

Region III Modification

METALS

**REGION III MODIFICATIONS**  
to the  
**LABORATORY DATA VALIDATION FUNCTIONAL GUIDELINES**  
for  
**EVALUATING INORGANICS ANALYSES**

April 1993

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U. S. Environmental Protection Agency  
Environmental Science Center  
701 Mapes Road  
Ft. Meade, MD 20755-5350

## INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation. In some aspects, it is equivalent to a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. These Guidelines have been updated to include all requirements in the ILM03.0 Statement of Work (SOW) for Inorganics.

The review of laboratory data using these guidelines is considered equivalent to EPA Region III's "IM2" level of review.

Those areas where specific SOPs are possible are primarily areas in which definitive performance requirements are established. These requirements are concerned with specifications that are not sample dependent; they specify performance requirements on matters that should be fully under a laboratory's control. These specific areas include blanks, calibration standards, calibration verification standards, laboratory control standards, and interference check standards. In particular, mistakes such as calculation and transcription errors must be rectified by resubmission of corrected data sheets by the laboratory.

This document is intended for technical review. Some areas of overlap between technical review and Contract Compliance Screening (CCS) exist; however, determining contract compliance is not intended to be a goal of these guidelines. It is assumed that the CCS is available and can be utilized to assist in the data review procedure.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does not constitute either a new requirement standard or full acceptance of the data. Any decision to utilize data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which are out of specification may be required to rerun or resubmit data even if the previously submitted data have been utilized due to urgent program needs; data which do not meet specified requirements are never fully acceptable. The only exception to this requirement is in the area of requirements for individual sample analysis; if the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid and legally defensible.

All data reviews must have, as a cover sheet, the Inorganic Regional Data Assessment (IRDA) form (CLP data only). (A copy is attached at the end of this document.) If mandatory actions are required, they should be specifically noted on this form. In addition, this form is to be used to summarize overall deficiencies requiring attention, as well as general laboratory performance and any discernable trends in the quality of the data. (This form is not a replacement for the data review.) Sufficient supplementary documentation must accompany the form to clearly identify the problems associated with a Case. The form and any attachments must be submitted to the Regional Technical Project Officer (TPO).

It is the responsibility of the data reviewer to notify the Regional TPO concerning problems and shortcomings with regard to laboratory data. If there is an urgent requirement, the TPO may be contacted by telephone to expedite corrective action. It is recommended that all items for TPO action be presented at one time. In any case, the Inorganic Regional Data Assessment form must be completed and submitted.

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PRELIMINARY REVIEW

In order to use this document effectively, the reviewer should have a general overview of the Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in their analysis are essential information. Background information on the site is helpful but often this information is very difficult to locate. The site project officer is the best source for answers or further direction.

CCS is a source of a large quantity of summarized information. It can be used to alert the reviewer of problems in the Case or what may be sample-specific problems. This information may be utilized in data validation. If CCS is unavailable, those criteria affecting data validity must be addressed by the data reviewer.

Cases routinely have unique samples which require special attention by the reviewer. Field blanks, field duplicates, and performance audit samples need to be identified. The sampling records should provide:

1. Project Officer for site
2. Complete list of samples with notations on:
  - a) sample matrix
  - b) blanks\*
  - c) field duplicates\*
  - d) field spikes\*
  - e) QC audit sample\*
  - f) shipping dates
  - g) labs involved

\* If applicable.

The chain-of-custody record includes sample descriptions and date of sampling. Although sampling date is not addressed by contract requirements, the reviewer must take into account lag time between sampling and shipping while assessing sample holding times.

**INORGANICS PROCEDURE**

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

- I. Holding Times (CCS - Lab holding times only)
- II. Calibration
  - Initial (CCS)
  - Initial and Continuing Calibration Verification (CCS)
- III. Blanks (CCS)
- IV. ICP Interference Check Sample (CCS)
- V. Laboratory Control Samples (CCS)
- VI. Duplicate Sample (CCS)
- VII. Matrix Spike Sample (CCS)
- VIII. Furnace Atomic Absorption QC (CCS)
- IX. ICP Serial Dilution (CCS)
- X. Sample Result Verification (CCS - 10%)
- XI. Field Duplicates
- XII. Overall Assessment of Data for a Case

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I. HOLDING TIMES

A. Objective

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

Note: The holding time is based on the date of collection, rather than verified time of sample receipt, and date of digestion/distillation. It is a technical evaluation rather than a contractual requirement.

B. Criteria

Technical requirements for sample holding times have only been established for water matrices. The following holding time and preservation requirements were established under 40 CFR 136 (Clean Water Act) and are found in Volume 49, Number 209 of the Federal Register, page 43260, issued on October 26, 1984.

METALS: 6 months; preserved to pH < 2

MERCURY: 28 days; preserved to pH < 2

CYANIDE: 14 days; preserved to pH > 12

C. Evaluation Procedure

Actual holding times are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of analysis found in the laboratory raw data (Digestion logs and instrument run logs). Examine the digestion and/or distillation logs to determine if samples were preserved at the proper pH.

D. Action

1. If 40 CFR 136 criteria for holding times and/or preservation are not met, qualify all results >IDL as biased low (L) and results <IDL as biased low (UL).
2. If holding times are exceeded, the reviewer must use professional judgment to determine the reliability of the data and the effects of additional storage on the same results. The expected bias would be low and the reviewer may determine that results < IDL are unusable (R).
3. It is further required that metals and cyanide in properly preserved non-aqueous samples be analyzed from the time of sample collection within the holding times as specified in 40CFR136 (see above). The narrative should state that due to the limited information concerning holding times for soil samples, the water sample holding times were applied for soil samples.

## II. CALIBRATION

### A. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

### B. Criteria

#### 1. Initial Calibration

Instruments must be calibrated daily and each time the instrument is set up.

##### a. ICP Analysis

- 1) A blank and at least one standard must be used in establishing the analytical curve.

##### b. Atomic Absorption Analysis (AA)

- 1) A blank and at least three standards, one of which must be at the Contract Required Detection Limit (CRDL), must be used in establishing the analytical curve.
- 2) The correlation coefficient must be  $\geq 0.995$ .

Note: The correlation coefficient of 0.995 is a technical criterion and not contractual.

##### c. Mercury Analysis

- 1) A blank and at least four standards must be used in establishing the analytical curve.
- 2) The correlation coefficient must be  $\geq 0.995$ .

##### d. Cyanide Analysis

- 1) A blank and at least three standards must be used in establishing the analytical curve.
- 2) A midrange standard must be distilled.
- 3) A correlation coefficient  $\geq 0.995$  is required for photometric determination.

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#### Calibration

2. Initial and Continuing Calibration Verification (ICV and CCV)
  - a. Analysis results must fall within the control limits of 90 - 110 percent Recovery (%R) of the true value for all analytes except mercury and cyanide.
  - b. Analysis results for mercury must fall within the control limits of 80 - 120%R.
  - c. Analysis results for cyanide must fall within the control limits of 85 - 115%R.

#### C. Evaluation Procedure

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank.
2. Verify that the correlation coefficient is  $\geq 0.995$ .
3. Check the distillation log and verify that the mid range CN standard was distilled.
4. Recalculate one or more of the ICV and CCV %R per type of analysis (ICP, GFAA, etc.) using the following equation and verify that the recalculated value agrees with the laboratory reported values on Form IIA. Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows (e.g., 89-111%).

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

Where,

Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

True = concentration (in ug/L) of each analyte in the ICV or CCV source

5. Check the CRQL (CRI and CRA) standard and verify % Recovery falls within 90-110%.

#### D. Action

1. If the minimum number of standards as defined in section B were not used for initial calibration, the reviewer should use professional judgement to qualify the data. However, if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R). In each case make note in the TPO section.

## Calibration

2. If the correlation coefficient is  $<0.995$ , qualify results  $> IDL$  as estimated (J), and results  $< IDL$  as estimated (UJ).

Note: For critical samples further evaluation of the calibration curve may be warranted to determine if qualification is necessary.

3. If the midrange CN standard was not distilled, qualify all associated results as estimated (J).
4. If the ICV or CCV %R falls outside the acceptance windows, the following table is to be used.

Qualifier		Percent Recovery (%R)		
$> IDL$	$< IDL$	General	CN	Hg
L	UL	75-89	70-84	65-79
K	None	$> 110$	$> 115$	$> 120$
L	R	$< 75$	$< 70$	$< 65$

5. If the CRDL standard %R falls outside the acceptance windows ( $\pm 10\%$ ), (except Hg and CN) use the following guidelines to qualify the data:
- 5a. If the CRDL recovery for an element is  $> 110\%$  and the reported sample results are  $> IDL$  but  $< 2X$  CRDL qualify the affected data as biased high (K).
- 5b. If the CRDL recovery for an element falls between 50% and 89%, qualify the affected data  $> IDL$  but  $< 2X$  CRDL as biased low (L), and the affected data  $< IDL$  as biased low (UL).
- 5c. If the CRDL recovery for an element is  $< 50\%$ , qualify the affected data  $> IDL$  but  $< 2X$  CRDL as biased extremely low (L), and the results  $< IDL$  as unusable (R).

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III. BLANKS

A. Objective

The assessment of blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples. If problems with any blank exist, all data associated with the Case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the Case, or if the problem is an isolated occurrence not affecting other data.

B. Criteria

No contaminants should be in the blank(s).

C. Evaluation Procedures

Review the results reported on the Blank Summary (Form III) as well as the raw data (ICP printouts, strip charts, printer tapes, bench sheets, etc.) for all blanks and verify that the results were accurately reported.

D. Action

1. Results >IDL but <5 times the amount in any blank should be qualified as (B).
2. If any blank has a negative result whose absolute value is >CRDL then all samples reported below 5X CRDL are qualified as biased low (L) and any non-detects (results <IDL) associated with this blank are qualified biased low (UL).
3. Any blank with a value below the negative IDL must be carefully evaluated to determine its effect on sample data.
4. When evaluating blanks, the reviewer must consider the source. Blanks can be categorized into blanks generated in the field (equipment rinsates, bottle blanks, field blanks) and blanks generated in the laboratory (preparation blanks, method blanks). The criteria for evaluation of laboratory generated blanks apply to any blank associated with the samples. If the highest level of contamination is found in a field blank, all affected samples (i.e., per SDG or sampling event) should be qualified appropriately. The following are examples of applying the blank qualification guidelines.

Note: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results reported on Form I will not be on the same basis (units, dilution) as the calibration blank data reported on Form III. The reviewer may find it easier to work from the raw data when applying 5X criteria to soil sample data/calibration blank data.

## Blanks

5. The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines. Any deviations must be clearly stated in the data review narrative.

**Example 1:** Sample result is greater than the Instrument Detection Limit (IDL), but is less than 5X the initial or continuing calibration (ICB or CCB) blank result.<sup>1</sup>

## Aqueous:

Iron CCB Result:	50 ug/L
Iron IDL:	15 ug/L
Iron Sample Result:	100 ug/L
Reported Sample Result:	100 B ug/L

## Soil:

Iron CCB Result:	50 ug/L (10mg/Kg)
Iron IDL:	3 mg/Kg (15ug/L)
Iron Sample Result:	55 mg/Kg
Sample % Solids:	85%
5X CCB Result:	59 mg/Kg
Reported Sample Result:	55 B mg/Kg

<sup>1</sup>Please note that qualification of sample results due to ICB or CCB contamination affects only those samples analyzed immediately after or before the ICB or CCB containing the contamination. If both CCBs bracketing samples are contaminated, qualification should be based on the higher of the two CCB values.

**Example 2:** Sample result is greater than the Instrument Detection Limit (IDL) but is less than 5X the equipment rinsate blank result.<sup>2</sup>

## Aqueous:

Sodium Prep Blank Result:	22 ug/L
Sodium Equipment Blank Result <sup>2</sup> :	26 ug/L
Sodium CCB Result:	19 ug/L
Sodium IDL:	15 ug/L
Sodium Sample Result	60 ug/L
Reported Sample Result <sup>2</sup> :	60 B ug/L

## Soil:

Sodium Prep Blank Result:	8 mg/Kg
Sodium Equipment Blank Result <sup>2</sup> :	45 ug/L
Sodium CCB Result:	30 ug/L
Sodium IDL:	15 ug/L
Sample Result :	52 mg/Kg (Dry weight)
Sample % Solids:	80.0%
5X Equipment Blank Result:	56 mg/Kg
Reported Sample Result <sup>2</sup> :	52 B mg/Kg

<sup>2</sup>Please note that qualification of the sample result is based on the highest level of contamination found in the associated blanks.

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#### Blanks

6. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

**IV. ICP INTERFERENCE CHECK SAMPLE (ICS)****A. Objective**

The ICP Interference Check Sample verifies the contract laboratory's interelement and background correction factors.

**B. Criteria**

1. An ICS must be run at the beginning and end of each sample analysis run (or a minimum of twice per 8 hour working shift, whichever is more frequent).
2. Results for the ICS solution AB analysis must fall within the control limits of  $\pm 20\%$  of the true value.

**C. Evaluation Procedure**

1. Recalculate from the raw data (ICP printout) one or more of the recoveries using the following equation (%R) and verify that the recalculated value agrees with the laboratory reported values on Form IV.

$$\text{ICS \%R} = \frac{\text{Found Solution AB}}{\text{True Solution AB}} \times 100$$

Where,

Found Solution AB = concentration (in ug/L) of each analyte measured in the analysis of solution AB

True Solution AB = concentration (in ug/L) of each analyte in solution AB

2. Check ICS raw data for results with an absolute value > IDL for those analytes which are not present in the ICS solution.

**D. Action**

1. For samples with concentrations of Al, Ca, Fe, and Mg which are comparable to or greater than their respective levels in the Interference Check Sample:
  - a. If the ICS recovery for an element is >120% and the sample results are <IDL, this data is acceptable for use.
  - b. If the ICS recovery for an element is >120% and the reported sample results are >IDL, qualify the affected data as biased high (K).

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#### ICP Interference Check Sample

- c. If the ICS recovery for an element falls between 50 and 79% and the sample results are >IDL, qualify the data as biased low (L).
  - d. If the results are <IDL and the ICS recovery for that analyte falls within the range of 50-79%, the possibility of false negatives may exist. Qualify the data for these samples as biased low (UL).
  - e. If the ICS recovery results for an element is <50%, qualify the affected data >IDL as biased low (L). If the ICS recovery results for an element fall <50%, qualify results <IDL as unusable (R).
2. If results >IDL are observed for elements which are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS (false positives) qualify sample results >IDL as biased high (K).
3. If negative results are observed for elements that are not present in the EPA ICS solution, and their absolute value is >IDL, the possibility of false negatives in the samples may exist. If the absolute value of the negative results is >IDL, an evaluation of the associated sample data should be made. For samples with comparable or higher levels of interferents, qualify all results <IDL for the affected analytes as biased low (UL) and sample results >IDL as biased low (L).
4. In general, the sample data can be accepted if the concentrations of Al, Ca, Fe and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. If these elements are present at concentrations greater than the level in the ICS, or other elements are present in the sample at >10 mg/L, the reviewer should investigate the possibility of other interference effects by using Table 2 given on page D-26 of the ILM03.0 SOW. These analyte concentration equivalents presented in the Table should be considered only as estimated values, since the exact value of any analytical system is instrument specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is >2X CRDL and also greater than 10% of the reported concentration of the affected element, qualify the affected results as biased high (K).

**V. LABORATORY CONTROL SAMPLE (LCS)****A. Objective**

The laboratory control sample serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation.

**B. Criteria**

1. All aqueous LCS recoveries must fall within the control limits of 80-120%, except Ag and Sb which have no control limits.
2. All solid LCS results must fall within the control limits established by the EPA. This information is available from EMSL/LV.

**C. Evaluation Procedure**

1. Review Form VII and verify that results fall within the control limits.
2. Check the raw data (ICP printout, strip charts, beach sheets) to verify the reported recoveries on Form VII. Recalculate one or more of the recoveries (%R) using the following equation:

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

Where,

LCS Found = Concentration (in ug/L for aqueous, mg/kg for solid) of each analyte measured in the analysis of LCS solution

LCS True = Concentration (in ug/L for aqueous, mg/kg for solid) of each analyte in the LCS source

**D. Action****1. Aqueous LCS**

- a. If the LCS recovery for any analyte falls within the range of 50-79%, qualify results >IDL as biased low (L). If the LCS recovery is >120% qualify results >IDL as biased high (K).
- b. If results are <IDL and the LCS recovery is greater than 120%, the data are acceptable.

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#### Laboratory Control Sample (LCS)

- c. If results are <IDL and the LCS recovery falls within the range of 50-79%, qualify the data for the affected analytes as biased low (UL).
- d. If the LCS recovery is <50%, qualify all positive sample results as biased low (L), and qualify all non-detects as unusable (R).

#### 2. Solid LCS

- a. If the solid LCS recovery for an analyte falls less than the lower limit of the EPA control limits, qualify all results >IDL as biased low (L). If the solid LCS recovery is greater than the upper limit of the EPA control limit, qualify all results >IDL as biased high (K).
- b. If the LCS results are higher than the control limits and the sample results are <IDL, the data are acceptable.
- c. If the solid LCS results are lower than the control limits, qualify all results <IDL as biased low (UL).

**VI. DUPLICATE SAMPLE ANALYSIS****A. Objective**

Duplicate analyses are indicators of laboratory precision based on each sample matrix.

**B. Criteria**

1. Samples identified as field blanks cannot be used for duplicate sample analysis.
2. A control limit of  $\pm 20\%$  (35% for soil) for the Relative Percent Difference (RPD) shall be used for duplicate sample values  $> 5X$  CRDL.
3. A control limit of  $\pm$  CRDL ( $\pm 2X$  CRDL for soil) shall be used for duplicate sample values  $< 5X$  CRDL, including the case when only one of the duplicate sample values is  $< 5X$  CRDL.

**C. Evaluation Procedure**

1. Review Form VI and verify that results fall within the control limits.
2. Check the raw data and recalculate one or more RPD using the following equation to verify that results have been correctly reported on Form VI.

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

Where,

S = First Sample Value (original)

D = Second Sample Value (duplicate)

3. Verify that the field blank was not used for duplicate analysis.

**D. Action**

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same matrix as estimated (J).
2. If the field blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgment exercised when evaluating the data.

Note: This information must be included on the IRDA form.

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**VII. MATRIX SPIKE SAMPLE ANALYSIS**

**A. Objective**

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology.

**B. Criteria**

1. Samples identified as field blanks cannot be used for spiked sample analysis.
2. Spike recovery (%R) must be within the limits of 75-125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

**C. Evaluation Procedure**

1. Review Form V and verify that the results fall within the specified limits.
2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported on Form V.

$$\%R = \frac{(SSR-SR)}{SA} \times 100$$

Where,

SSR = Spiked Sample Result  
SR = Sample Result  
SA = Spike Added

3. Verify that the field blank was not used for spike analysis.

**D. Action**

1. If the spike recovery is >125% and the reported sample results are <IDL, the data is acceptable for use.
2. If the spike recovery is >125% and the results are >IDL, qualify the data as biased high (K).  
If the recovery is <75%, qualify the results >IDL as biased low (L).
3. If the spike recovery falls within the range of 30-74% and the sample results are <IDL, qualify the data as biased low (UL).
4. If spike recovery results fall <30% and the sample results are <IDL, qualify the data for these samples as unusable (R). If sample results are >IDL, qualify the data as biased extremely low (L).

**Matrix Spike Sample**

5. If the field blank was used for matrix spike analysis, all other QC data must be carefully checked and professional judgment exercised when evaluating the data.

Note: This information must be included on the IRDA form.

Note: If the matrix spike recovery does not meet criteria (except in Ag), a post digestion spike is required for all methods except furnace, but this data is not used to qualify sample results. However, this information must be included in the IRDA report. For flame AA, ICP and CN analyses, when the pre-digestion/pre-distillation spike recovery falls outside the control limits and the sample result does not exceed 4X the spike added, a post-digestion/post-distillation spike must be performed for those elements that do not meet the specified criteria (exception: Ag). The unspiked aliquot of the sample should be spiked at 2X the indigenous level or 2X CRDL, whichever is greater. If post-digestion/post-distillation spike %R falls outside the control limits make a note in the TPO section.

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**VIII. FURNACE ATOMIC ABSORPTION QC**

**A. Objective**

Duplicate injection and furnace post digestion spikes establish the precision and accuracy of the individual analytical determinations.

**B. Criteria**

1. For sample concentrations > CRDL, duplicate injections must agree within  $\pm 20\%$  Relative Standard Deviation (RSD), (or Coefficient of Variation (CV)), otherwise the sample must be rerun once (at least two additional injections).
2. Spike recovery must be  $\geq 85\%$  and  $\leq 115\%$ .
3. The Furnace Atomic Absorption Scheme must be followed as described in the ILM03.0 SOW.

**C. Evaluation Procedure**

1. Check raw data to verify that duplicate injections agree within  $\pm 20\%$  RSD (or CV) for sample concentrations > CRDL.
2. Review Furnace AA raw data to verify that the Furnace Atomic Absorption Scheme has been followed.

**D. Action**

1. If duplicate injections are outside the  $\pm 20\%$  RSD (or CV) limits and the sample has not been rerun once as required, qualify the results >IDL as estimated (J) and results <IDL as estimated (UJ).
2. If the rerun sample results do not agree within  $\pm 20\%$  RSD (or CV), qualify the results >IDL as estimated (J) and results <IDL as estimated (UJ).
3. If the analytical/post digestion spike recovery is <40%, qualify results >IDL as biased low (L).
4. If the analytical/post digestion spike recovery is  $\geq 10\%$  but <40%, qualify results <IDL as biased low UL.
5. If the furnace analytical/post digestion spike recovery is <10%, qualify all results >IDL as biased low (L) and results <IDL as unusable (R).

Furnace Atomic Absorption QC

6. When sample absorbance is  $< 50\%$  of the post digestion spike absorbance then:
  - a. If the furnace analytical/post digestion spike recovery is  $< 85\%$ , qualify results  $> IDL$  as biased low (L). If the recovery is  $> 115\%$ , qualify the results  $> IDL$  as biased high (K).
  - b. If the furnace analytical/post digestion spike recovery is  $< 85\%$ , qualify the results  $< IDL$  as biased low (UL).
7. If Method of Standard Additions (MSA) is required but has not been done, qualify results  $> IDL$  as estimated (J), and results  $< IDL$  estimated (UJ).
8. If any of the samples run by MSA have not been spiked at the appropriate levels, qualify results  $> IDL$  as estimated (J) and results  $< IDL$  as estimated (UJ).
9. If the MSA correlation coefficient is  $< 0.995$ , qualify results  $> IDL$  as estimated (J) and results  $< IDL$  as estimated (U).

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**IX. ICP SERIAL DILUTION**

**A. Objective**

The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix.

**B. Criteria**

If the analyte concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL), an analysis of a 5-fold dilution must agree within 10% Difference (%D) of the original results.

**C. Evaluation Procedures**

1. Check the raw data and recalculate the %D using the following equation to verify that the dilution analysis results agree with results reported on Form IX.

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

Where,

I = Initial Sample Result

S = Serial Dilution Result (Instrument Reading x 5)

2. Check the raw data for evidence of negative interference, i.e., results of the dilution sample are significantly higher than the original sample.

**D. Action**

1. When criteria are not met, qualify the results as estimated (J).
2. If evidence of negative interference is found, use professional judgment to qualify the data.

## **X. SAMPLE RESULT VERIFICATION**

### **A. Objective**

The objective is to ensure that the reported quantitation results are accurate.

### **B. Criteria**

Analyte quantitation must be calculated according to the appropriate SOW.

### **C. Evaluation Procedures**

The raw data should be examined to verify the correct calculation of sample results reported by the laboratory. Digestion and distillation logs, instrument printouts, strip charts, etc., should be compared to the reported sample results.

1. Examine the raw data for any anomalies (i.e., baseline shifts, negative absorbances, omissions, legibility, etc.).
2. Verify that there are no transcription or reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.
3. Verify that results fall within the linear range of the ICP (Form XIII) and within the calibrated range for the non-ICP parameters.
4. Verify that sample results are  $> 5X$  ICP IDL, if ICP analysis results are used for As, Tl, Se, or Pb.

Note: When the laboratory provides both ICP and furnace results for an analyte in a sample and the concentration is  $> \text{ICP IDL}$ , the results can assist in identifying quantitation problems.

### **D. Action**

If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer may determine qualification of the data is warranted.

METALS

**XI. FIELD DUPLICATES**

**A. Objective**

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than lab duplicates which measure only lab performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field samples.

**B. Criteria**

There are no review criteria for field duplicate analyses comparability.

**C. Evaluation Procedures**

Samples which are field duplicates should be identified using EPA Sample Traffic Reports or sample field sheets. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference (RPD), if appropriate.

**D. Action**

Any evaluation of the field duplicates should be provided with the reviewer's comments.

**XII. OVERALL ASSESSMENT OF DATA FOR A CASE**

It is appropriate for the data reviewer to make professional judgments and express concerns and comments on the validity of the overall data for a Case. This is particularly appropriate when there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations in order to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the data quality objectives were provided. The cover form and supplementary documentation must be included with the review.

## GLOSSARY A

### Data Qualifier Definitions

For the purposes of this document the following code letters and associated definitions are provided.

- U - The analyte was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
- J - The associated value is an estimated quantity.
- R - The data are unusable. (Note: The analyte may or may not be present.)
- UJ - The analyte was analyzed for, but was not detected. The associated detection limit is an estimate and may be inaccurate or imprecise.
- K - The analyte is present. The reported value may be biased high. The actual value is expected to be lower than reported.
- L - The analyte is present. The reported values may be biased low. The actual value is expected to be higher than reported.
- UL - The analyte was not detected, and the reported quantitation limit is probably higher than reported.

**GLOSSARY B**  
**Additional Terms**

Associated Samples	Any sample related to a particular QC analysis. For example: - For ICV, all samples run under the same calibration curve. - For duplicate RPD, all SDG samples digested/distilled of the same matrix.
AA	Atomic Absorption
Calibration Curve	A plot of absorbance versus concentration of standards.
Case	A finite, usually predetermined number of samples collected in a given time period for a particular site. A Case consists of one or more Sample Delivery Groups.
CCB	Continuing Calibration Blank - a deionized water sample run every ten samples designed to detect any carryover contamination.
CCS	Contract Compliance Screening - process in which SMO inspects analytical data for contractual compliance and provides EMSL/LV, laboratories and the Regions with their findings.
CCV	Continuing Calibration Verification - a standard run every ten samples designed to test instrument performance.
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limit
CV	Coefficient of Variation
EMSL/LV	Environmental Monitoring System Laboratory/Las Vegas (P.O. Box 15027, Las Vegas, Nevada 89114)
Field Blank	Field blanks are intended to identify contaminants that may have been introduced in the field. Examples are travel blanks, rinsate blanks, and decontamination blanks.
Field Duplicate	A duplicate sample generated in the field, not in the laboratory.
Holding Time	The time from sample collection to laboratory analysis.
ICB	Initial Calibration Blank - first blank standard run to confirm the calibration curve.
ICP	Inductively Coupled Plasma

## Region III Modifications

### METALS

#### Glossary B

ICS

Interference Check Sample

ICV

Initial Calibration Verification - first standard run to confirm the calibration curve.

Initial Calibration

The establishment of a calibration curve with the appropriate number of standards and concentration range. The calibration curve plots absorbance or emission versus concentration of standards.

IRDA

Inorganic Regional Data Assessment

LCS

Laboratory Control Sample - supplied by EPA

MS

Matrix Spike - introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology.

MSA

Method of Standard Addition

Post digestion Spike

The addition of a known amount of standard after digestion. (Also identified as analytical spike, or spike, for furnace analyses.)

QAC

Quality Assurance Coordinator

RPD

Relative Percent Difference

RSCC

Regional Sample Control Center

RSD

Relative Standard Deviation

Serial Dilution

A sample run at a specific dilution to determine whether any significant chemical or physical interferences exist due to sample matrix effects. (ICP only)

SDG

Sample Delivery Group - defined by one of the following, whichever occurs first:

- case of field samples
- each twenty field samples in a Case
- Each 14-day calendar period during which field samples in a Case are received, beginning with receipt of the first sample in the SDG.

SMO

Sample Management Office

SOP

Standard Operating Procedure

SOW

Statement of Work

