the QAPP/SAP.

1. In accordance with the SAP, QAPP, and/or method, a field sample is prepared and analyzed in duplicate for each matrix.
2. Field samples (not equipment or bottle blanks and not PE samples) must be used to assess laboratory precision.
3. Relative Percent Differences (RPDs) or absolute differences between the laboratory duplicate samples must be within the QC acceptance criteria specified in the method for the specific matrix.

C. EVALUATION/D. ACTION

C. EVALUATION	D. ACTION
<p>1. Verify that laboratory duplicate samples were prepared and analyzed at the proper frequency and that duplicate sample results are provided for each sample matrix. If more than one analytical method was used to report sample results for an analyte, then verify that laboratory duplicate sample results are provided for that analyte by each method.</p>	<p>All potential impacts on the sample data resulting from laboratory duplicate sample anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.</p> <p>1. If the laboratory did not prepare and analyze laboratory duplicates at the frequency specified in the method for each sample matrix and method, then the validator must use professional judgment to determine whether the associated sample data should be qualified.</p>
<p>2. Verify that a field sample was chosen for the laboratory duplicate sample.</p>	<p>2. If an equipment blank, a bottle blank, or a PE sample was used for the duplicate sample analysis, then the validator should note this information in the Data Validation Memorandum and discuss the impact on assessing laboratory precision, sample matrix effects and, ultimately, data usability.</p>
<p>3. Verify that all Relative Percent Differences or absolute differences between the sample and the laboratory duplicate are within the QC acceptance criteria specified in the method for the specific matrix.</p>	<p>Note: Action applies to the affected analyte in <u>all</u> samples of the same matrix prepared and analyzed by the same method.</p> <p>3. If any RPD or absolute difference for a laboratory duplicate sample is outside the method QC acceptance criteria, then the validator should:</p> <ol style="list-style-type: none"> a. Estimate (J) positive detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. b. Estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method.

C. EVALUATION	D. ACTION
<p>3. Continued from above.</p> <p>Note: The CLP SOW ILM05.4 laboratory duplicate sample method QC acceptance criteria do not differentiate between aqueous and soil matrices. Because laboratory variability arising from the sub-sampling of non-homogenous soil samples is common, the data validation criteria in Table INORG-IX-3 should be used for non-aqueous matrices. If data quality objectives allow for other criteria to be used, then the validation criteria should be documented in the EPA-approved site-specific QAPP or amendment to the QAPP.</p>	<p>3. c. If any analyte is detected at concentrations less than the sample quantitation limit or are non-detects in both aqueous laboratory duplicate samples, then no action is taken.</p> <p>d. If the majority of the laboratory duplicate sample results are outside method QC acceptance criteria, then the validator may use professional judgment to estimate (J) all positive detects and estimate (UJ) all non-detects in all samples of the same matrix.</p>
<p>*4. Check and recalculate the analytical concentrations and RPD for at least one duplicate sample per analytical method. Verify that the recalculated value agrees within $\pm 10\%$ of the reported value.</p>	<p>4. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p>

C. EVALUATION	D. ACTION
<p>5. Evaluate the appropriateness of qualifying only the laboratory duplicate sample results or only a subset of samples of the same matrix for the affected analyte. Field duplicate sample data should be evaluated to identify overall precision issues.</p>	<p>5. Generally, action based on the laboratory duplicate sample results is applied to the affected analyte in <u>all</u> samples of the same matrix prepared and analyzed by the same method in a sample delivery group (SDG). If there is information to indicate that the poor laboratory precision is limited to the duplicate sample or to a select group of samples in the SDG, then professional judgment may be used to apply the action only to the field sample used for the laboratory duplicate sample analysis or to a select group of samples in the SDG. All justifications for not qualifying the entire data set should be documented in the Data Validation Memorandum, and the potential impact on data usability in meeting the project DQOs should be discussed.</p>
<p>6. Evaluate laboratory duplicate precision data to confirm the laboratory's ability to generate precise data and field duplicate precision data to assess overall precision.</p>	<p>6. If precision data for the laboratory duplicate sample and field duplicate pair indicate a heterogeneous matrix at the site or potential sampling error, then the validator may use professional judgment to qualify <u>all</u> field sample results. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting the project DQOs should be discussed. Refer to Section INORG-X (Field Duplicates) for additional guidance.</p>

* **Note:** The following subsection is applicable only to a Tier III data validation:

C.4

Table INORG-IX-1:

QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE SAMPLES*

Sample Results	Laboratory Duplicate Sample Results	
	RPD or Abs. Diff. \leq QC Limit	RPD or Abs. Diff. $>$ QC Limit
Detects	A	J
Non-detects	A	UJ

* If QC acceptance criteria for the specific matrix are specified in the method, then use the method QC criteria in this table. If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in Table INORG-IX-2 or INORG-IX-3.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the method QC acceptance criteria.

Table INORG-IX-2:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE
SAMPLES - AQUEOUS MATRICES***

Sample Results	Aqueous Laboratory Duplicate Sample Results			
	Both Sample and Duplicate $\geq 5xQL$		One or Both Sample and Duplicate $< 5xQL$	
	RPD $\leq 20\%$	RPD $> 20\%$	Abs. Diff. $\leq QL$	Abs. Diff. $> QL$
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

* If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in this table (from Appendix I). If QC acceptance criteria for the specific matrix are specified in the method, then use the criteria in Table INORG-IX-1 above.

QL = Sample Quantitation Limit

- When applying the absolute difference criteria, the sample quantitation limit for the sample (vs. the duplicate sample) is used.
- No action is applied when both sample and duplicate values are detected at $< QL$ or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the QC acceptance criteria.

Table INORG-IX-3:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON LABORATORY DUPLICATE
SAMPLES - NON-AQUEOUS MATRICES***

Sample Results	Non-Aqueous Laboratory Duplicate Sample Results			
	Both Sample and Duplicate $\geq 5xQL$		One or Both Sample and Duplicate $< 5xQL$	
	RPD $\leq 35\%$	RPD $> 35\%$	Abs. Diff. $\leq 2xQL$	Abs. Diff. $> 2xQL$
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

* If QC acceptance criteria for the specific matrix are not specified in the method, then use the criteria in this table. If QC acceptance criteria for the specific matrix are specified in the method, then use the criteria in Table INORG-IX-1 above.

QL = Sample Quantitation Limit

- When applying the absolute difference criteria, the sample quantitation limit for the sample (vs. the duplicate sample) is used.
- No action is applied when both sample and duplicate values are detected at $< QL$ or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix, prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the laboratory duplicate results are outside the QC acceptance criteria.

E. EXAMPLES

Example #1: (Both aqueous sample and laboratory duplicate sample concentrations $\geq 5xSQL$; RPD $> 20\%$; Poor laboratory precision by ICP-AES; Acceptable laboratory precision by ICP-MS)

Aqueous sample MACH79 and laboratory duplicate sample MACH79D, digested and analyzed by ICP-AES under CLP SOW ILM05.4, have a high RPD for lead. Sample MACH79 has a lead concentration of 60 ug/L and laboratory duplicate sample MACH79D has a lead concentration of 81 ug/L. Both results are greater than 5xSQL with an RPD of 30%.

Analyte (method)	MACH79		MACH79D		RPD	RPD Criteria
	Sample Conc. (ug/L)	SQL/5xSQL (ug/L)	Sample Conc. (ug/L)	SQL/5xSQL (ug/L)		
Lead (ICP-AES)	60	10/ 50	81	10/ 50	30%	20%

The SDG also contained some samples which were analyzed for lead by ICP-MS. ICP-MS aqueous sample MACJ07 has a lead concentration of 9.3 ug/L and laboratory duplicate sample MACJ07D has a lead concentration of 10.5 ug/L. Both results are greater than 5xSQL with an RPD of 12%.

Analyte (method)	MACJ07		MACJ07D		RPD	RPD Criteria
	Sample Conc. (ug/L)	SQL/5xSQL (ug/L)	Sample Conc. (ug/L)	SQL/5xSQL (ug/L)		
Lead (ICP-MS)	9.3	1/ 5	10.5	1/ 5	12%	20%

Laboratory precision for lead is acceptable for the ICP-MS analysis but did not meet method QC criteria by ICP-AES analysis. The validator evaluates the field duplicate pair analyzed by ICP-AES and determines that the RPDs and absolute differences are within the field duplicate QC criteria indicating acceptable overall precision for this sampling event. The validator then concludes that the poor laboratory precision for lead in this sample is specific to the ICP-AES analysis. The validator estimates (J) positive detects and estimates (UJ) non-detects for lead in all aqueous samples reported from the ICP-AES analysis on the Data Summary Table. The validator discusses the poor laboratory precision for lead analyzed by ICP-AES in the Data Validation Memorandum and notes that laboratory precision for the other analytes as well as for lead analyzed by ICP-MS was acceptable.

E. EXAMPLES (continued)

Example #2: (Both soil sample and laboratory duplicate sample concentrations < 5xSQL; Absolute difference > 2xSQL; Poor laboratory precision for manganese; Acceptable laboratory precision for thallium)

Soil sample MAAZ33 (1.02 g wet wt.) and laboratory duplicate MAAZ33D (1.01 g wet wt.), digested and analyzed under CLP SOW ILM05.4 by ICP-AES, have a percent solids of 92%. Sample MAAZ33 has a manganese concentration of 3.0 mg/kg (dry wt.) and a thallium concentration of 2.1J mg/kg (dry wt.). The duplicate sample MAAZ33D has a manganese concentration of 7.9 mg/kg (dry wt.) and a non-detect for thallium. The validator notes that all results are less than 5 times the sample quantitation limits (QLs).

Analyte	MAAZ33		MAAZ33D		Abs. Diff. (mg/kg)	Abs. Diff. Criteria (2xSQL) (mg/kg)
	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)		
Manganese	3.0	1.6/ 8.0	7.9	1.6/ 8.0	4.9	3.2
Thallium	2.1J	2.7/ 13.5	2.7U	2.7/ 13.5	NA	NA

The validator uses the laboratory duplicate QC criteria specified in Table INORG-IX-3 for non-aqueous matrices. Since the sample values are less than 5xSQL, the validator uses the 2xSQL criteria to evaluate the absolute difference between the sample duplicate concentrations rather than the RPD criteria. The duplicate precision for thallium by ICP-AES is not evaluated since both results are below the SQL or non-detects. For manganese the absolute difference between the duplicate samples is greater than the 2xSQL criteria. The validator evaluates the field duplicate pair and determines that the RPDs and absolute differences are within the QC criteria specified in the QAPP, indicating acceptable overall precision for this sampling event. The validator then concludes that the lack of laboratory precision in this sample is due to poor laboratory technique. The validator estimates (J) positive detects and estimates (UJ) non-detects for manganese in all soil samples on the Data Summary Table. The validator discusses the poor laboratory precision for manganese in the Data Validation Memorandum and notes that laboratory precision for the other analytes was acceptable.

E. EXAMPLES (continued)

Example #3: (One soil sample value $\geq 5xSQL$ and laboratory duplicate sample value $< 5xSQL$;
Absolute difference $> 2xSQL$; Poor laboratory precision)

Soil sample MACC11 (1.02 g wet wt.) and laboratory duplicate sample MACC11D (1.04 g wet wt.), digested and analyzed under CLP SOW ILM05.4, have a percent solids of 88%. Sample MACC11 has a potassium concentration of 3900 mg/kg (dry wt.). The duplicate sample MACC11D has a potassium concentration of 2100 mg/kg (dry wt.). The validator notes that one result is less than $5xSQL$ and one result is greater than $5xSQL$.

Analyte	MACC11		MACC11D		Abs. Diff. (mg/kg)	Abs. Diff. Criteria ($2xSQL$) (mg/kg)
	Sample Conc. (mg/kg)	SQL/ $5xSQL$ (mg/kg)	Sample Conc. (mg/kg)	SQL/ $5xSQL$ (mg/kg)		
Potassium	3900	557/ 2790	2100	546/ 2730	1800	1114

The validator uses the laboratory duplicate QC criteria specified in Table INORG-IX-3 for non-aqueous matrices. Since one sample value is less than $5xSQL$ and the other sample value is greater than $5xSQL$, the QC criteria of $2xSQL$ rather than the RPD must be used to evaluate the differences between the sample concentrations. The absolute difference between the duplicate samples is 1800 mg/kg which is greater than $2xSQL$. The validator evaluates the field duplicate pair and determines that the RPDs and absolute differences are within the QC criteria specified in the QAPP, indicating overall precision for this sampling event was acceptable. The validator then concludes that the poor laboratory precision in this sample is due to poor laboratory technique. The validator estimates (J) positive detects and estimates (UJ) non-detects for potassium in all soil samples on the Data Summary Table. The validator discusses the poor laboratory precision for potassium in the Data Validation Memorandum and notes that laboratory precision for the other analytes was acceptable.

X. FIELD DUPLICATES**A. OBJECTIVE**

Field duplicates measure the cumulative effects of both field and laboratory precision and hence provide an indication of overall precision. Therefore, field duplicates may have greater variability than laboratory duplicates which measure only laboratory precision. It is also expected that non-aqueous matrices will have a greater variance than aqueous matrices due to the heterogeneity of most non-aqueous samples (such as soil/sediment samples).

B. CRITERIA

1. The frequency of field duplicate analysis must support the site-specific Data Quality Objectives (DQOs) and must be documented in the EPA-approved QAPP or SAP.
2. a. Aqueous Field Duplicates
 - i. For all analytes detected at concentrations greater than or equal to five times the sample quantitation limit in both field duplicate samples of aqueous matrices, the RPD must be less than or equal to 30 percent.
 - ii. For all analytes detected at concentrations less than five times the sample quantitation limit in either field duplicate sample of aqueous matrices, the absolute difference must be less than or equal to twice the sample quantitation limit.
- b. Non-Aqueous Field Duplicates
 - i. For all analytes detected at concentrations greater than or equal to five times the sample quantitation limit in both field duplicate samples of non-aqueous matrices, the RPD must be less than or equal to 50 percent.
 - ii. For all analytes detected at concentrations less than five times the sample quantitation limit in either field duplicate sample of non-aqueous matrices, the absolute difference must be less than or equal to four times the sample quantitation limit.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
<p>1. a. Identify the samples which are field duplicates from the Chain-of-Custody Record and/or the Traffic Report.</p> <p>b. Verify that the appropriate number of field duplicates per matrix sampled were collected and analyzed to support project DQOs.</p>	<p>All potential impacts on the sample data resulting from field duplicate anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.</p> <p>1. a. If field duplicates are not listed on the Chain-of-Custody Record or the Traffic Report, then the validator should contact the sampler to ascertain if field duplicates were collected. If the forms were completed incorrectly or if field duplicates were not collected, then the validator should document this on the Data Validation Worksheet and in the Data Validation Memorandum.</p> <p>b. If field duplicates were not collected at the required frequency to support project DQOs, then the validator should note the absence of field precision data in the Data Validation Memorandum and discuss how the lack of field precision data might potentially increase uncertainty surrounding site decisions.</p>

C. EVALUATION	D. ACTION
<p>2. a. Aqueous Field Duplicates</p> <p>i. Calculate the RPD for all analytes detected at concentrations greater than or equal to 5x the sample quantitation limit in both aqueous field duplicates.</p> <p>ii. Calculate the absolute difference for all analytes detected at concentrations less than 5x the sample quantitation limit in either one or both of the aqueous field duplicate samples (including the case where one duplicate sample result is a non-detect and the other result is a positive detect).</p>	<p>Note: Action applies to the affected analyte in <u>all</u> samples of the same matrix prepared and analyzed by the same method.</p> <p>2. a. Aqueous Field Duplicates</p> <p>i. If any analyte is detected at concentrations greater than or equal to 5x the sample quantitation limit in both aqueous field duplicate samples and has an RPD greater than 30%, then the validator should:</p> <ul style="list-style-type: none"> - Estimate (J) positive detects and estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. <p>ii. If any analyte is detected at concentrations less than 5x the sample quantitation limit in either one or both of the aqueous field duplicate samples and the absolute difference is greater than 2x the sample quantitation limit, then the validator should:</p> <ul style="list-style-type: none"> - Estimate (J) positive detects and estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. - If any analyte is detected at concentrations less than the sample quantitation limit in both of the field duplicate samples, or if any analyte is a non-detect in both of the field duplicate samples, then no action is taken.

C. EVALUATION	D. ACTION
<p>2. b. Non-Aqueous Field Duplicates</p> <p>i. Calculate the RPD for all analytes detected at concentrations greater than or equal to 5x the sample quantitation limit in both non-aqueous field duplicates.</p> <p>ii. Calculate the absolute difference for all analytes detected at concentrations less than 5x the sample quantitation limit in either one or both of the non-aqueous field duplicate samples (including the case where one duplicate sample result is a non-detect and the other result is a positive detect).</p> <p>Note: When applying the criteria of 4x the sample quantitation limit, the sample quantitation limit is calculated using the sample weight, volume, and percent solids for the sample versus the duplicate sample.</p>	<p>2. b. Non-Aqueous Field Duplicates</p> <p>i. If any analyte is detected at concentrations greater than or equal to 5x the sample quantitation limit in both non-aqueous field duplicate samples and has an RPD greater than 50%, then the validator should:</p> <ul style="list-style-type: none"> - Estimate (J) positive detects and estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. <p>ii. If any analyte is detected at concentrations less than 5x the sample quantitation limit in either one or both of the non-aqueous field duplicate samples and the absolute difference is greater than 4x the sample quantitation limit, then the validator should:</p> <ul style="list-style-type: none"> - Estimate (J) positive detects and estimate (UJ) non-detects for the affected analyte in all samples of the same matrix prepared and analyzed by the same method. - If any analyte is detected at concentrations less than the sample quantitation limit in both of the field duplicate samples, or if any analyte is a non-detect in both of the field duplicate samples, then no action is taken.
<p>*3. Check and recalculate the analytical concentrations for at least one positive detect and one sample quantitation limit (for a diluted sample or soil sample) for each analytical method in each field duplicate sample.</p>	<p>3. If calculation and/or transcription errors are detected, then the validator should follow the procedures outlined in Section INORG-XIV (Analyte Quantitation and Reported Quantitation Limits), D.1-D.3.</p>

C. EVALUATION	D. ACTION
<p>4. a. Evaluate the appropriateness of qualifying only the field duplicate sample results or only a subset of samples of the same matrix for the affected analyte.</p> <p>b. Evaluate field duplicate precision data to assess overall precision and to verify the field sampler's ability to collect representative duplicate samples. Laboratory duplicate sample data should be evaluated to verify the laboratory's ability to generate precise data. Matrix spike data can also be evaluated to identify overall matrix issues.</p>	<p>4. a. Generally, action based on field duplicate results is applied to the affected analyte across <u>all</u> samples of the same matrix prepared and analyzed by the same method. If there is information to indicate that the matrix heterogeneity and/or potential sampling error are limited to the field duplicate samples or to a specific subset of samples of the same matrix, then professional judgment may be used to apply the action only to the field duplicate samples or to a specific subset of samples of the same matrix. The validator should discuss in the Data Validation Memorandum the justification for not qualifying all samples of the same matrix and limiting the qualification to specific samples. The potential impact on the representativeness and usability of the data in meeting project DQQs should be discussed.</p> <p>b. If field duplicate data indicate poor field precision and general sample heterogeneity and/or possible sampling error, then professional judgment may be used to qualify data for all analytes in all samples of the same matrix. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting project DQOs should be discussed. Refer to Section IX (Laboratory Duplicate Samples) for additional guidance.</p>

* **Note:** The following subsection is applicable only to a Tier III data validation:

C.3

Table INORG-X-1:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON FIELD DUPLICATES -
AQUEOUS MATRICES**

Sample Results	Aqueous Field Duplicate Sample Results			
	Both Duplicates $\geq 5xQL$		One or Both Duplicates $< 5xQL^*$	
	RPD $\leq 30\%$	RPD $> 30\%$	Abs. Diff. $\leq 2xQL$	Abs. Diff. $> 2xQL$
Detects	A	J	A	J
Non-detects	A	UJ	A	J

QL = Sample Quantitation Limit

* No action is taken when both field duplicate results are positive detects $< QL$ or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the field duplicate results are outside QC acceptance criteria.

Table INORG-X-2:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON FIELD DUPLICATES -
NON-AQUEOUS MATRICES**

Sample Results	Non-Aqueous Field Duplicate Sample Results			
	Both Duplicates $\geq 5xQL$		One or Both Duplicates $< 5xQL^*$	
	RPD $\leq 50\%$	RPD $> 50\%$	Abs. Diff. $\leq 4xQL$	Abs. Diff. $> 4xQL$
Detects	A	J	A	J
Non-detects	A	UJ	A	UJ

QL = Sample Quantitation Limit

* No action is taken when both field duplicate results are positive detects $< QL$ or are non-detects.

Note: Qualification refers to the affected analyte in all samples of the same matrix prepared and analyzed by the same method. Professional judgment may be used to qualify all positive detects and non-detects if the majority of the field duplicate results are outside QC acceptance criteria.

E. EXAMPLES

Example #1: (Both soil field duplicate sample concentrations $\geq 5 \times \text{SQL}$; RPD > 50%; Acceptable laboratory precision)

Soil samples MADF61 and MADF62 are field duplicates analyzed under CLP SOW ILM05.4. Sample MADF61 has a detected manganese concentration of 120 mg/kg. Sample MADF62 has a detected manganese concentration of 275 mg/kg. The validator notes that both results are greater than 5x the sample quantitation limit (SQL). (The samples contain 82% and 91% solids, respectively, and 1.0 g wet weight was used for each.) Therefore, the validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 78%.

Analyte	Sample MADF61		Duplicate MADF62		RPD	RPD Criteria
	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)		
Manganese	120	1.8/ 9.0	275	1.6/ 8.0	78%	50%

The validator reviews the laboratory duplicate sample data and determines that laboratory precision was acceptable. The validator estimates (J) positive detects and estimates (UJ) non-detects for manganese in all soil samples on the Data Summary Table based on poor field precision. The validator notes the qualification and justification in the Data Validation Memorandum and also notes that poor field precision may be due to a heterogeneous matrix or may be a result of sampling error.

E. EXAMPLES (continued)

Example #2: (Both aqueous field duplicate sample concentrations < 5xSQL; Absolute difference > 2xSQL; Acceptable laboratory precision)

Aqueous samples MAEL21 and MAEL22 are field duplicates analyzed under CLP SOW ILM05.4. Sample MAEL21 has a detected cobalt concentration of 59 ug/L, and sample MAEL22 has a detected cobalt concentration of 187 ug/L. The validator notes that both field duplicate results are below 5x the sample quantitation limit. Since both field duplicate sample values are less than 5xSQL, the validation criteria of 2xSQL is used to evaluate the difference between the sample concentrations. The validator determines that the absolute difference between the field duplicates is 128 ug/L, which is greater than 2xSQL.

Analyte	Sample MAEL21		Duplicate MAEL22		Abs. Diff. (ug/L)	Abs. Diff. Criteria (2xSQL) (ug/L)
	Sample Conc. (ug/L)	SQL/ 5xSQL (ug/L)	Sample Conc. (ug/L)	SQL/ 5xSQL (ug/L)		
Cobalt	59	50/ 250	187	50/ 250	128	100

The validator reviews the laboratory duplicate sample data and determines that laboratory precision was acceptable. The validator estimates (J) positive detects and estimates (UJ) non-detects for cobalt in all aqueous samples on the Data Summary Table based on poor field precision. The validator notes the qualification and justification in the Data Validation Memorandum and also notes that poor field precision may be due to sampling error.

E. EXAMPLES (continued)

Example #3: (One soil field duplicate sample concentration < 5xSQL, other soil field duplicate sample concentration \geq 5xSQL; Absolute difference > 4xSQL; Poor field and laboratory precision.)

Soil samples MADL22 and MADL23 are field duplicates analyzed under CLP SOW ILM05.4. Sample MADL22 has a detected zinc concentration of 18 mg/kg; sample MADL23 has a detected zinc concentration of 54 mg/kg. The validator notes that one field duplicate result is less than 5xSQL and the other result is greater than 5xSQL. Therefore, the validation criteria of 4xSQL rather than the RPD is used to evaluate the difference between the sample concentrations. The validator calculates the 4xSQL criteria using the sample quantitation limit of the sample. (The samples contain 89% and 95% solids, respectively, and 1.0 g wet weight was used for each.) The absolute difference between the field duplicates is 36 mg/kg which is greater than 4xSQL.

Analyte	Sample MADL22		Duplicate MADL23		Abs. Diff. (mg/kg)	Abs. Diff. Criteria (4xSQL) (mg/kg)
	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)	Sample Conc. (mg/kg)	SQL/ 5xSQL (mg/kg)		
Zinc	18	6.7/ 34	54	6.3/ 32	36	27

The validator reviews the laboratory duplicate sample data and determines that laboratory precision was unacceptable for zinc. The validator is unable to determine the source of the imprecision since both laboratory and field precision were poor. The validator estimates (J) positive detects and estimates (UJ) non-detects for zinc in all soil samples on the Data Summary Table. The validator notes the qualifications and justifications in the Data Validation Memorandum and also notes that the source of the imprecision cannot be determined.