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Environment, Safety, Health Directorate

OIO-DO: Operations Integration Office

Technical Procedure

ROUTINE VALIDATION OF METALS ANALYTICAL DATA

Subject Matter Expert:

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REVISION HISTORY

Document Number and Revision *[Include revision number, beginning with Revision 0]*	**Effective Date** *[Document Control Coordinator inserts effective date]*	**Description of Changes** *[List specific changes made since the previous revision]*
OIO-TP-5165, Rev. 0.1	8/19/2015	Periodic Review. Minor revision, changed document type and organization.

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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 ANDPRECUATIONS

2.1 Background

This procedure conforms to the requirements of U.S. 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 National Nuclear Security Administration (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 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.

5.0 RECORDS

Records generated by this document will be submitted to the Operations Integration Office Records Management designated point of contact for document management in accordance with P1020-1 Laboratory Records Management, and with the ADESH-AP-006, Records Management Plan:

- Completed Data Validation Cover Sheets
- Completed General Chemistry Analytical Data Validation Checklists

6.0 PROCESS FLOW CHART

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

7.0 ATTACHMENTS

Attachment 1: *5165-1 Data Validation Cover Sheet*
Attachment 2: *5165-2 Metals Analytical Data Validation Checklist*
Attachment 3: *5167-3 Guidance for the Qualifier and Reason Code Application*

ATTACHMENT 1 – 5165-1 EXAMPLE OF A DATA VALIDATION COVER SHEET

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Section I.

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

CONTRACT LABORATORY NAME: _____

VALIDATOR: _____ ORGANIZATION: _____

ANALYTICAL SUITE (CHECK ALL THAT APPLY):

- ☐ **TPH-GRO**
- ☐ **TPH-DRO**
- ☐ **GENERAL CHEMISTRY**
- ☐ **HIGH EXPLOSIVES**
- ☐ **METALS**
- ☐ **RADIOCHEMISTRY**
- ☐ **DIOXIN FURANS**
- ☐ **PCB CONGENERS**
- ☐ **LCMSMS HIGH EXPLOSIVES**
- ☐ **LCMSMS PERCHLORATES**
- ☐ **ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS**
- ☐ **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: _____

(Attach additional comment sheets as necessary)

ATTACHMENT 2 – 5165-2 GENERAL CHEMISTRY ANALYTICAL DATA VALIDATION CHECKLIST

Page 1 of 4

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 percent relative standard deviation (%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 Sample Management Office (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 (RL).	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
☐	☐	☐	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%.	UJ, I7d	J+, I7d
☐	☐	☐	15. Metals interference check sample was not analyzed with the samples.	R, I7f	R, I7f

ATTACHMENT 2 – 5165-2 GENERAL CHEMISTRY ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)
Page 2 of 4

Yes	No	N/A (Check One)		Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
☐	☐	☐	16. The sample result is ≤5 times the concentration of the related analyte in the method blank.	R, I2	J-, I2
☐	☐	☐	17. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5 times.	UJ, I2a	J-, I2a
☐	☐	☐	18. The sample result is ≤5 times the concentration of the related analyte in the instrument blank and continuing calibration blank.	N/A	J+, I2b
☐	☐	☐	19. Continuing calibration blanks were not analyzed at the appropriate method frequency.	R, I2c	R, I2c
☐	☐	☐	20. The sample result is ≤5 times the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank.	N/A	U, I4
☐	☐	☐	21. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	N/A	J+, I4a
☐	☐	☐	22. The associated matrix spike (MS) recovery was <10%. Follow the external laboratory limits located within the associated data package.	N/A	U, I4b
☐	☐	☐	23. The associated matrix spike recovery was <the lower acceptance limit (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 upper acceptance limit (UAL). Follow the external laboratory limits located within the associated data package.	UJ, I6b	J+, I6b
☐	☐	☐	25. Required matrix spike information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. If the laboratory control sample (LCS) information is present, do not reject. Qualify data based on the LCS information.	R, I6c	☐

ATTACHMENT 2 – 5165-2 GENERAL CHEMISTRY ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)
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Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	26. The sample and the duplicate sample results were ≥5 the RL and the duplicate relative percent difference (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/MS duplicate (MSD) information is available. Qualify according to MS/MSD criteria.	R, I12c	R, I12c
☐	☐	☐	32. The quantitating internal standard (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 <60% but >10% for metals window in relation to the initial calibration blank. Follow the method-specific windows.	UJ, I1b	J, I1b
☐	☐	☐	34. The IS area count for the quantitating 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

ATTACHMENT 2 – 5167-2 GENERAL CHEMISTRY ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)

Yes	No	N/A (Check One)		Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	**Detected Analyte**
☐	☐	☐	36. Serial dilution sample % difference (%D) was >10% and the sample result was >50 times the method detection limit (MDL) (>100 times the MDL for inductively coupled plasma mass spectrometry). 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 instrument detection limit (IDL) and the estimated detection limit (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 by and/or used under advisement of the LANL project chemist.	UJ, R, I19	J, R, I19

ATTACHMENT 3 – 5165-2 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

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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 instrument detection limit (IDL) and the estimated detection limit (EDL).
2	UJ	J	I10a	The sample and the duplicate sample results were ≥5 times the reporting limit (RL) and the duplicate relative percent difference (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 Sample Management Office (SMO) or external laboratory for information.
4	R	R	I12	The laboratory control sample (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 matrix spike (MS)/MS duplicate (MSD) information is available. Qualify according to MS/MSD criteria.
8	R	R	I16	The instrument performance sample did not pass the method acceptance criteria.
9	UJ	J	I16a	The mass calibration is not within 0.1 amu or percent relative standard deviation (%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 >50 times the MDL (>100 times the MDL for inductively coupled plasma mass spectrometry). Qualify ONLY the sample used for the serial dilution.
13	UJ	J	I18a	Serial dilution sample was not analyzed with the samples.

ATTACHMENT 3 – 5165-2 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION (CONT.)

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
14	UJ, R	J, R	I19	The project chemist identified quality deficiencies in the reported data that require further qualification. This code can ONLY be used by and/or used under advisement of the project chemist.
15	R	J	I1a	The quantitating internal standard (IS) area is <10% for metals window in relation to the initial calibration blank (ICB). Follow method-specific windows.
16	UJ	J	I1b	The IS area count for the quantitating IS <60% but >10% for metals window in relation to the ICB. Follow method-specific windows.
17	UJ	J	I1c	The IS area count for the quantitating IS is >125% in relation to the ICB. 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.
23	N/A	U	I4	The sample result is ≤5 times the concentration of the related analyte in the method blank, indicating 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 >5 times.
25	N/A	U	I4b	The sample result is ≤5 times the concentration of the related analyte in the ICB/continuing calibration blank (CCB), indicating the reported detection is considered indistinguishable from contamination in the blank.
26	UJ	J	I4c	CCBs were not analyzed at the appropriate method frequency.
27	N/A	U	I4d	The sample result is ≤5 times the concentration of the related analyte in the trip blank, equipment blank, or rinsate, indicating 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.

ATTACHMENT 3 – 5165-2 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION (CONT.)

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29	R	R	I6	The associated MS recovery was <10%. Follow the external laboratory limits located within the associated data package.
30	UJ	J-	I6a	The associated MS recovery was < the LAL but >10%. Follow the external laboratory limits located within the associated data package.
31	UJ	J+	I6b	The associated MS recovery was > the UAL. Follow the external laboratory limits located within the associated data package.
32	R	R	I6c	Required MS 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 RL.
34	UJ	J	I7a	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.
35	UJ	J	I7c	The initial calibration verification (ICV) and/or continuous calibration verification (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 <2 times the published method for holding times.
40	R	J-	I9a	The extraction/analytical holding time are exceeded by >2 times 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.

Los Alamos
NATIONAL LABORATORY
EST. 1943

Environment, Safety and Health

Electronic Public Reading Room - Posting of Controlled Procedures

Operations Integration Office Management Approval:

Print Name	Signature	Date
Ellena Martinez	Ellena Martinez	3/4/16

Derivative Classifier:

☐ OUO ☐ UCNI ☒ Unclassified ☐ Classified

Print Name	Signature	Date
Larry W. Maassen	Larry Maassen	3/4/16

List of Controlled Documents:

Procedure No.	Title/Description
Air Monitoring (ENV)	
ENV-ES-TPP-003	Technical Project Plan for the Neighborhood Environmental Watch Network (NEWNET)
ENV-ES-TPP-007	Technical Project Plan for the Direct Penetrating Radiation Monitoring Network (DPRNET)
Data Validation (ADESH)	
OIO-TP-5161	Routine Validation of Volatile Organic Compound Analytical Data
OIO-TP-5162	Routine Validation of Semivolatile Organic Compound Analytical Data
OIO-TP-5163	Routine Validation of Organochlorine Pesticide and Polychlorinated Biphenyl Analytical Data
OIO-TP-5165	Routine Validation of Metals Analytical Data
General Field Work	
OIO-TP-222	Shipping/Receiving of Environmental Samples by the Sample Management Office (SMO)
OIO-QP-219	Sample Control and Field Documentation
Soil, Foodstuffs, and Biota Sampling (ENV)	
ENV-ES-TPP-002	Technical Project Plan for Biota Dose Assessment
ENV-ES-TP-003	Collection of Soil and Vegetation Samples for the Environmental Surveillance Program
ENV-ES-TP-004	Produce Sampling
ENV-ES-TP-007	Game Animal Sampling
ENV-ES-TP-006	Sampling Soil and Vegetation at Facility Sites
SOP-5247	Collection of Benthic Macroinvertebrates in the Rio Grande
ENV-ES-TP-008	Collection of Crawfish in the Rio Grande
Well Drilling, Construction, Development, Maintenance, and Abandonment	
ENV-RCRA-QP-010	Land Application of Groundwater