

## Appendix K

### Inorganic Data Validation Worksheets

Note: Refer to Attachment J of Part I, Data Validation Manual: The Data Quality System, for the generic forms for the Data Validation Report (DQO Summary Form, Communication Form, Chain-of-Custody Form/Traffic Report).

**REGION I, EPA-NE INORGANIC REGIONAL DATA ASSESSMENT (IRDA)\***

CASE #: \_\_\_\_\_  
 LAB NAME: \_\_\_\_\_  
 SDG #: \_\_\_\_\_  
 SOW #/CONTRACT #: \_\_\_\_\_  
 EPA-NE DV TIER LEVEL: \_\_\_\_\_  
 PO: \*\*ACTION \_\_\_\_\_ FYI \_\_\_\_\_

SITE NAME: \_\_\_\_\_  
 # OF SAMPLES/MATRIX: \_\_\_\_\_  
 VALIDATION CONTRACTOR: \_\_\_\_\_  
 VALIDATOR'S NAME: \_\_\_\_\_  
 DATE DP REC'D BY EPA-NE: \_\_\_\_\_  
 DV COMPLETION DATE: \_\_\_\_\_

**ANALYTICAL DATA QUALITY SUMMARY**

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

- o = Data had no problems or were qualified due to minor contractual problems.
- m = Data were qualified due to major contractual problems.
- z = Data were rejected as unusable due to major contractual problems.

ACTION ITEMS: (z items) \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

AREAS OF CONCERN: (m items) \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

COMMENTS: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

\* This form assesses the analytical data quality in terms of contractual compliance only. It does not assess sampling errors and/or non-contractual analytical issues that affect data quality.

\*\* Check "ACTION" only if contractual defects resulted in reduced payment/data rejection recommendations.

Validator: \_\_\_\_\_

Date: \_\_\_\_\_

INSTRUCTIONS ON REVERSE SIDE

**DRAFT 11/08**

## GUIDANCE FOR COMPLETING THE IRDA

The IRDA form provides the laboratory's CLP PO and other contract management personnel with an overview of the contractual analysis and reporting deficiencies found in an analytical data package and identifies those contractual deficiencies that resulted in reduced payment/data rejection recommendations/actions. The IRDA form is used to summarize analytical data quality only in terms of contractual compliance. Sampling errors and non-contractual analytical errors that affect data quality are not summarized on this form, but rather are documented in the Tier I Validation Cover Letter and Tier II/III Data Validation Reports. For instance, if the sampler did not ship the samples until after the holding time had expired, a notation would not be made on the IRDA form since the laboratory is not responsible for the sampler's actions.

The IRDA form should be completed as follows:

1. Fill in all the header information (with the exception of the PO Action/FYI field): Case Number, Site Name, Laboratory Name, number and matrix of samples in the data package, SDG number, Validation Contractor, SOW#/Contract#, Data Validator's Name, EPA-NE Data Validation Tier Level (i.e., I, II, III or partial II/III), Date the Data Package was received by EPA-NE, and the Data Validation Completion Date.
2. Summarize the contractual problems discovered during data validation by fraction and by evaluation criteria in the "Analytical Data Summary" table, and in the "Action Items" and "Areas of Concern" sections as described in items 3 through 6 below. Use the Data Validation Memoranda as a guide when completing the IRDA form.
3. The following qualifiers must be utilized to document contractual problems on the IRDA forms.

o	=	Data had no problems or were qualified due to minor contractual problems
m	=	Data were qualified due to major contractual problems
z	=	Data were rejected as unusable due to major contractual problems
4. If the data were acceptable, or were qualified due to minor contractual problems, enter the qualifier "o" into the appropriate column (fraction) and row (evaluation criteria). No further documentation is necessary on the IRDA form. An example of a minor problem would be an inorganic analyte that slightly exceeded the SOW-specified %R continuing calibration criterion.
5. If the data were qualified due to major contractual problems, enter the qualifier "m" into the appropriate column (fraction) and row (evaluation criteria). Use different superscripts ( $m^1$ ,  $m^2$ , etc.) for each major contractual problem identified and provide a brief description of each major problem in the "Areas of Concern" section. An example of a major contractual problem resulting in data qualification would be an ICP-MS internal standard that had an extremely low % relative intensity (below the lower limit of the SOW-specified acceptance criteria) and reanalysis was not performed.
6. If the data were rejected as unusable due to major contractual problems, enter the qualifier "z" in the appropriate column (method/parameter) and row (evaluation criteria). Use a different superscript ( $z^1$ ,  $z^2$ , etc.) for each major contractual problem identified and provide a brief description of each major problem in the "Action Items" section. An example of a major contractual problem resulting in data rejection would be contractual holding time criteria that were exceeded for cyanide.
7. Complete the PO Action/FYI field using the information contained in the "Action Items" and "Areas of Concern" sections. PO Action should be indicated with a check mark (✓) in the space following "Action" only if the contractual defects resulted in reduced payment or data rejection. If no PO Actions are indicated, then a check mark (✓) should be placed in the space following "FYI".
8. The validator who completed the IRDA form must sign his/her name in the "Validator" field and enter the IRDA completion date in the "Date" field.

REGION I INORGANIC DATA VALIDATION

The following data package has been validated:

Lab Name \_\_\_\_\_ SOW/Method No. \_\_\_\_\_  
Case/Project No. \_\_\_\_\_ Sampling Date(s) \_\_\_\_\_  
SDG No. \_\_\_\_\_ Shipping Date(s) \_\_\_\_\_  
No. of Samples/Matrix \_\_\_\_\_ Date Rec'd by lab \_\_\_\_\_

Traffic Report Sample Nos. \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Equipment Blank No. \_\_\_\_\_  
Bottle Blank No. \_\_\_\_\_  
Field Duplicate Nos. \_\_\_\_\_  
PES Nos. \_\_\_\_\_

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, revision \_\_\_\_\_ was used to evaluate the data and/or approved modifications to the EPA-NE Functional Guidelines were used to evaluate the data and are attached to this cover page: (attach modified criteria from EPA approved QAPP or amendment to QAPP).

A Tier II or Tier III evaluation was used to validate the data (circle one). If a Tier II validation with a partial Tier III was used, then identify samples, parameters, etc., that received partial Tier III validation.

The data were evaluated based upon the following parameters:

- Overall Evaluation of Data
- Data Completeness (CSF Audit - Tier I)
- Preservation and Technical Holding Times
- ICP-MS Tune
- Calibrations
- Blanks
- ICP-AES Interference Check Sample (ICS)
- ICP-MS Interference Check Sample (ICS)
- ICP-MS Internal Standards
- Matrix Spikes
- Laboratory Duplicate Samples
- Field Duplicates
- ICP Serial Dilutions
- Sensitivity Check
- Performance Evaluation Samples/Accuracy Check
- Analyte Quantitation and Reported Quantitation Limits
- System Performance

Region I Definitions and Qualifiers:

- A - Acceptable Data
- J - Numerical value associated with analyte is an estimated quantity.
- R - The data are rejected as unusable. The R replaces the numerical value or sample quantitation limit.
- U - Analyte not detected at that numerical sample quantitation limit.
- UJ - The sample quantitation limit is an estimated quantity.
- BB, EB - Analyte detected in aqueous bottle blank or aqueous equipment blank associated with soil/sediment samples.

Validator's Name \_\_\_\_\_ Company Name \_\_\_\_\_ Phone Number \_\_\_\_\_

Date Validation Started \_\_\_\_\_ Date Validation Completed \_\_\_\_\_

Check if all criteria are met and no hard copy worksheet is provided. Indicate NA if worksheet is not applicable to the analytical method. Note: there is no standard worksheet for System Performance; however, the validator must document all system performance issues in the Data Validation Memorandum.

INORG Worksheets:

INORG	COMPLETE SDG FILE (CSF) AUDIT	_____
INORG-I	PRESERVATION AND TECHNICAL HOLDING TIMES	_____
INORG-II	ICP-MS TUNE	_____
INORG-III-A/B	CALIBRATIONS	_____
INORG-IV-A/B	BLANKS	_____
INORG-IV-C.1	BLANKS	_____
INORG-IV-C.2	BLANKS	_____
INORG-V-A	ICP-AES INTERFERENCE CHECK SAMPLE - ICSAB	_____
INORG-V-B.1	ICP-AES INTERFERENCE CHECK SAMPLE - ICSA	_____
INORG-V-B.2	ICP-AES INTERFERENCE CHECK SAMPLE - ICSA	_____
INORG-VI-A	ICP-MS INTERFERENCE CHECK SAMPLE - ICSAB	_____
INORG-VI-B	ICP-MS INTERFERENCE CHECK SAMPLE - ICSA	_____
INORG-VII	ICP-MS INTERNAL STANDARDS	_____
INORG-VIII	MATRIX SPIKES	_____
INORG-IX	LABORATORY DUPLICATE SAMPLES	_____
INORG-X	FIELD DUPLICATES	_____
INORG-XI	ICP SERIAL DILUTIONS	_____
INORG-XII-A/B	SENSITIVITY CHECK	_____
INORG-XIII-A	PE SAMPLES/ACCURACY CHECK- LCS	_____
INORG-XIII-B	PE SAMPLES/ACCURACY CHECK- PE RESULTS	_____
INORG-XIV	ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS	_____
TABLE II-WORKSHEET	OVERALL EVALUATION OF DATA	_____

I certify that all criteria were met for the worksheets checked above.

Signature: \_\_\_\_\_

Name: \_\_\_\_\_

Date: \_\_\_\_\_

The data validator generates a Data Validation Report, applicable to Data Validation Tiers II and III, that consists of the following components in the order specified below: (Refer to Part I - Data Validation Manual, Section 11 for a description of each of the Data Validation Report components).

1. Organic Regional Data Assessment/Inorganic Regional Data Assessment (ORDA/IRDA) Form
2. Data Validation Memorandum
  - a. Narrative
  - b. Table I-Qualifier Recommendation Summary Table
  - c. Table II-Overall Evaluation of Data
  - d. Data Summary Tables
3. Standard Data Validation Worksheets
  - a. Manual
  - b. Automated Data Review Reports
4. Support Documentation
  - a. Copy of non-CLP analytical method, e.g., DAS methods, modified EPA methods
  - b. Copies of PES Score Reports/Vendor PES QC Acceptance Limits
  - c. Copies of Telephone Logs/Communication Forms for:
    - RSCC communications
    - Requests for laboratory data resubmissions/clarifications
    - Communications with samplers resolving sampling problems
    - Communications with PO/DV Chemist/Lead Chemist to report contractually-deficient data for rejection/reduced payment
    - Communications with EPA Site Manager concerning possible data rejection
    - EPA Site Manager authorization for alternate DV tier
  - d. Copies of data supporting recommendations for reduced payment resulting from CSF Audit and/or PE sample result evaluation
  - e. Original data to support recommendations for data rejection/non-payment identified from Tier II or Tier III data validation
  - f. Copies of field sampling notes and/or field report supplied by field sampler
  - g. Copies of EPA-approved amendments to QAPP and/or SAP describing modified criteria to be used for validating site data
5. CSF Completeness Evidence Audit
6. DQO Summary Form

The data validator is responsible for implementing all corrective actions required by the contractor Lead Chemist in response to EPA-NE data validation oversight findings.

EPA-NE - Data Validation Worksheet  
**Overall Evaluation of Data - Data Validation Memorandum - Table II**

Site: \_\_\_\_\_ Case: \_\_\_\_\_ SDG: \_\_\_\_\_

INORGANICS					
DQO (List all DQOs)	Sampling and/or Analytical Method Appropriate? (Yes or No)	Measurement Error		Sampling Variability**	Potential Usability Issues
		Analytical Error	Sampling Error*		

\* The evaluation of "sampling error" cannot be completely assessed in data validation.  
 \*\* Sampling variability is not assessed in data validation.

Validator: \_\_\_\_\_ Date: \_\_\_\_\_







**III. CALIBRATIONS**

**A. Instrument Calibration** - List all calibration correlation coefficients that are outside the method QC acceptance criteria.  
 Calibration correlation QC acceptance criteria: \_\_\_\_\_  
 Calibration Type: \_\_\_\_\_

Date/Time	Instr.	Analyte	Corr. Coef.	Samples Affected	Action

**B. Initial and Continuing Calibration Verifications** - List all ICV and CCV analyte recoveries that are outside the method QC acceptance criteria.  
 ICV method QC acceptance criteria: \_\_\_\_\_  
 CCV method QC acceptance criteria: \_\_\_\_\_

Date	Instr.	Analyte	ICV/CCV #	% R	Samples Affected	Action

**C. Quantitation Limit Check Standard** - List all QL Check Standard analytes that are outside method QC acceptance criteria.  
 QL Check Standard method QC acceptance criteria: \_\_\_\_\_

Date	Instr.	Analyte	QL Check Std. #	% R	Affected Range	Samples Affected	Action

Comments: \_\_\_\_\_

Validator: \_\_\_\_\_

Date: \_\_\_\_\_



EPA-NE - Data Validation Worksheet  
 INORG-IV-C.1  
 IV. BLANKS

C.1 Blank Contamination Worksheet

Circle the highest concentration of each contaminant.

Analyte	Date Analyzed	ICB	CCB							PBW	PBS	EB	BB	Max. Conc.	Action Level
			1	2	3	4	5	6	7						
Aluminum															
Antimony															
Arsenic															
Barium															
Beryllium															
Cadmium															
Calcium															
Chromium															
Cobalt															
Copper															
Iron															
Lead															
Magnesium															
Manganese															
Mercury															
Nickel															
Potassium															
Selenium															
Silver															
Sodium															
Thallium															
Vanadium															
Zinc															
Cyanide															

Validator: \_\_\_\_\_ Date: \_\_\_\_\_























**XII. SENSITIVITY CHECK**

**A. Method Detection Limit Study** - List all analytes that are outside the MDL criteria.

- Has an appropriate MDL study been submitted with seven replicates for each analyte and matrix of interest?      **Y**   **N**
- Were samples analyzed within one year of the MDL study and on the same instrument?                      **Y**   **N**

Matrix	Analyte	MDL	MDL > QL	Samples Affected	Action

If an MDL study has not been submitted, use only the LFB results to evaluate data.

**B. Laboratory Fortified Blank** - List all LFB analytes that are outside the LFB criteria.

- Has an appropriate and complete LFB been submitted at the proper frequency?                      **Y**   **N**
- Does the LFB contain all target analytes at their QLs?    **Y**   **N**

Matrix	Method	Analyte	% R	Method QC Limits % R	Samples Affected	Action

Comments: \_\_\_\_\_

Validator: \_\_\_\_\_ Date: \_\_\_\_\_





EPA-NE - Data Validation Worksheet  
**INORG-XIV**

**XIV. ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS**

Recalculate, from the raw data, the concentrations for one positive detect and one reported sample quantitation limit for a non-detect in a diluted sample or soil sample per analytical method. (Note: Although Section XIV, C.2.a, requires that one calculation for each method in each sample be performed, the validator is only required to reproduce an example, for each method, of one positive detect and one sample quantitation limit calculation on this worksheet.)

Do all soil/sediment samples have % solids greater than 30%?      **Y**      **N**

- If no, were any steps employed to address the high moisture content? \_\_\_\_\_
- Indicate the action and list the affected sample nos.: \_\_\_\_\_

Method		Calculation
<b>ICP-AES</b>		
Sample No.:		
Reported Analyte:		
Reported Value:		
Non-Detected Analyte:		
Reported Quantitation Limit:		
<b>ICP-MS</b>		
Sample No.:		
Reported Analyte:		
Reported Value:		
Non-Detected Analyte:		
Reported Quantitation Limit:		
<b>Mercury</b>		
Sample No.:		
Reported Value:		
Sample No.:		
Reported Quantitation Limit:		
<b>Cyanide</b>		
Sample No.:		
Reported Value:		
Sample No.:		
Reported Quantitation Limit:		

Validator: \_\_\_\_\_

Date: \_\_\_\_\_