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Waste & Environmental Services

Standard Operating Procedure

for **ROUTINE VALIDATION OF METALS ANALYTICAL DATA**

APPROVAL SIGNATURES:

Subject Matter Expert:	Organization	Signature	Date
Bill Hardesty	WES-EDA	Signature on file	4/21/08
Quality Assurance Specialist:	**Organization**	**Signature**	**Date**
Laura Ortega	QA-IQ	Signature on file	5/14/08
Responsible Line Manager:	**Organization**	**Signature**	**Date**
Craig Eberhart	WES-EDA	Signature on file	4/21/08

1.0   PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine Metals analytical data. This procedure is a mandatory document and shall be implemented by all Los Alamos National Laboratory (LANL or Laboratory) personnel and contractors who evaluate routine Metals analytical data for the specific LANL projects.

2.0   BACKGROUND AND PRECAUTIONS

2.1   Background

This procedure conforms to the requirements of Environmental Protection Agency (EPA) Methodologies and the EPA document, "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review." LANL data validation is performed according to procedures based upon the NNSA Model Data Validation Procedure. Data qualifiers and reason codes are assigned according to the specifications in this method specific procedure.

2.2   Precautions

Nothing in this procedure precludes the data validator from going beyond the minimum requirements specified within this procedure. If additional directions are required, the data validator shall reference NNSA Model Data Validation Procedure, EPA method specific guidelines and/or National Functional Guidelines for Inorganic Data Review. Implementation of this procedure may be followed by a more focused and data use-specific evaluation of the data by the project chemist, especially if the implementation of this procedure indicates the data may contain technical deficiencies.

3.0   EQUIPMENT AND TOOLS

None.

4.0   STEP-BY-STEP PROCESS DESCRIPTION

4.1   Qualifications for Data Validators

Data Validator	1.	Possess a minimum of a bachelor's degree in chemistry, or one of the physical sciences
		AND
		either two (2) years of experience in generating analytical data in an environmental analytical laboratory
		AND
		two (2) years of data validation experience.
	2.	Complete Attachment 1, Data Validation Cover Sheet, and Attachment 2, Metals Analytical Data Validation Checklist, during data validation.
	3.	Refer to Attachment 3, Guidance for the Qualifier and Reason Code Application, for additional guidance.

4.2   Records

Data Validator	1.	Submit the following records generated by this procedure to the Records Processing Facility:

- Completed Data Validation Cover Sheets; and
- Completed Metals Analytical Data Validation Checklists.

5.0   PROCESS FLOW CHART

For specific validation criteria follow the NNSA Model for Data Validation.

6.0   ATTACHMENTS

Attachment 1   5165-1 Data Validation Cover Sheet (1 page)

Attachment 2   5165-2 Metals Analytical Data Validation Checklist (4 pages)

Attachment 3   5165-3 Guidance for the Qualifier and Reason Code Application (4 pages)

7.0   REVISION HISTORY

Author: Bill Hardesty

Revision No. *[Enter current revision number, beginning with Rev.0]*	Effective Date *[DCC inserts effective date for revision]*	Description of Changes *[List specific changes made since the previous revision]*	Type of Change *[Technical (T) or Editorial (E)]*
0		New Document	T

Using a CRYPTOCard, click here to record "self-study" training to this procedure.

If you do not possess a CRYPTOCard or encounter problems, contact the EP training specialist.

ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

5165-1 **Example of a Data Validation Cover Sheet**	Records Use only **• Los Alamos** NATIONAL LABORATORY — EST. 1943 —

Section I.

REQUEST NUMBER: _____ VALIDATION DATE: _____ LAB CODE: _____

CONTRACT LABORATORY NAME: _____

VALIDATOR: _____ ORGANIZATION: _____

ANALYTICAL SUITE (CHECK ALL THAT APPLY):

☐ **TPH-GRO**   ☐ **HIGH EXPLOSIVES**   ☐ **DIOXIN FURANS**   ☐ **LCMSMS PERCHLORATES**

☐ **TPH-DRO**   ☐ **METALS**   ☐ **PCB CONGENERS**   ☐ **ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS**

☐ **GENERAL CHEMISTRY**   ☐ **RADIOCHEMISTRY**   ☐ **LCMSMS HIGH EXPLOSIVES**

☐ **OTHER (DESCRIBE):** _____

Section II.   Completeness Check

YES	NO	N/A	(CHECK ONE)	YES	NO	N/A	(CHECK ONE)
☐	☐	☐	1. CHAIN-OF-CUSTODY FORM(S)	☐	☐	☐	6. RAW/BSS DATA
☐	☐	☐	2. CASE NARRATIVE	☐	☐	☐	7. QUALITY CONTROL FORMS
☐	☐	☐	3. SAMPLE RESULT FORMS	☐	☐	☐	8. QUANTITATION REPORTS
☐	☐	☐	4. SAMPLE CHROMATOGRAMS	☐	☐	☐	9. TICS FORMS
☐	☐	☐	5. STANDARD CHROMATOGRAMS	☐	☐	☐	10. TICS MASS SPECTRA

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

VALIDATOR'S SIGNATURE:_____ DATE: _____

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ATTACHMENT 2: METALS ANALYTICAL DATA VALIDATION CHECKLIST

5165-2 **Metals Analytical Data Validation Checklist**	Records Use only Los Alamos NATIONAL LABORATORY — EST. 1943 —

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				**Non-detected Analyte**	**Detected Analyte**
☐	☐	☐	1. The holding time was >1 and ≤2 times the applicable holding time requirement.	**UJ, I9**	**J-, I9**
☐	☐	☐	2. The holding time was >2 times the applicable holding time requirement.	**R, I9a**	**J-, I9a**
☐	☐	☐	3. The instrument performance sample did not pass method acceptance criteria.	**R, I16**	**R, I16**
☐	☐	☐	4. The mass calibration is not within 0.1 amu or %RSD is >5% for any isotope (Be, Mg, Co, In, Pb).	**UJ, I16a**	**J, I16a**
☐	☐	☐	5. Samples were analyzed outside specific method tune time criteria.	**N/A**	**J, I16b**
☐	☐	☐	6. The required instrument performance sample information is missing. Contact the SMO or external laboratory for information.	**R, I16c**	**R, I16c**
☐	☐	☐	7. The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.	**UJ, R, I7**	**J, I7**
☐	☐	☐	8. The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD criteria and/or the associated multipoint calibration correlation coefficient is <0.995.	**UJ, I7a**	**J+, I7a**
☐	☐	☐	9. The initial Calibration Verification (ICV) and/or Continuing Calibration Verification (CCV) were recovered outside the method-specific limits.	**UJ, I7c**	**J, I7c**
☐	☐	☐	10. The ICV and/or CCV were not analyzed at the appropriate method frequency.	**UJ, I7d**	**J+, I7d**
☐	☐	☐	11. Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.	**R, I7f**	**R, I7f**

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				**Non-detected Analyte**	**Detected Analyte**
☐	☐	☐	12. Metals interference check sample percent recover value is <50%.	R, I2	J-, I2
☐	☐	☐	13. Metals interference check sample percent recovery value is ≥50% and <80%	UJ, I2a	J-, I2a
☐	☐	☐	14. Metals interference check sample percent recovery value is >120%.	N/A	J+, I2b
☐	☐	☐	15. Metals interference check sample was not analyzed with the samples.	R, I2c	R, I2c
☐	☐	☐	16. The sample result is ≤5X the concentration of the related analyte in the method blank.	N/A	U, I4
☐	☐	☐	17. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5X.	N/A	J+, I4a
☐	☐	☐	18. The sample result is ≤5X the concentration of the related analyte in the instrument blank and continuing calibration blank.	N/A	U, I4b
☐	☐	☐	19. Continuing calibration blanks were not analyzed at the appropriate method frequency.	UJ, I4c	J, I4c
☐	☐	☐	20. The sample result is ≤5X the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank.	N/A	U, I4d
☐	☐	☐	21. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, I4e	R, I4e
☐	☐	☐	22. The associated matrix spike recovery was <10%. Follow the external laboratory limits located within the associated data package.	R, I6	R, I6
☐	☐	☐	23. The associated matrix spike recovery was <the LAL but >10%. Follow the external laboratory limits located within the associated data package.	UJ, I6a	J-, I6a
☐	☐	☐	24. The associated matrix spike recovery was > the UAL. Follow the external laboratory limits located within the associated data package.	UJ, I6b	J+, I6b

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	25. Required matrix spike information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. If the LCS information is present, do not Reject. Qualify data based on the LCS information.	R, I6c	R, I6c
☐	☐	☐	26. The sample and the duplicate sample results were ≥5X the RL and the duplicate RPD was >20% for water samples and >35% for soil samples.	UJ, I10a	J, I10a
☐	☐	☐	27. The duplicate sample was not prepared and/or analyzed with the samples for unspecified reasons. The duplicate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	UJ, I10d	J, I10d
☐	☐	☐	28. The LCS percent recovery was <10%. Follow the external laboratory limits located within the associated data package.	R, I12	R, I12
☐	☐	☐	29. The LCS percent recover was < the LAL but >10%. Follow the external laboratory limits located within the associated data package.	UJ, I12a	J-, I12a
☐	☐	☐	30. The LCS percent recovery was > the UAL. Follow the external laboratory limits located within the associated data package.	N/A	J+, I12b
☐	☐	☐	31. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. Do not Reject if MS/MSD information is present. Qualify according to MS/MSD criteria.	R, I12c	R, I12c
☐	☐	☐	32. The quantitating IS area count is <10% for metals window in relation to the initial calibration blank. Follow the method-specific windows.	R, I1a	J, I1a
☐	☐	☐	33. The IS area count for the quantitating IS is <60% but >10% for metals window in relation to the initial calibration blank. Follow the method-specific windows.	UJ, I1b	J, I1b

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				**Non-detected Analyte**	**Detected Analyte**
☐	☐	☐	34. The IS area count for the quantitating IS is >125% in relation to the metals initial calibration blank. Follow method-specific windows.	UJ, I1c	J, I1c
☐	☐	☐	35. Required IS information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, I1d	R, I1d
☐	☐	☐	36. Serial dilution sample %D was >10% and the sample result was >50X the MDL (>100X the MDL for ICPMS). Qualify ONLY the sample used for the serial dilution.	UJ, I18	J, I18
☐	☐	☐	37. Serial dilution sample was not analyzed with the samples.	UJ, I18a	J, I18a
☐	☐	☐	38. The sample result was reported as detected between the IDL and the EDL.	N/A	J, I1
☐	☐	☐	39. Duplicate, dilution, or reanalysis.	UJ, I88	J, I88
☐	☐	☐	40. Qualification of data via data validation did not occur based on Quality Control requirements in this procedure. Adhere to the external laboratory qualifiers found within the Form I analytical data summary sheets generated by the external laboratory.	U, U_LAB	J, J_LAB, NQ, NQ
☐	☐	☐	41. The LANL project chemist identified quality deficiencies in the reported data that require further qualification. This code can ONLY be used and/or under advisement by the LANL project chemist.	UJ, R, I19	J, R, I19

ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION	
5165-3 **Guidelines for the Qualifier and Reason Code Application**	Records Use only **Los Alamos** NATIONAL LABORATORY — EST.1943 —

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
1	N/A	J	I1	The sample result was reported as detected between the IDL and the EDL.
2	UJ	J	I10a	The sample and the duplicate sample results were ≥5X the RL and the duplicate RPD was >20% for water samples and >35% for soil samples.
3	UJ	J	I10d	The duplicate sample was not prepared and/or analyzed with the samples for unspecified reasons. The duplicate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
4	R	R	I12	The LCS percent recovery was <10%. Follow the external laboratory limits located within the associated data package.
5	UJ	J-	I12a	The LCS percent recovery was < the Lower Acceptance Limit (LAL) but >10%. Follow the external laboratory limits located within the associated data package.
6	N/A	J+	I12b	The LCS percent recovery was > Upper Acceptance Limit (UAL). Follow the external laboratory limits located within the associated data package.
7	R	R	I12c	The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. Do not Reject if MS/MSD information is present. Qualify according to MS/MSD criteria.
8	R	R	I16	The instrument performance sample did not pass the method acceptance criteria.

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
9	UJ	J	I16a	The mass calibration is not within 0.1 amu or %RSD exceeds 5% for any isotope (Be, Mg, Co, In, Pb).
10	N/A	J	I16b	Samples were analyzed outside specific method tune time criteria.
11	R	R	I16c	The required instrument performance sample information is missing. Contact the SMO or external laboratory for information.
12	UJ	J	I18	Serial dilution sample RPD was >10% and the sample results was >50X the MDL (>100X the MDL for ICPMS). Qualify ONLY the sample used for the serial dilution.
13	UJ	J	I18a	Serial dilution sample was not analyzed with the samples.
14	UJ, R	J, R	I19	The project chemist identified quality deficiencies in the reported data that requires further qualification. This code can ONLY be used and/or under the advisement by the project chemist.
15	R	J	I1a	The quantitating IS area could is <10% for metals window in relation to the initial calibration blank. Follow method-specific windows.
16	UJ	J	I1b	The IS area count for the quantitating IS is <60% but >10% for metals window in relation to the initial calibration blank. Follow method-specific windows.
17	UJ	J	I1c	The IS area count for the quantitiating IS is >125% in relation to the metals initial calibration blank. Follow method-specific windows.
18	R	R	I1d	Required IS information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
19	R	J-	I2	Metals interference check sample percent recovery value is <50%.
20	UJ	J-	I2a	Metals interference check sample percent recovery value is ≥50% and <80%.
21	N/A	J+	I2b	Metals interference check sample percent recovery value is >120%.
22	R	R	I2c	Metals interference check sample was not analyzed with the samples.

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
23	N/A	U	I4	The sample result is ≤5X the concentration of the related analyte in the method blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.
24	N/A	J	I4a	The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5X.
25	N/A	U	I4b	The sample result is ≤5X the concentration of the related analyte in the ICB/CCB, which indicates the reported detection is considered indistinguishable from contamination in the blank.
26	UJ	J	I4c	Continuing calibration blanks were not analyzed at the appropriate method frequency.
27	N/A	U	I4d	The sample result is ≤5X the concentration of the related analyte in the trip blank, equipment blank, or rinsate, which indicates the reported detection is considered indistinguishable from contamination in the blank.
28	R	R	I4e	Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
29	R	R	I6	The associated matrix spike recovery was <10%. Follow the external laboratory limits located within the associated data package.
30	UJ	J-	I6a	The associated matrix spike recovery was < the LAL but >10%. Follow the external laboratory limits located within the associated data package.
31	UJ	J+	I6b	The associated matrix spike recovery was > the UAL. Follow the external laboratory limits located within the associated data package.
32	R	R	I6c	Required matrix spike information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
33	UJ, R	J	I7	The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
34	UJ	J	I7a	The affected analytes were analyzed with a initial calibration curve that exceeded the %RSD criteria and/or the associated multipoint calibration correlation coefficient is <0.995.
35	UJ	J	I7c	The ICV and/or CCV were recovered outside the method-specific limits.
36	UJ	J	I7d	The ICV and/or CCV were not analyzed at the appropriate method frequency.
37	R	R	I7f	Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.
38	UJ	J	I88	Duplicate, dilution, or reanalysis.
39	UJ	J-	I9	The extraction/analytical holding time are exceeded by <2X the published method for holding times.
40	R	J-	I9a	The extraction/analytical holding time are exceeded by >2X the published method for holding times.
41	U	J, NQ	U_LAB, J_LAB, NQ	Qualification of the data via data validation did not occur because of Quality Control requirements in this procedure. Adhere to external laboratory qualifiers found within the Form I analytical data summary sheets generated by the external laboratory.

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