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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 

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 (Check One)		Assign Qualifier Listed Below If Criterion = Yes	
				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

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Guidelines for the Qualifier and Reason Code Application

Records Use only

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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