

## PART IV

# Inorganic Data Validation Functional Guidelines

November 2008

## INORGANIC DATA VALIDATION FUNCTIONAL GUIDELINES - PART IV

The requirements to be checked in validation are listed below. "CCS" indicates that some of the contractual requirements for these items will also be checked by Contract Compliance Screening (CCS). CCS requirements are not always the same as data validation criteria.

I.	Preservation and Technical Holding Times.....	(CCS).....	INORG-I-1
II.	ICP-MS Tune.....	(CCS).....	INORG-II-1
III.	Calibrations.....	(CCS).....	INORG-III-1
IV.	Blanks.....	(CCS).....	INORG-IV-1
V.	ICP-AES Interference Check Sample (ICS).....	(CCS).....	INORG-V-1
VI.	ICP-MS Interference Check Sample (ICS).....	(CCS).....	INORG-VI-1
VII.	ICP-MS Internal Standards.....	(CCS).....	INORG-VII-1
VIII.	Matrix Spikes.....	(CCS).....	INORG-VIII-1
IX.	Laboratory Duplicate Samples.....	(CCS).....	INORG-IX-1
X.	Field Duplicates.....		INORG-X-1
XI.	ICP Serial Dilutions.....	(CCS).....	INORG-XI-1
XII.	Sensitivity Check.....	(CCS).....	INORG-XII-1
XIII.	Performance Evaluation Samples/Accuracy Check.....	(CCS).....	INORG-XIII-1
XIV.	Analyte Quantitation and Reported Quantitation Limits.....		INORG-XIV-1
XV.	System Performance.....		INORG-XV-1
XVI.	Overall Evaluation of Data.....		INORG-XVI-1

### Appendices

Appendix I	CLP SOW ILM05.4/Inorganic Analysis
Appendix J	Inorganic Functional Guidelines Action Tables
Appendix K	Inorganic Data Validation Worksheets

## I. PRESERVATION AND TECHNICAL HOLDING TIMES

## A. OBJECTIVE

The objective is to ascertain the validity of the analytical results based on the preservation techniques which were used and the holding time of the sample from time of collection to time of sample preparation and sample analysis.

## 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. REGION I PRESERVATION CRITERIA

SAMPLE TYPE	PRESERVATION CODE
Metals Aqueous <sup>a</sup>	2
Cyanide Aqueous <sup>a</sup>	1, 3
Metals (no Hg) Soil/Sediment/Sludge/Oily Waste/Wipe/Ash <sup>b</sup>	4
Mercury Soil/Sediment/Sludge/Oily Waste/Ash <sup>b</sup>	1
Cyanide Soil/Sediment/Sludge/Oily Waste <sup>b</sup>	1
Metals Biological Tissue <sup>c</sup>	5
Metals (no Hg) Air Filters <sup>d</sup>	4

Preservation Code:

1. Cool ( $\leq 6^{\circ}\text{C}$ )
2. Preserve with  $\text{HNO}_3$  to pH less than 2
3. Preserve with  $\text{NaOH}$  to pH more than 12; add reducing agent in the presence of oxidants (e.g., chlorine); remove sulfides as required by the method
4. Room temperature
5. Freeze

## REFERENCES

- a. 40 CFR, Part 136, Appendix C
- b. SW-846: Chapter 3; 3000, 6000, 7000, 9000 Series
- c. Evaluation of Dredged Material for Discharge in Waters of the U.S. - Testing Manual, EPA 823-B-97-001, February 1997, and QA/QC Guidance for Sampling and Analysis of Sediments, Waters, and Tissue for Dredged Material Evaluations, Chemical Evaluation, EPA 823-B-95-001, April 1995
- d. Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, Compendium Method IO-3.1, EPA/625/R-96/010a, June 1999.

2. REGION I TECHNICAL HOLDING TIME CRITERIA

PARAMETER*	CRITERIA
Metals (except mercury)	Properly preserved samples must be analyzed within 6 months of sample collection.
Mercury <sup>a</sup>	Properly preserved samples must be analyzed within 28 days of sample collection.
Cyanide	Properly preserved samples must be analyzed within 14 days of sample collection.

\* See Section B.1 above for applicable sample types.

<sup>a</sup> Mercury by cold vapor AA.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
<p>1. Preservation</p> <p>a. Examine the sample records (EPA Traffic Reports and/or COC Forms), Sample Receipt forms (DC-1 Form), laboratory tracking/storage forms, sample preparation records, and the data package narrative to verify that samples were properly preserved and maintained by the sampler and/or the laboratory according to Region I preservation criteria. If adequate documentation on field sample preservation is not present in the data package, then the validator must contact the sampler and/or laboratory to obtain the missing information.</p>	<p>All potential impacts on the sample data resulting from preservation and/or holding time 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. Preservation</p> <p>a. If the sampler cannot be contacted or cannot produce adequate preservation documentation, then the validator should assume that the samples were not preserved and should document on the preservation and holding times worksheet the date that sampler contact was attempted and/or established. If the laboratory cannot provide adequate sample preservation information, then the validator should use professional judgment to accept, qualify, or reject the sample data.</p>

C. EVALUATION	D. ACTION
<p>1. a. Continued from above.</p> <p>i. Verify that aqueous metals samples were preserved with HNO<sub>3</sub> according to Region I preservation criteria.</p>	<p>1. a. Continued from above.</p> <p>If the samples were not preserved properly in the field and/or if the laboratory failed to properly preserve or maintain sample preservation, then the validator should take the following actions:</p> <p>i. If data package documentation does not list the pH of each aqueous metals sample, then the validator should contact the laboratory to obtain any omitted information.</p> <ul style="list-style-type: none"> <li>- If aqueous metals samples were not preserved with HNO<sub>3</sub> according to Region I preservation criteria, then the validator should estimate (J) positive detects and estimate (UJ) non-detects for the affected samples. Professional judgment should be used to reject (R) non-detects based on the pH of the sample and chemistry of the analytes of interest.</li> <li>- If the sample pH was adjusted with HNO<sub>3</sub> according to Region I preservation criteria upon laboratory receipt of samples for metals which either were not acid-preserved in the field or were received at an elevated pH (e.g., due to high sample alkalinity), then the validator may use professional judgment to accept the positive detects and non-detects for that sample. The validator should take into consideration the length of time that the acid resided in the sample prior to sample preparation and the analyte's stability. The acid should be in the sample at least 24 hours prior to sample preparation.</li> </ul>

C. EVALUATION	D. ACTION
<p>1. a. ii. Verify that aqueous cyanide samples were preserved with NaOH according to Region I preservation criteria.</p> <p>iii. Verify that aqueous cyanide samples were tested and treated, if needed, for oxidizing agents (e.g., chlorine) and sulfides, according to method requirements.</p>	<p>1. a. ii. If data package documentation does not list the pH of each aqueous cyanide sample, then the validator should contact the laboratory to obtain any omitted information. If aqueous cyanide samples were not preserved with NaOH according to Region preservation criteria, then the validator should estimate (J) positive detects and estimate (UJ) non-detects for the affected samples. Professional judgment should be used to reject (R) non-detects based on the sample pH and chemistry.</p> <p>iii. If field or data package documentation does not indicate that a check for the presence of oxidants or sulfides was performed, or that samples were not treated for these interferences, then the validator should contact the sampler and/or laboratory to obtain any omitted information. Professional judgment should be used if a check was not performed or if samples were not treated in the presence of oxidants or sulfides. In this case, the validator should document in the Data Validation Memorandum all justifications for qualifying or not qualifying data, taking into consideration all available information about the sample matrix constituents, including any historical information that may exist for the site.</p>

C. EVALUATION	D. ACTION
<p>1. a. iv. Verify that inorganic samples were refrigerated or frozen (as required) according to Region I preservation criteria.</p>	<p>1. a. iv. For all matrices, if the Region I temperature preservation criteria were not met, then the validator should use professional judgment to accept, qualify, or reject the positive detects and non-detects for the affected samples. The validator should document all justifications for qualifying or not qualifying sample data in the Data Validation Memorandum.</p> <p>Professional judgment should be used when the laboratory has reported transportation cooler temperatures that slightly exceed the upper limits of the preservation criteria (&gt; 6°C). In this case, the laboratory procedure for monitoring cooler temperature may be in question. In this event, all justifications for qualifying or not qualifying sample data should be documented in the Data Validation Memorandum.</p>
<p>2. Technical Holding Times</p> <p>a. Verify that inorganic samples were analyzed within the technical holding time criteria. Establish technical holding times by comparing sampling dates reported on the EPA Traffic Report and/or COC Forms with dates of analysis reported on tabulated result forms.</p> <ul style="list-style-type: none"> <li>• Verify that aqueous and soil/sediment metals (excluding mercury) samples were analyzed within 6 months of sample collection.</li> </ul>	<p>2. Technical Holding Times</p> <p>a. If aqueous and soil/sediment metals and cyanide samples were not analyzed within the technical analytical holding time criteria, then the validator should estimate (J) positive detects and estimate (UJ) non-detects. Professional judgment should be used to reject (R) non-detects based on the magnitude of the holding time exceedance and the stability of the analyte.</p>

C. EVALUATION	D. ACTION
<p>2. a. Continued from above.</p> <ul style="list-style-type: none"> <li>• Verify that aqueous and soil/sediment mercury samples were analyzed within 28 days of sample collection.</li> <li>• Verify that aqueous and soil/sediment cyanide samples were analyzed within 14 days of sample collection.</li> </ul> <p>Note: Due to limited information concerning holding times for non-aqueous matrices, the holding times for water matrices should be applied to non-aqueous matrices.</p> <p>* b. Check the raw data including digestion/distillation logs and instrument run logs to verify reported sample digestion/distillation and analysis dates.</p>	<p>2. a. Continued from above.</p> <p>For other matrices, the validator should estimate (J) positive detects and should use professional judgment to estimate (UJ) or reject (R) non-detects when technical holding time criteria are exceeded.</p> <p>For all matrices, if analytical technical holding time criteria were grossly exceeded, then the validator should use professional judgment to estimate (J) positive detects and estimate (UJ) or reject (R) non-detects, taking into consideration the analyte's stability and the effects of additional storage on the sample results. The validator should use professional judgment to determine the reliability of the data.</p> <p>b. If discrepancies between the raw data and reported data are found, then the validator should contact the laboratory to obtain 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.2.b

Table INORG-I-1:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON PRESERVATION**

Sample Results	Aq. Metals: HNO <sub>3</sub> to pH < 2? Aq. Cyanide <sup>1</sup> : NaOH to pH > 12?		Temperature Criteria Met?	
	Y	N	Y	N
<b>Detects</b>	A	J	A	Professional Judgment
<b>Non-detects</b>	A	UJ or R*	A	Professional Judgment

\* Professional judgment may be used to estimate (UJ) or reject (R) non-detects based on the analyte's stability and magnitude of exceedance.

<sup>1</sup> Estimate (J) positive detects and use professional judgment to estimate (UJ) or reject (R) non-detects when cyanide samples are not tested nor treated, if needed, for oxidants or sulfides.

Table INORG-I-2:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON TECHNICAL HOLDING TIMES**

Sample Results	Technical Holding Time (Aqueous/Soil/Sediment)	
	Metals: HT ≤ 6 Months	Metals: HT > 6 Months
	Mercury: HT ≤ 28 Days	Mercury: HT > 28 Days
	Cyanide: HT ≤ 14 Days	Cyanide: HT > 14 Days
<b>Detects</b>	A	J
<b>Non-detects</b>	A	UJ or R*

\* Professional judgment may be used to estimate or reject non-detects based on the analyte's stability, magnitude of exceedance, and the effects of additional storage on the sample results.

- For other matrices, estimate (J) positive detects and use professional judgment to estimate (UJ) or reject (R) non-detects when Region I technical holding time criteria are not met.

## E. EXAMPLES

Example #1: (Proper preservation; Analysis holding time exceeded)

Mercury soil sample MADD09 was sampled on 3/1/07 and was received at the laboratory on 3/2/07. Upon examination of the Traffic Report and the laboratory sample receipt and tracking information, the validator determines that the sample was shipped and stored at 4°C. As noted in the data package narrative, due to a laboratory tracking error, the laboratory analyzed the sample on 3/31/07, 30 days from the sampling date. The validator estimates (J) the positive detects and estimates (UJ) the non-detects for mercury in sample MADD09 on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

Example #2: (Improper preservation; Analysis holding time exceeded)

Aqueous mercury samples MAED54 and MAED55 were analyzed by CLP SOW ILM05.4. The validator examines the data package and determines that the laboratory did not report the pH. The validator contacts the laboratory to determine whether the pH was checked by the laboratory and notes that it was not checked. The validator then examines the Traffic Report contained in the data package and notes that the sampler failed to record what, if any, preservation techniques were utilized. The validator contacts the sampler who has no record of the samples being preserved with HNO<sub>3</sub>.

The sampling date for MAED54 and MAED55 was 6/1/07 and the analysis date was 6/30/07, 29 days from sampling. The aqueous mercury sample exceeded the technical holding time criteria by one day. The validator examines the Form I and notes that mercury is reported at 2 ug/L for MAED54 and non-detected for MAED55. The validator reports the mercury positive detect as estimated (J) and uses professional judgment to reject (R) the non-detect on the Data Summary Table since there is no record of samples being preserved with acid. The validator notes in the Data Validation Memorandum that the sample data are qualified based on improper preservation (without acid) and exceeded technical holding times.

Example #3: (Proper preservation; Analysis holding time grossly exceeded)

Cyanide soil samples were sampled on 8/1/07 and received at the laboratory on 8/2/07. Upon examination of the Traffic Report, laboratory receipt information, and sample tracking records, the validator determines that the samples were properly preserved at 4°C. All samples were not analyzed until 9/1/07, 31 days from the sampling date, due to a laboratory tracking error, and the analysis holding time was grossly exceeded. The validator estimates (J) positive detects and uses professional judgment to reject (R) non-detects for cyanide in all samples on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

## II. ICP-MS TUNE

## A. OBJECTIVE

Inductively coupled plasma-mass spectrometer (ICP-MS) tunes are performed to verify proper mass calibration and resolution and serves as an initial demonstration of instrument stability and precision.

## B. CRITERIA

ICP-MS tuning (instrument performance) criteria are not sample-specific. Since conformance is determined using standard materials, these criteria should be met under all circumstances. The CLP ICP-MS method QC acceptance criteria listed in Appendix I should be used as the default criteria when none exist for the ICP-MS 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.

## C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
1. a. Verify that the ICP-MS instrument was tuned prior to instrument calibration.	<p>All potential impacts on the sample data resulting from tuning 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> 1. a. If the ICP-MS instrument was not tuned prior to calibration, then the validator should reject (R) all data not associated with a tune. The validator may need to obtain additional information from the laboratory. Rejected data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
<p>1. b. i. Verify that the ICP-MS tuning solution contained the method-required analytes representing the mass regions of interest.</p> <p>* ii. Verify that the method-required number of analyses or scans of the ICP-MS tuning solution was performed.</p>	<p>1. b. If the laboratory did not use the required analytes spanning the analytical range to tune the instrument or if the tuning solution was not analyzed at the required frequency, then the validator must use professional judgment to determine whether the associated sample data should be qualified. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p>
<p>2. Verify from the reported results that the mass calibration, spectrometer resolution (peak width) and %RSD of the absolute signals for the analytes in the tuning solution are within the method QC acceptance criteria.</p>	<p>2. a. If tabulated results forms are not present for each ICP-MS tune under which samples are analyzed, then the validator should contact the laboratory to obtain the tabulated forms.</p> <p>b. If the mass calibration is not within the method-required mass range of the true mass for any isotope in the tuning solution, then the validator should estimate (J) all positive detects and estimate (UJ) all non-detects in all samples associated with that tune.</p> <p>c. If the mass resolution (peak width) is not within the method QC acceptance criteria at the specified peak height, then the validator should estimate (J) all positive detects and estimate (UJ) all non-detects in all samples associated with that tune.</p> <p>d. If the %RSD of the absolute signals of any analyte in the tuning solution is not within the method QC acceptance criteria, then the validator should estimate (J) all positive detects and estimate (UJ) all non-detects in all samples associated with that tune to indicate possible instrument instability.</p>

C. EVALUATION	D. ACTION
*3. Check raw data to verify that the tune data are accurately reported on the tabulated forms.	3. 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.
*4. Review standard preparation logs (if available in the data package) to verify that the analytes in the tuning solution were at the method-required concentrations.	4. If standards preparation data are not included in the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to validate sample data. If necessary, the validator should contact the laboratory to obtain standards preparation information.

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

C.1.b.ii, C.3, C.4

Table INORG-II-1:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON THE ICP-MS TUNE**

Sample Results	Mass Calibration (amu) > QC Limit	Mass Resolution/ Peak Width (amu) > QC Limit	%RSD > QC Limit
Detects	J	J	J
Non-detects	UJ	UJ	UJ

**E. EXAMPLES**Example #1: (Mass calibration outside criteria)

Tabulated tuning data generated under CLP SOW ILM05.4 show a measured mass of 24.16 amu which exceeds the method QC criteria of  $\leq 0.1$  amu from the true mass of 24 amu for magnesium. The validator estimates (J) all positive detects and estimates (UJ) all non-detects in all samples associated with that tune on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

Example #2: (Mass resolution/peak width outside criteria)

The validator examines the tabulated tune data generated under EPA Method 6020. The peak width for cobalt is 1.3 amu at 10% peak height which is outside the method criteria of less than 0.9 amu full width at 10% peak height. The validator estimates (J) all positive detects and estimates (UJ) all non-detects in all samples associated with that tune on the Data Summary Table. The validator discusses the instrument performance and the resulting qualifications in the Data Validation Memorandum.

Example #3: (ICP-MS tune %RSD outside criteria)

The validator examines the tabulated tune data generated under CLP SOW ILM05.4. The %RSD is 7.5% for beryllium and 6.3% for indium, which exceed the method QC criteria of 5% RSD. The validator estimates (J) all positive detects and estimates (UJ) all non-detects in all samples associated with that tune on the Data Summary Table. The validator discusses the instrument instability and precision problem and the resulting qualifications in the Data Validation Memorandum.

## III. CALIBRATIONS

## A. OBJECTIVE

Compliance requirements for instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing calibration verification (CCV) demonstrates that the instrument calibration is still valid by checking the performance of the instrument on a continual basis. The Quantitation Limit Check Standard verifies that the instrument is capable of producing acceptable quantitative data at the low end of the calibration curve.

## 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. Initial Calibration

- a. Initial calibration standards containing target analytes at method-specified concentrations are analyzed daily, each time the instrument is set up, and prior to the analysis of any field samples, QC samples, and blanks. The method-required number of calibration standards and replicates must be analyzed for each initial calibration using the same instrumental conditions that are used to analyze field samples, QC samples, and blanks.
- b. For methods which require the determination of correlation coefficients for the calibration curve, correlation coefficients must be within the method QC acceptance criteria.

## 2. Initial and Continuing Calibration Verifications

- a. Initial and continuing calibration verification standards must contain target analytes at method-specified concentrations and must be from a source different from that of the initial calibration standards. The ICV is analyzed immediately after initial calibration and the CCV is analyzed at a frequency of every ten samples or every two hours during an analytical run, whichever is more frequent, and at the end of the run after the last sample.
- b. The initial and continuing calibration verification recoveries for all target analytes must be within the QC acceptance criteria specified in the method.
- c. For methods which require replicate analyses (i.e., replicate integrations), the percent relative standard deviation (%RSD) must be within the method QC acceptance criteria.

3. Quantitation Limit Check Standard
  - a. The Quantitation Limit Check Standard must contain target analytes in the reagent blank at or near the quantitation limit according to the method requirements and is analyzed after the initial calibration is verified. Some methods may require the analysis of a Laboratory Fortified Blank in place of or in addition to the QL Check Standard. (See Section XII, Sensitivity Check.)
  - b. The Quantitation Limit Check Standard recoveries must be within the method QC acceptance criteria for all target analytes.

**C. EVALUATION/ D. ACTION**

C. EVALUATION	D. ACTION
<p>1. Initial Calibration</p> <ol style="list-style-type: none"> <li>a. Verify that the correct number of instrument calibration standards were prepared and analyzed at the method-required concentrations and frequency, and that the correct number of replicate analyses was performed.</li> </ol>	<p>All potential impacts on the sample data resulting from initial and continuing calibration 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. Initial Calibration</p> <ol style="list-style-type: none"> <li>a. If the laboratory did not use the required number of standards at the correct concentrations, frequency, and number of replicates when analyzing the initial calibration standards, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.</li> </ol>

C. EVALUATION	D. ACTION
<p>*1. b. For methods that require determination of correlation coefficients for the calibration curve, verify that the method-required correlation coefficient criteria were met.</p>	<p>1. b. If the correlation coefficient for any target analyte did not meet the method QC acceptance criteria, then the validator should:</p> <ul style="list-style-type: none"> <li>i. Estimate (J) positive detects for the affected analyte in all samples associated with the initial calibration.</li> <li>ii. Estimate (UJ) non-detects for the affected analyte in all samples associated with the initial calibration.</li> <li>iii. Depending on the degree of the deviation from linearity, the validator may use professional judgment to reject (R) all positive detects and/or all non-detects. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</li> </ul>
<p>2. Initial and Continuing Calibration Verifications</p> <p>a. Verify that the ICV and CCV standards were analyzed at the method-required concentrations and frequency. Verify that the source of the ICV and CCV is different from that of the initial calibration standards.</p>	<p>2. Initial and Continuing Calibration Verifications</p> <p>Qualification is based on samples associated with a particular ICV or CCV. For an ICV not meeting criteria, action is applied to the affected analyte in all samples reported from the same analytical run. For a CCV not meeting criteria, generally, action is applied to the affected analyte in all samples bracketed by that CCV, that is, in all sample analyzed between the previous acceptable CCV analysis and the subsequent acceptable CCV analysis in the same analytical run.</p> <p>a. If the laboratory did not analyze the ICV and CCV standards at the required concentrations and frequency and did not use a separate source, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.</p>

C. EVALUATION	D. ACTION
<p>2. b. Verify that all ICV and CCV recoveries are within the QC acceptance criteria specified in the method.</p>	<p>2. b. i. If the ICV or CCV percent recovery for any target analyte is greater than the upper limit of the method QC acceptance criteria but within the following ranges:</p> <ul style="list-style-type: none"> <li>- ICP-AES/MS: upper limit <math>&lt; \%R \leq 125\%</math></li> <li>- Hg, CN: upper limit <math>&lt; \%R \leq 130\%</math></li> </ul> <p>then the validator should:</p> <ul style="list-style-type: none"> <li>- Estimate (J) positive detects for the affected analyte in all samples associated with that ICV or CCV.</li> <li>- Accept non-detects for the affected analyte in all samples associated with that ICV or CCV.</li> </ul> <p>ii. If the ICV or CCV percent recovery is greater than the following limits:</p> <ul style="list-style-type: none"> <li>- ICP-AES/MS: <math>\%R &gt; 125\%</math></li> <li>- Hg, CN: <math>\%R &gt; 130\%</math></li> </ul> <p>then the validator should:</p> <ul style="list-style-type: none"> <li>- Reject (R) positive detects for the affected analyte in all samples associated with that ICV or CCV.</li> <li>- Accept non-detects for the affected analyte in all samples associated with that ICV or CCV.</li> </ul>

C. EVALUATION	D. ACTION
<p>2. b. Continued from above.</p>	<p>2. b. iii. If the ICV or CCV percent recovery is less than the lower limit of the method QC acceptance criteria but within the following ranges:</p> <ul style="list-style-type: none"> <li>- ICP-AES/MS: <math>75\% \leq \%R &lt; \text{lower limit}</math></li> <li>- Hg, CN: <math>70\% \leq \%R &lt; \text{lower limit}</math></li> </ul> <p>then the validator should:</p> <ul style="list-style-type: none"> <li>- Estimate (J) positive detects for the affected analyte in all samples associated with that ICV or CCV.</li> <li>- Estimate (UJ) non-detects for the affected analyte in all samples associated with that ICV or CCV.</li> </ul> <p>iv. If the ICV or CCV percent recovery is less than the following limits:</p> <ul style="list-style-type: none"> <li>- ICP-AES/MS: <math>\%R &lt; 75\%</math></li> <li>- Hg, CN: <math>\%R &lt; 70\%</math></li> </ul> <p>then the validator should:</p> <ul style="list-style-type: none"> <li>- Reject (R) positive detects and non-detects for the affected analyte in all samples associated with that ICV or CCV.</li> </ul>

C. EVALUATION	D. ACTION
<p>2. c. Evaluate the appropriateness of qualifying the affected analyte in all samples which are reported from the same analytical run.</p> <p>d. Evaluate the appropriateness of qualifying additional analytes when the majority of analytes have ICV or CCV recoveries outside the QC criteria specified in the method.</p> <p>* e. Verify that, for methods which require replicate analyses (i.e., replicate integrations), the %RSD is within the method QC acceptance criteria.</p>	<p>2. c. Generally, action applies to the affected analyte in samples associated with the specific CCV. However, professional judgment may be used to qualify the affected analyte in all samples reported from the same analytical run. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p> <p>d. For ICP-AES or ICP-MS multi-analyte analysis, if the majority of the ICV or CCV recoveries are outside method QC acceptance criteria, this may indicate a more serious problem with the instrument's stability. The validator should use professional judgment to qualify or reject all analytes in all samples associated with that ICV or CCV based on the number of analytes with recoveries outside QC limits and the direction and degree of deviation. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p> <p>e. If any %RSD for replicate analyses in the ICV or CCV is outside the method QC acceptance criteria, then the validator should use professional judgment to either accept or qualify associated sample data. The validator should evaluate whether the instrument consistently generates erratic responses or whether the imprecision is isolated to a specific ICV or CCV. The %RSDs of other QC samples should also be evaluated. 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>3. Quantitation Limit Check Standard</p> <p>a. Verify that the Quantitation Limit Check Standard contains all target analytes at concentrations equal to or near their quantitation limits, as required by the method, and that the QL Check Standard was analyzed at the required frequency.</p> <p>b. Verify that all QL Check Standard recoveries are within the method QC acceptance criteria.</p>	<p>3. Quantitation Limit Check Standard</p> <p>Qualification is based on samples associated with a particular QL Check Standard. For a QL Check Standard not meeting criteria, generally, action is applied to the affected analyte in all samples bracketed by that QL Check Standard, that is, in all sample analyzed between the previous acceptable QL Check Standard analysis and the subsequent acceptable QL Check Standard analysis from the same analytical run.</p> <p>a. If a QL Check Standard was not analyzed for all target analytes at the required concentration and frequency, the validator should use professional judgment to assess the impact of analytical sensitivity on data quality. The validator should review any other low level QC data to determine the action to be taken.</p> <p>b. If any of the QL Check Standard recoveries are outside the method QC acceptance criteria, then the QL Check Standard results should be used to qualify sample data for the affected analytes that are included in the solution. The validator should use professional judgment to qualify sample data for analytes not present in the check standard, taking into account information that may exist in the sample delivery group for other low level standards.</p> <p>Note: If the QL Check Standard was spiked at low level concentrations other than the quantitation limit, then qualifications for positive detects should be based on 2x the true value of the low level check standard rather than on 2x the QL.</p>

C. EVALUATION	D. ACTION
<p>3. b. Continued from above.</p>	<p>3. b. i. If a QL Check Standard recovery for any target analyte is greater than the upper limit of the method QC acceptance criteria, but less than or equal to 180%, then the validator should:</p> <ul style="list-style-type: none"> <li>- Estimate (J) positive detects less than 2x the QL Check Standard true value for the affected analyte in all samples associated with that standard to indicate potential high bias.</li> <li>- Accept non-detects for the affected analyte in all samples associated with that QL Check Standard.</li> </ul> <p>ii. If a QL Check Standard analyte recovery is greater than 180%, then the validator should:</p> <ul style="list-style-type: none"> <li>- Use professional judgment to estimate (J) or reject (R) positive detects less than 2x the QL Check Standard true value for the affected analyte in all samples associated with that standard.</li> <li>- Use professional judgment to accept or estimate positive detects greater than or equal to 2x the QL Check Standard true value but less than the true value of the next highest concentration QC sample to indicate potential high bias.</li> <li>- Accept non-detects for the affected analyte in all samples associated with that QL Check Standard.</li> </ul>

C. EVALUATION	D. ACTION
<p>3. b. Continued from above.</p>	<p>3. b. iii. If a QL Check Standard analyte recovery is less than the lower limit of the method QC acceptance criteria, but greater than or equal to 50%, then the validator should:</p> <ul style="list-style-type: none"> <li>- Estimate (J) positive detects less than 2x the QL Check Standard true value for the affected analyte in all samples associated with that standard to indicate potential low bias.</li> <li>- Estimate (UJ) non-detects for the affected analyte in all samples associated with that QL Check Standard to indicate potential low bias.</li> </ul> <p>iv. If a QL Check Standard analyte recovery is less than 50%, then the validator should:</p> <ul style="list-style-type: none"> <li>- Use professional judgment to estimate (J) or reject (R) positive detects less than 2x the QL Check Standard true value for the affected analyte in all samples associated with that standard.</li> <li>- Use professional judgment to accept or estimate positive detects greater than or equal to 2x the QL Check Standard true value but less than the true value of the next highest concentration QC sample to indicate potential low bias.</li> <li>- Reject (R) non-detects for the affected analyte in all samples associated with that QL Check Standard to indicate that the data are unusable due to the possibility of false negatives.</li> </ul>

C. EVALUATION	D. ACTION
<p>3. b. Continued from above.</p>	<p>3. b. v. The validator should use professional judgment to estimate (J) or reject (R) low level sample results when the QL Check Standard exceeds the acceptance criteria, taking into consideration project DQOs.</p> <p>c. The validator should use professional judgment to take action on positive detects greater than 2x the true value of the low level check standard, taking into consideration the laboratory's accuracy for other QC samples and standards and project DQOs. The validator should evaluate all relevant QC data which may provide information on the laboratory's ability to accurately quantitate target analytes at concentration ranges greater than 2x the true value of the low level check standard. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.</p> <p>d. For ICP-AES or ICP-MS multi-analyte analysis, if the majority of the Quantitation Limit Check Standard recoveries are outside method QC acceptance criteria, this may indicate a more serious problem with the instrument's stability and/or accuracy at the low end of the calibration curve. The validator may use professional judgment to qualify or reject all sample results associated with that QL Check Standard based on the number of analytes with recoveries outside QC limits and the degree of deviation. 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>*4. a. Review standard preparation logs (if provided in the data package) to ensure that all initial calibration, initial calibration verification, continuing calibration verification and Quantitation Limit Check Standard concentrations are accurate and traceable.</p> <p>* b. i. Check and recalculate the initial calibration, initial calibration verification, continuing calibration verification, and QL Check Standard concentrations and percent recoveries for at least one analyte per method (if standards preparation documentation was provided in the data package). Verify that the calculated values agree within 10% of the laboratory reported values.</p> <p>ii. For methods that require determination of calibration curve correlation coefficients, check and recalculate the correlation coefficient for at least one target analyte per method. Verify that the recalculated value agrees within <math>\pm 10\%</math> of the laboratory reported value.</p>	<p>4. a. If standards preparation data have not been submitted with the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to facilitate the validation of sample data. If necessary, the validator should contact the laboratory to obtain standards preparation information.</p> <p>b. If errors greater than 10% are detected in the standard concentration 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 subsections are applicable to a Tier III data validation:

C.1.b, C.2.e, C.4.a, C.4.b

Table INORG-III-1:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON ICV AND CCV  
PERCENT RECOVERIES**

Sample Results	% Recovery				
	ICP: %R < 75% Hg/CN: %R < 70%	ICP: 75% ≤ %R < LL Hg/CN: 70% ≤ %R < LL	LL ≤ %R ≤ UL	ICP: UL < %R ≤ 125% Hg/CN: UL < %R ≤ 130%	ICP: %R > 125% Hg/CN: %R > 130%
Detects	R	J	A	J	R
Non-detects	R	UJ	A	A	A

LL = Lower limit of method QC acceptance criteria  
 UL = Upper limit of method QC acceptance criteria

Table INORG-III-2:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON QUANTITATION LIMIT  
CHECK STANDARD RECOVERIES**

Sample Results	% Recovery				
	%R < 50%	50% ≤ %R < LL	LL ≤ %R ≤ UL	UL < %R ≤ 180%	%R > 180%
Detects*	J/R (< 2x TV) ** Prof. Judg. (≥ 2x TV)**	J (< 2x TV)	A	J (< 2x TV)	J/R (< 2x TV)** Prof. Judg. (≥ 2x TV)**
Non-detects	R	UJ	A	A	A

LL = Lower limit of method QC acceptance criteria  
 UL = Upper limit of method QC acceptance criteria

- \* Action is applied to positive detects less than 2x the true value of the QL Check Standard.
- \*\* Professional judgment may be used to estimate or reject positive detects less than 2x the true value taking into account project DQOs. Professional judgment should be used to accept or estimate positive detects greater than or equal to 2x the true value of the Check Standard but less than the next highest concentration QC sample or standard.

**E. EXAMPLES**Example #1: (High ICV recovery for mercury)

Samples were analyzed for mercury by CLP SOW ILM05.4 in two analytical runs. The first run occurred on 8/1/07 and the second run occurred on 8/2/07. The ICV analyzed on 8/1/07 recovered at 118% and was within the method QC acceptance criteria of 80-120% recovery. The ICV analyzed on 8/2/07 recovered at 122%, above the upper QC limit. Therefore, only the samples analyzed and reported from the second run on 8/2/07 were affected. The validator estimates (J) the positive detects for mercury reported from the 8/2/07 run and accepts the mercury non-detects on the Data Summary Table. The validator notes in the Data Validation Memorandum that the qualified mercury results may be biased high.

Example #2: (Low CCV recovery for aluminum)

Samples were analyzed for aluminum by ICP-AES under CLP SOW ILM05.4. In the analytical run on 8/25/07, the CCV3 recovered at 82% for aluminum, below the method QC acceptance criteria of 90-110% recovery but above 75%. The validator notes that the other calibration verifications in the same run, ICV, CCV1, CCV2, and CCV4, all recovered within the method QC acceptance criteria. Samples MAED52 through MAED57, analyzed between CCV2 and CCV3, and samples MAED58 through MAED60, analyzed between CCV3 and CCV4, were associated with the low CCV3 recovery. As a result, samples MAED52 through MAED60 were not bracketed by acceptable CCVs. The validator estimates (J) the positive detects and estimates (UJ) the non-detects for aluminum in samples MAED52 through MAED60 on the Data Summary Table. The validator notes in the Data Validation Memorandum that the aluminum results in these samples may be biased low.

Example #3: (High QL Check Standard recovery for chromium)

Chromium was analyzed by ICP-AES under CLP SOW ILM05.4. The CRI standard, or QL Check Standard, recovered at 140% for chromium, above the method QC criteria of 70-130%. Therefore, the validator estimates (J) positive detects less than 2x the QL and accepts non-detects on the Data Summary Table for chromium results reported from that analytical run. The validator discusses the potential high bias in the low level results and notes the sample qualifications in the Data Validation Memorandum.

Example #4: (Low QL Check Standard recovery for lead)

Lead was analyzed by ICP-AES under CLP SOW ILM05.4. The CRI standard, or QL Check Standard, recovered at 40%, well below the method QC criteria of 70-130%. Therefore, the validator uses professional judgment to reject (R) positive detects less than 2x the QL since low level lead near the QL was of concern at the site. Professional judgment was used to estimate (J) positive detects greater than or equal to 2x the QL but less than the ICV true value since the ICV was the next highest concentration standard in the analytical run, and non-detects were accepted for lead on the Data Summary Table. The validator notes in the Data Validation Memorandum that accuracy at the low end of the calibration curve is questionable.

## IV. BLANKS

## A. OBJECTIVE

The purpose of blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory and/or field activities and to subsequently assess their contribution to measurement error. The criteria for evaluation of laboratory blanks (preparation/method blanks and calibration/instrument blanks) may be applied to any blank associated with the samples. If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent measurement error associated with the entire data set, or if the problem is an isolated occurrence limited to specific samples.

## 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 frequency and types of blanks collected and analyzed must support the site-specific Data Quality Objectives (DQOs) as documented in the EPA-approved QAPP or SAP. Different types of blanks may be used to identify the source of potential contamination resulting in analytical and/or sampling measurement error. The following table lists types of blanks, the environment of those blanks, and the possible sources of contamination associated with those blanks:

BLANK	LABORATORY/FIELD	IDENTIFIES CONTAMINATION FROM
Preparation (Method) Blank	Laboratory	Laboratory and Reagents
Calibration (Instrument) Blank	Laboratory	Instrumentation
Bottle Blank	Field	Sample Container
Equipment Blank (Rinsate)	Field	Sampling Equipment

Note: Aqueous equipment (rinsate) blank results and bottle blank results will be used to determine blank action levels for aqueous samples typically based on a volume of 1 liter of blank sample. Ideally, soil/sediment blanks should be used to determine soil/sediment blank actions for soil/sediment samples based on a known weight of blank sample. However, aqueous equipment blanks and bottle blanks are often collected to evaluate contamination associated with soil/sediment sampling. **Aqueous equipment (rinsate) blank results and bottle blank results will not be used to determine blank action levels for non-aqueous samples.** Analytes that are present in both the non-aqueous sample and the associated aqueous equipment blank or bottle blank will be flagged EB (Equipment Blank) or BB (Bottle Blank), respectively. The degree of "sampling error" that this flagged sample result represents will be left to the determination of the end user.

2. Preparation (Method) Blanks

A preparation blank must be prepared (e.g., digested or distilled) and analyzed with each sample delivery group, with each batch of samples of similar matrix in each sample delivery group, or whenever a sample digestion or distillation is performed. The preparation blank must undergo all preparation and analysis procedures performed on samples. The preparation blank must be analyzed on the same instrument used to analyze the samples associated with the preparation blank.

3. Calibration (Instrument) Blanks

An initial calibration blank must be analyzed following instrument calibration at the beginning of the run but after the initial calibration verification and prior to sample analysis. A continuing calibration blank must be analyzed after every 10 samples or every two hours during an analysis run, and at the end of the run for each analyte and on each instrument used to analyze samples.

4. For ICP-MS analysis, all blanks should be spiked with internal standards according to the method. Blank internal standards must meet method internal standard QC acceptance criteria.

5. No contaminants should be present in the blanks.

6. No negative results should be observed in the blanks.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	<p>All potential impacts on the sample data resulting from blank 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>Action regarding unsuitable blank results depends on the circumstances and origin of the blank. <b>Qualification should be based upon a comparison of the sample concentration(s) with the highest blank concentration associated with the samples.</b> More than one blank action level for a given analyte may exist in a sample delivery group if samples are associated with different blanks. This may occur when samples are prepared in different batches, samples are analyzed in a separate analytical sequence, or if samples are associated with different equipment or bottle blanks. In cases of specific preparation and/or calibration blank contamination, the validator should use professional judgment to qualify only those samples associated with that isolated blank contamination. Likewise, the validator may need to apply blank qualifications to a sample delivery group based on associated equipment or bottle blank data that exist in another sample group data package. <b><u>Sample results must not be corrected by subtracting any blank values.</u></b></p>

C. EVALUATION	D. ACTION
<p>1. a. Verify that the correct number and type of blanks have been collected and analyzed in accordance with the EPA-approved QAPP or SAP.</p> <p>b. Ascertain if aqueous equipment (rinsate) blanks or aqueous bottle blanks have been collected with non-aqueous samples to identify sources of field contamination.</p>	<p>1. a. If the correct number and type of blanks have not been collected and analyzed, then the validator should note this deviation from the EPA-approved QAPP or SAP in the Data Validation Memorandum. The validator should use professional judgment to qualify sample data when blank data are absent.</p> <p>When required equipment (rinsate) or bottle blanks are not identified on the chain of custody, the validator must contact the sampler or site project manager to obtain this information and note this contact on the Blanks validation worksheet.</p> <p>b. If positive results are detected in the aqueous equipment (rinsate) blanks and/or bottle blanks <u>and</u> the associated <u>non-aqueous</u> samples, then the validator should flag (EB or BB) those detected analytes in the associated non-aqueous samples to indicate to the end user that an indeterminate amount of sampling error has potentially affected the sample results. (See Example #4.)</p>
<p>2. a. Verify that a preparation blank analysis has been reported once per matrix, preparation and analytical method, batch of samples prepared, and SDG.</p>	<p>2. a. If preparation blanks were not analyzed at the required frequency and for each matrix, preparation and analytical method, preparation batch, and SDG, then the validator should use professional judgment to determine whether the associated sample data should be qualified.</p>

C. EVALUATION	D. ACTION
<p>*2. b. Verify from the raw data that the digestion/distillation and analysis dates and times, sample IDs, etc., are accurately reported on the tabulated result forms.</p>	<p>2. b. If review of the raw data reveals discrepancies and/or transcription errors, 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>
<p>3. a. Verify from the Blanks form and Analysis Run Log form that a calibration blank was analyzed after initial calibration of the instrument, at the beginning of the run but after the initial calibration verification, after every 10 samples or every two hours, and at the end of the run.</p> <p>* b. Verify from the raw data that the analysis dates and times, sample IDs, sequence of blank analyses, etc., are accurately reported on the tabulated result forms.</p>	<p>3. a. If calibration blanks were not analyzed at the required frequency, then the validator should use professional judgment to determine whether the associated sample data should be qualified.</p> <p>b. If review of the raw data reveals discrepancies and/or transcription errors, 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>4. a. For ICP-MS data, verify that the blank internal standard responses meet method QC acceptance criteria.</p> <p>* b. Check 10% of the raw data for each blank to verify that internal standard responses and/or percent relative intensities have been correctly transcribed to tabulated forms and that percent relative intensities have been correctly calculated.</p>	<p>4. a. If blank internal standard responses do not meet method QC acceptance criteria, then the validator should use professional judgment in applying blank actions. The possibility of false positives or false negatives being incorrectly reported for the blank should be evaluated.</p> <p>b. If the laboratory has incorrectly transcribed and/or calculated the data, 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. Target Analyte Contaminants Detected in Blanks</p>	<p>5. Target Analyte Contaminants Detected in Blanks</p> <p>If a contaminant is found in a blank but <u>not</u> in the associated sample, then no action is taken. If a contaminant is found in both a blank and the associated sample, then the validator should note this problem in the Data Validation Memorandum and qualify the data according to the following guidance.</p> <p><b>Blank Action Level:</b> The blank action level is determined for each contaminant detected in the blanks associated with each sample. The blank action level is determined by multiplying by five the highest concentration of each contaminant detected among the blanks associated with each sample.</p> <p><b>Note:</b> If the blank action level for an analyte is determined using the value from an equipment blank or bottle blank, then the positive values in the equipment or bottle blank should be reported unqualified on the Data Summary Tables. However, if the blank action level is determined using the value from a laboratory blank (e.g., preparation or calibration blank), then the positive values in the equipment or bottle blanks should be qualified. (See Example #6.)</p>

C. EVALUATION	D. ACTION
<p>5. a. Determine if any target analytes are present at concentrations greater than or equal to the MDL in any of the blanks.</p>	<p>5. a. If any target analyte is present at a concentration greater than or equal to the MDL in any of the blanks, then the following actions are taken.</p> <ul style="list-style-type: none"> <li>i. Positive sample results greater than the blank action level:                             <ul style="list-style-type: none"> <li>- If a positive sample result for an analyte is greater than the blank action level (5 times the highest concentration in any associated blank) and greater than or equal to the sample quantitation limit, then the analyte's concentration should be reported as unqualified.</li> <li>- If a positive sample result is greater than the blank action level but is less than the sample quantitation limit, then no further qualification is necessary and the estimated sample result should be reported on the Data Summary Table.</li> </ul> </li> <li>ii. Positive sample results less than or equal to the blank action level:                             <ul style="list-style-type: none"> <li>- If a positive sample result for an analyte is less than or equal to the blank action level but is greater than or equal to the sample quantitation limit, then the sample quantitation limit for that analyte should be elevated to the concentration found in the sample and reported as not detected (U). (See Example #2.) The validator may use professional judgment to determine if further elevation of the quantitation limit is required.</li> </ul> </li> </ul>

C. EVALUATION	D. ACTION
<p>5. a. Continued from above.</p>	<p>5. a. ii. Continued from above.</p> <ul style="list-style-type: none"> <li>- If a positive sample result for an analyte is less than or equal to the blank action level and is also less than the sample quantitation limit, then the sample quantitation limit should be reported on the Data Summary Tables as a non-detect (U). (See Example #1.)</li> </ul> <p>Note:</p> <p>The validator should note that blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results may not be on the same basis (units, dilution) as the calibration blank data. These factors must be taken into consideration when applying the "5x" criteria. The reviewer may find it easier to work from the raw data when comparing soil sample data to blank data. (See Example #5.)</p> <p>Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample data is deemed necessary. If the validator determines that the contamination originates from a source other than the sample, the sample data should be qualified. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurrence can be detected when contaminants are found in the diluted sample result but are absent in the undiluted sample result. Since both results may not be reported, it may be impossible to verify this source of contamination. In this case, the "5x" rule may not apply; the target analyte should be reported as not detected (U), and an explanation of the data qualification rationale should be provided in the Data Validation Memorandum.</p>

C. EVALUATION	D. ACTION
<p>5. b. Determine if contamination greater than the CRQL for any analyte exists in any of the blanks.</p> <p>c. Determine if blank contaminants are consistently found in the laboratory blanks. Determine if instrument contamination is isolated to specific sample runs.</p>	<p>5. b. If contamination greater than the CRQL for any analyte exists in any blank, depending on the concentration of the contaminant in the sample compared to the blank, as well as the project DQOs, the validator may use professional judgment to reject (R) positive results for the affected analyte in samples associated with that blank due to the interference. This serious problem should be discussed in the Data Validation Memorandum.</p> <p>c. While preparation, equipment, and bottle blanks are associated with specific samples, the association of calibration blanks to samples analyzed within the same analytical run is not always straightforward. Generally, for ICBs not meeting blank criteria, action applies to all samples analyzed in the same analytical run as the ICB. For CCBs, the validator should review the blank data to determine whether the blank contamination is isolated to specific CCBs or if the blank contamination appears consistently throughout the analytical run. If the validator determines that instrument contamination is isolated to a specific CCB and is limited to a specific set of samples, then the validator may use professional judgment to apply blank actions only to those samples bracketed by the affected CCB. If the validator determines that contamination is not isolated to a specific set of samples within a sample run, then the validator may use professional judgment to apply blank actions to all samples analyzed in the same analytical run. If target analytes are consistently found in the laboratory blank(s), it may indicate a systematic problem in the laboratory and should be noted in the Data Validation Memorandum.</p>

C. EVALUATION	D. ACTION
<p>5. d. Determine if blank contamination exists solely in field blanks.</p> <p>e. Evaluate the overall contamination in each type of blank to ascertain probable source(s) of contamination. For example, a contaminated equipment blank might indicate decontamination problems if the preparation, calibration, and bottle blanks were all clean.</p>	<p>5. d. If contamination exists solely in the equipment (rinsate) or bottle blank at levels greater than the CRQL, then the validator should notify the sampler. The call should be documented in a telephone log that is included in the Data Validation Memorandum and the date of contact should be noted on the Blank Analysis Worksheet.</p> <p>e. If a review of the various types of blanks identifies a potential source of blank contamination, then the validator should discuss this problem in the Data Validation Memorandum. The validator should identify whether the measurement error is a result of either sampling or analytical error or both (see the Data Validation Manual, p. 1).</p>
<p>6. <b>Negative Blank Results Reported For Target Analytes</b></p> <p>a. Determine if any target analytes are reported at concentrations less than or equal to the negative MDL in any of the blanks. (Note: The tabulated forms may not report negative values. It may be necessary to review the raw data for this information.)</p>	<p>6. <b>Negative Blank Results Reported For Target Analytes</b></p> <p><b>Negative Blank Action Level:</b> The negative blank action level is determined for each target analyte reported at less than or equal to the negative MDL in the blanks associated with each sample. The negative blank action level is the absolute value of five times the lowest negative value for each analyte reported among all of the blanks associated with each sample.</p> <p>a. Any target analyte reported as a negative value less than or equal to the negative MDL in any blank should be carefully evaluated to determine its effect on the sample data.</p> <p>i. Positive sample results greater than the negative blank action level:</p> <ul style="list-style-type: none"> <li>- If a positive sample result for an analyte is greater than the negative blank action level and greater than or equal to the sample quantitation limit, then the sample result should be reported as unqualified.</li> </ul>

C. EVALUATION	D. ACTION
<p>6. a. Continued from above.</p>	<p>6. a. i. Continued from above.</p> <ul style="list-style-type: none"> <li>- If a positive sample result for an analyte is greater than the negative blank action level and is less than the sample quantitation limit, then no further qualification is necessary and the estimated sample result should be reported on the Data Summary Table.</li> </ul> <p>ii. Positive sample results less than or equal to the negative blank action level:</p> <ul style="list-style-type: none"> <li>- If a positive sample result for an analyte is less than or equal to the negative blank action level and greater than or equal to the sample quantitation limit, then estimate (J) the positive detect for the affected analyte to indicate potential low bias. The validator may use professional judgment to determine if further qualification of sample results greater than the negative blank action level is required.</li> <li>- If a positive sample result for an analyte is less than or equal to the negative blank action level and is less than the sample quantitation limit, then no further action is required and the estimated sample result should be reported on the Data Summary Table.</li> </ul> <p>iii. Estimate (UJ) non-detects for the affected analyte in all samples associated with the negative blank to indicate potential low bias.</p>

C. EVALUATION	D. ACTION
<p>6. b. Determine if negative blank values less than the negative quantitation limit for any analyte exist in any of the blanks.</p> <p>c. Determine if both negative and positive blank values are associated with a particular analyte.</p>	<p>6. b. Any blank reported with a negative blank value should be carefully evaluated to determine its effect on the sample data. Negative calibration blank values may indicate problems with the initial calibration or instrument/baseline drift. Negative blank values less than the negative quantitation limit may indicate a major instrument problem or a major change in instrument conditions and should be noted in the Data Validation Memorandum. If negative blank values less than the negative quantitation limit exist in any blank, then the validator may use professional judgment to reject (R) non-detects or positive detects for the affected analyte in samples associated with the blank. This serious problem should be discussed in the Data Validation Memorandum.</p> <p>c. If both negative and positive blank results are reported for an analyte and are associated with the same sample, then the validator should use professional judgment when applying blank actions. The following general guidance should be used when the positive sample result is less than or equal to both the positive and negative blank action levels:</p> <ul style="list-style-type: none"> <li>- If a positive sample result is greater than or equal to the sample quantitation limit, then the sample quantitation limit should be elevated to the concentration found in the sample and reported as a non-detect (U) for positive blank actions. The sample quantitation limit is then estimated (UJ) for negative blank actions on the Data Summary Table.</li> </ul>

C. EVALUATION	D. ACTION
6. c. Continued from above.	6. c. Continued from above.  - If a positive sample result is less than the sample quantitation limit, then the sample quantitation limit is reported as a non-detect (U) due to positive blank actions. The sample quantitation limit is then estimated (UJ) due to negative blank actions on the Data Summary Table.
*7. Review the raw data to confirm the presence of target analytes in the blanks and to evaluate the presence of additional contaminants. Confirm any negative results reported for the blanks as well as any negative blank results in the raw data that were not reported on the tabulated forms.	7. If review of the raw data suggests that additional contaminants or additional negative results are present or, conversely, the review indicates false positive or false negative results have been reported, then the validator should contact the laboratory to obtain additional information and/or 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.2.b, C.3.b, C.4.b, C.7

Table INORG-IV-1:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON  
POSITIVE BLANK RESULTS**

Positive Blank Result	Sample Result	Action	
≥ MDL	> Blank Action Level and	≥ QL	A
		< QL	No further action (report estimated sample result)
	≤ Blank Action Level and	≥ QL	U - Raise the QL to the sample result and report as a non-detect
		< QL	U - Report the QL
	Non-detect (U)		A

Blank Action Level = 5x the highest blank concentration associated with the sample

QL = Sample Quantitation Limit

Note: Aqueous equipment (rinsate) and bottle blank results are not used to determine blank action levels for non-aqueous samples. Analytes present in both the non-aqueous sample and the associated aqueous equipment or bottle blanks should be flagged EB or BB, respectively.

Table INORG-IV-2:

**QUALIFICATION OF INORGANIC ANALYTES BASED ON  
NEGATIVE BLANK RESULTS**

Negative Blank Result	Sample Result	Action	
≤ (-MDL)	> Negative Blank Action Level and	≥ QL	A
		< QL	No further action (Report estimated sample result)
	≤ Negative Blank Action Level and	≥ QL	J - Estimate the sample result
		< QL	No further action (report estimated sample result)
	Non-detect (U)		UJ - Estimate the QL

Negative Blank Action Level = absolute value of 5x the lowest negative blank value associated with the sample

QL = Sample Quantitation Limit

## E. EXAMPLES

Example #1: (Initial calibration blank target analyte contaminant; Sample result  $\leq$  blank action level and  $<$  QL)

The initial calibration blank (ICB) contained the highest concentration of copper among all of the blanks analyzed. In addition, all field samples analyzed were associated with the same contaminated initial calibration blank. Copper is detected in an aqueous field sample at less than the CRQL and less than 5x the ICB concentration.

<u>5x Rule</u>	
	<u>ug/L</u>
Initial Calibration Blank Result	8
CRQL	25
Copper Sample Result	21 J
Blank Action Level	40 (5x8)
Qualified Sample Result	25 U

In this case, the copper sample result is less than the blank action level of 40 ug/L and is, therefore, reported as a non-detect at the CRQL on the Data Summary Table. The validator notes this problem in the Data Validation Memorandum.

Example #2: (Equipment blank target analyte contaminant; Sample result  $\leq$  blank action level and  $\geq$  QL)

An equipment blank was associated with a sample delivery group of aqueous samples. The equipment blank contained the highest concentration of sodium among all of the blanks analyzed. In addition, all aqueous field samples analyzed were associated with the same contaminated equipment blank. Sodium is detected in an aqueous field sample at greater than the CRQL and less than 5x the equipment blank concentration.

<u>5x Rule</u>	
	<u>ug/L</u>
Equipment Blank Result	6400
CRQL	5000
Sodium Sample Result	8700
Blank Action Level	32000 (5x6400)
Qualified Sample Result	8700 U

In this case, the sodium sample result is less than the blank action level of 32000 ug/L. The validator elevates the sample quantitation limit for sodium to the sample concentration on the Data Summary Table and reports the result as 8700 U since the result is less than the blank action level. This problem is noted in the Data Validation Memorandum.

## E. EXAMPLES (continued)

Example #3: (Preparation blank contamination; Sample result > blank action level)

Calcium is detected in an aqueous field sample at greater than 5x the preparation blank concentration, the highest level of calcium detected among all of the blanks associated with the sample.

	<u>5x Rule</u>
Preparation Blank Result	<u>ug/L</u> 1300
CRQL	5000
Calcium Sample Result	18200
Blank Action Level	6500 (5x1300)

The calcium sample result is greater than the blank action level of 6500 ug/L and is reported unqualified on the Data Summary Table.

Example #4: (Target analyte contamination in aqueous equipment blank associated with soil samples)

An equipment blank (rinsate) was associated with a sample delivery group of soil samples. The validator examines the data and finds that the equipment blank contains 20 ug/L of zinc. The validator then reviews all other blank data and finds no further zinc contamination. Soil sample MACN40 contains 11 mg/kg of zinc.

The validator reports the zinc result for sample MACN40 as 11 (EB) on the Data Summary Table to indicate to the end user that sampling error has potentially affected the sample results and notes this information in the Data Validation Memorandum.

## E. EXAMPLES (continued)

Example #5: (Preparation blank contaminant; Application of sample weights, % solids, and volumes)

Soil samples were analyzed under CLP SOW ILM05.4. The preparation blank contained the highest concentrations of nickel (3 mg/kg) and aluminum (5 mg/kg) among all of the blanks associated with the samples in this sample delivery group. Soil sample MACN42 contained nickel at 13 mg/kg and aluminum at 2 mg/kg.

The validator calculates the sample quantitation limits for nickel and aluminum in sample MACN42 for 1.0 g digested, and 80% solids.

$$\text{Nickel QL} = \frac{\text{CRQL}}{\% \text{ solids}} = \frac{4 \text{ mg/kg}}{0.8} = 5 \text{ mg/kg}$$

$$\text{Aluminum QL} = \frac{\text{CRQL}}{\% \text{ solids}} = \frac{20 \text{ mg/kg}}{0.8} = 25 \text{ mg/kg}$$

The validator applies the following actions to the nickel and aluminum results for sample MACN42:

<u>5x Rule - Sample MACN42</u>			
	<u>Nickel</u> <u>mg/kg (dry wt.)</u>		<u>Aluminum</u> <u>mg/kg (dry wt.)</u>
Prep. Blank Result	3	Prep. Blank Result	5
Sample QL	5	Sample QL	25
Sample Result	13	Sample Result	2 J
Blank Action Level	15 (5x3)	Blank Action Level	25 (5x5)
Qualified Sample Result	13 U	Qualified Sample Result	25 U

- The sample quantitation limit for nickel is elevated to the sample result on the Data Summary Table and is reported as 13 U since the result is greater than the sample quantitation limit but less than the blank action level.
- The aluminum sample result is replaced with the sample quantitation limit and is reported as 25 U on the Data Summary Table since the positive sample result of 2 mg/kg was below both the sample quantitation limit and the blank action level.

The validator notes all actions taken in the Data Validation Memorandum.

## E. EXAMPLES (continued)

Example #6: (Application of aqueous laboratory blank action levels to equipment blanks)

The preparation blank associated with a batch of aqueous samples was contaminated with 90 ug/L of iron and vanadium was not detected. The equipment blank associated with this batch of samples was contaminated with 80 ug/L of iron and 60 ug/L of vanadium. Since iron was detected in both the preparation blank and the equipment blank, the blank with the highest iron concentration is used to determine the blank action level. The preparation blank concentration is, therefore, used to determine the blank action level for iron.

	Iron <u>ug/L</u>	<u>5x Rule</u>	Vanadium <u>ug/L</u>
Preparation Blank Result	90		Preparation Blank Result 50 U
Equipment Blank Result	80		Equipment Blank Result 60
CRQL	100		CRQL 50
Blank Action Level	450 (5x90)		Blank Action Level 300 (5x60)
Qualified Equipment Blank	100 U		Qualified Equipment Blank 60

The iron positive detect in the equipment blank is reported as a non-detect at the CRQL, 100 U, on the Data Summary Table since the equipment blank result is less than the blank action level established by the preparation blank. The blank action level for vanadium is determined using the value from the equipment blank and, therefore, the vanadium positive detect in the equipment blank is reported unqualified as 60 ug/L on the Data Summary Table.

## E. EXAMPLES (continued)

Example #7: (Negative target analyte blank result; One positive sample result < negative blank action level, one non-detect sample result)

A preparation blank had a reported negative arsenic result of -7 ug/L, less than the negative arsenic MDL of 3 ug/L and the lowest negative arsenic value among all of the associated blanks. Aqueous sample MA2CB6 was reported with a positive arsenic result of 18 ug/L and aqueous sample MA2CB7 was reported as a non-detect at 10 U ug/L for arsenic. The negative blank action level for arsenic was calculated as 35 ug/L (5x |-7 ug/L|).

	<u>5x Rule</u>	Arsenic <u>ug/L</u>
MDL		3
Preparation Blank Result		-7
CRQL		10
Sample MA2CB6 Result		18
Sample MA2CB7 Result		10 U
Negative Blank Action Level		35 (5x  -7 )
Qualified Sample MA2CB6 Result		18 J
Qualified Sample MA2CB7 Result		10 UJ

The positive arsenic result for sample MA2CB6 is less than the negative blank action level of 35 ug/L and is, therefore, reported as an estimated value of 18 J ug/L on the Data Summary Table. The non-detect arsenic result for sample MA2CB7 is reported as estimated at the sample quantitation limit of 10 UJ ug/L on the Data Summary Table. The validator notes all actions taken in the Data Validation Memorandum and discusses the possible low bias in the results.

## E. EXAMPLES (continued)

Example #8: (Different blank actions for an aqueous equipment blank and a soil sample)

An SDG consisted of soil samples and an aqueous rinsate blank. The aqueous rinsate blank was prepared in a different batch from the soil samples. In the soil batch, the highest blank concentration for copper was 1.8 mg/kg in the preparation blank, and the copper result for soil sample MA1D7F was reported as 3.6 mg/kg. In the water batch, the highest blank concentration for copper was 14 ug/L in the aqueous preparation blank. The aqueous rinsate blank RB-04 was reported as 8 ug/L. The results and qualifications are summarized below.

<u>Soil Sample</u>		<u>5x Rule</u>	<u>Aqueous Rinsate Blank</u>	
	Copper <u>mg/kg</u>			Copper <u>ug/L</u>
Preparation Blank Result	1.8		Preparation Blank Result	14
Sample MA1DF7 Result	3.6		Rinsate Blank RB-04 Result	8
CRQL	2.5		CRQL	25
Blank Action Level (Soil)	9.0 (5x1.8)		Blank Action Level (Aq.)	70 (5x14)
Qualified MA1DF7 Result	3.6 U		Qualified RB-04 Result	25 U

The copper result in soil sample MA1D7F is reported on the Data Summary Table as a non-detect at 3.6 U mg/kg since it is less than the Blank Action Level (based on the associated soil preparation blank) and greater than the sample quantitation limit. The copper result in aqueous rinsate blank RB-04 is reported on the Data Summary Table as a non-detect at the quantitation limit of 25 U ug/L since it is less than the Blank Action Level (based on the associated aqueous preparation blank) and less than the quantitation limit. The validator notes all actions taken in the Data Validation Memorandum and discusses the copper contamination in the rinsate blank being attributed to contamination introduced in the laboratory.

E. EXAMPLES (continued)

Example #9: (Two equipment blanks with target analyte contaminants, each associated with different samples)

For a sample delivery group, the traffic report and field sampling notes indicate that eight aqueous samples were collected on sampling day three along with associated equipment blank EB-T03, and six aqueous samples were collected on sampling day four along with associated equipment blank EB-T04. The two equipment blanks contained the highest levels of zinc contamination for this SDG, 110 ug/L zinc in EB-T03 and 280 ug/L in EB-T04. Zinc was detected in two samples, MA14G7 (day three) and MA11A4 (day four).

<u>Sample Associated with EB-T03</u>		<u>Sample Associated with EB-T04</u>	
	<u>Zinc</u> <u>ug/L</u>		<u>Zinc</u> <u>ug/L</u>
Equipment Blank EB-T03 Result	110	Equipment Blank EB-T04 Result	280
Sample MA14G7 Result	720	Sample MA11A4 Result	1270
CRQL	60	CRQL	60
Blank Action Level	550 (5x110)	Blank Action Level	1400 (5x280)
Qualified MA14G7 Result	720	Qualified MA11A4 Result	1270 U

- Equipment blank EB-T03 (Blank Action Level = 550 ug/L) is associated with sample MA14G7. The validator reports the zinc result for sample MA14G7 as unqualified on the Data Summary Table since the result is greater than the blank action level and greater than the quantitation limit.
- Equipment blank EB-T04 (Blank Action Level = 1400 ug/L) is associated with sample MA11A4. The validator reports the zinc result for sample MA11A4 as a non-detect at the reported sample concentration on the Data Summary Table since the result is less than the blank action level and greater than the sample quantitation limit.

The validator notes all actions taken in the Data Validation Memorandum.

## E. EXAMPLES (continued)

Example #10: (Negative and positive blank results for the same analyte in the same sample)

Barium was detected in the preparation blank at 4 ug/L. Barium was also reported at a negative concentration of -3 ug/L in the initial calibration blank (ICB), less than the negative MDL for barium. Both of these blanks were associated with samples MANN11 and MASS22.

	<u>5x Rule</u>	<u>ug/L</u>
Preparation Blank Result		4
Pos. Blank Action Level		20 (5x4)
ICB Result		-3
Neg. Blank Action Level		15 (5x   -3   )
CRQL		10
Barium Sample MANN11 Result		12
Qualified Barium Sample MANN11 Result		12 UJ
Barium Sample MASS22 Result		9 J
Qualified Barium Sample MASS22 Result		10 UJ

- Sample MANN11: The barium sample quantitation limit is elevated to the sample concentration due to positive blank actions (12 U); and the sample quantitation limit is then estimated (UJ) due to negative blank actions. Therefore, professional judgment is used to report barium in sample MANN11 as an estimated non-detect, 12 UJ, on the Data Summary Table.
- Sample MASS22: Barium in sample MASS22, which was reported at less than the sample quantitation limit, is reported at the sample quantitation limit due to positive blank actions (10 U). The sample quantitation limit is then estimated (UJ) for negative blank actions. Therefore, professional judgment is used to report barium in sample MASS22 as estimated at the sample quantitation limit, 10 UJ, on the Data Summary Table.