

Identifier: SOP-5167

Revision: 0



Effective Date: 6/30/08

Next Review Date: 6/30/13

## Waste & Environmental Services

### Standard Operating Procedure

# for ROUTINE VALIDATION OF GENERAL CHEMISTRY ANALYTICAL DATA

#### APPROVAL SIGNATURES:

Subject Matter Expert:	Organization	Signature	Date
Bill Hardesty	WES-EDA		4/21/2008
Quality Assurance Specialist:	Organization	Signature	Date
Laura Ortega	QA-IQ		5/14/08
Responsible Line Manager:	Organization	Signature	Date
Craig Eberhart	WES-EDA		4/21/2008

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## 1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine General Chemistry 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 General chemistry 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, General Chemistry Analytical Data Validation Checklist, during data validation.
	3.	Refer to Attachment 3, Guidance for the Qualifier and Reason Code Application, for additional guidance.

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## 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 General Chemistry 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 5167-1 Data Validation Cover Sheet (1 page)

Attachment 2 5167-2 General Chemistry Analytical Data Validation Checklist (3 pages)

Attachment 3 5167-3 Guidance for the Qualifier and Reason Code Application (3 pages)

## 7.0 REVISION HISTORY

Author: Bill Hardesty

Revision No. <i>[Enter current revision number, beginning with Rev.0]</i>	Effective Date <i>[DCC inserts effective date for revision]</i>	Description of Changes <i>[List specific changes made since the previous revision]</i>	Type of Change <i>[Technical (T) or Editorial (E)]</i>
0	6/30/08	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.

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**ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET**

**5167-1**

**Example of a Data Validation Cover Sheet**

Records Use only



**Section I.**

REQUEST NUMBER: \_\_\_\_\_ VALIDATION DATE: \_\_\_\_\_ LAB CODE: \_\_\_\_\_

CONTRACT LABORATORY NAME: \_\_\_\_\_

VALIDATOR: \_\_\_\_\_ ORGANIZATION: \_\_\_\_\_

ANALYTICAL SUITE (CHECK ALL THAT APPLY):

- |  |  |   |  |
|--|--|---|--|
| <input type="checkbox"/> TPH-GRO                 | <input type="checkbox"/> HIGH EXPLOSIVES | <input type="checkbox"/> DIOXIN FURANS          | <input type="checkbox"/> LCMSMS PERCHLORATES                                 |
| <input type="checkbox"/> TPH-DRO                 | <input type="checkbox"/> METALS          | <input type="checkbox"/> PCB CONGENERS          | <input type="checkbox"/> ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS |
| <input type="checkbox"/> GENERAL CHEMISTRY       | <input type="checkbox"/> RADIOCHEMISTRY  | <input type="checkbox"/> LCMSMS HIGH EXPLOSIVES |  |
| <input type="checkbox"/> OTHER (DESCRIBE): _____ |  |   |  |

**Section II. Completeness Check**

YES	NO	N/A	(CHECK ONE)	YES	NO	N/A	(CHECK ONE)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. CHAIN-OF-CUSTODY FORM(S)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. RAW/BSS DATA
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. CASE NARRATIVE	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. QUALITY CONTROL FORMS
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. SAMPLE RESULT FORMS	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. QUANTITATION REPORTS
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. SAMPLE CHROMATOGRAMS	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. TICS FORMS
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. STANDARD CHROMATOGRAMS	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	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: \_\_\_\_\_

SOP-5167, Revision 0.0

LOS ALAMOS  
Environmental Restoration Project

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## ATTACHMENT 2: GENERAL CHEMISTRY ANALYTICAL DATA VALIDATION CHECKLIST

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### General Chemistry Analytical Data Validation Checklist

Records Use only



Yes No N/A (Check One)				Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. The holding time was >1 and ≤2 times the applicable holding time requirement.	UJ, I9	J-, I9
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. The holding time was >2 times the applicable holding time requirement.	R, I9a	J-, I9a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. The affected analytes are regarded as rejected because the analytical holding time was exceeded.	R, I9b	R, I9b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. The ICV and/or CCV were recovered outside the method specific limits.	UJ, I7c	J, I7c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. The ICV and/or CCV were not analyzed at the appropriate method frequency.	UJ, I7d	J, I7d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. The interference check sample percent recovery value is <50%.	R, I2	J-, I2
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. The interference check sample percent recovery value is ≥50% and <80%.	UJ, I2a	J-, I2a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	11. The interference check sample percent recovery value is >120%.	N/A	J+, I2b

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Yes No N/A (Check One)				Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	12. The interference check sample was not analyzed with the samples.	R, I2c	R, I2c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	13. The sample result is $\leq 5X$ the concentration of the related analyte in the method blank.	N/A	U, I4
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	14. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15. The sample result is $\leq 5X$ the concentration of the related analyte in the instrument blank and continuing calibration blank.	N/A	U, I4b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	16. Continuing calibration blanks were not analyzed at the appropriate method frequency.	UJ, I4c	J, I4c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	17. The sample result is $\leq 5X$ the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank.	N/A	U, I4d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	18. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	19. The associate matrix spike recovery was $<10\%$ . Follow the external laboratory limits located within the associated data package.	R, I6	R, I6
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	20. The associated matrix spike recovery was below the Lower Acceptance Limit (LAL) but $>10\%$ . Follow the external laboratory limits located within the associated data package.	UJ, I6a	J-, I6a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	21. The associated matrix spike recovery was above the Upper Acceptance Limit (UAL). Follow the external laboratory limits located within the associated data package.	UJ, I6b	J+, I6b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	22. Required matrix spike information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. If LCS information is present, do not reject. Qualify data based on LCS information.	R, I6c	R, I6c

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Yes No N/A (Check One)				Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	23. The sample and the duplicate sample results were $\leq 5X$ the RL and the duplicate RPD was $>20\%$ for water samples and $>35\%$ for soil samples.	UJ, I10a	J, I10a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	24. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	25. The LCS percent recovery was $<10\%$ . Follow the external laboratory limits located within the associated data package.	R, I12	R, I12
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	26. The LCS percent recover was $<$ the LAL but $>10\%$ . Follow the external laboratory limits located within the associated data package.	UJ, I12a	J-, I12a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	27. The LCS percent recovery was $>$ the UAL. Follow the external laboratory limits located within the associated data package.	N/A	J+, I12b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	28. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	29. Duplicate, dilution, or reanalysis	UJ, I88	J, I88
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	30. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	31. Qualification of data via data validation does 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 (no qualification)

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**ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION****5167-3****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	UJ	J	I10a	The sample and the duplicate sample results were $\geq 5X$ the RL and the duplicate RPD was $>20\%$ for water samples and $>35\%$ for soil samples.
2	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.
3	R	R	I12	The LCS percent recovery was $<10\%$ . Follow external laboratory limits located within the associated data package.
4	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.
5	N/A	J+	I12b	The LCS percent recovery was $>$ Upper Acceptance Limit (UAL). Follow the external laboratory limits located within the associated data package.
6	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.

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
7	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 and/or under advisement by the project chemist.
8	R	J-	I2	Metals interference check sample percent recovery value is <50%.
9	UJ	J-	I2a	Metals interference check sample percent recovery value is ≥50% and <80%.
10	N/A	J+	I2b	Metals interference check sample percent recovery value is >120%.
11	R	R	I2c	Metals interference check sample was not analyzed with the samples.
12	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.
13	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.
14	N/A	U	I4b	The sample result is ≤5X the concentration of the related analyte in the instrument blank and continuing calibration blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.
15	UJ	J	I4c	Continuing calibration blanks were not analyzed at the appropriate method frequency.
16	N/A	U	I4d	The sample result is ≤5X the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.
17	R	R	I4e	Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
18	R	R	I6	The associated matrix spike recovery was <10%. Follow the external laboratory limits located within the associated data package.
19	UJ	J-	I6a	The associated matrix spike recovery was < the LAL but > 10%. Follow the external laboratory limits located within the associated data package.

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
20	UJ	J+	I6b	The associated matrix spike recovery was > the UAL. Follow the external laboratory limits located within the associated data package.
21	R	R	I6c	Required matrix spike information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. If LCS information is present, do not Reject. Qualify data based on LCS information.
22	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.
23	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.
24	UJ	J	I7c	The Initial Calibration Verification (ICV) and/or Continuing Calibration Verification (CCV) were recovered outside the method specific limits.
25	UJ	J	I7d	The ICV and/or CCV were not analyzed at the appropriate method frequency.
26	R	R	I7f	Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.
27	UJ	J	I88	Duplicate, dilution, or reanalysis.
28	UJ	J-	I9	The extraction holding time was exceeded by <2X the published method for holding times.
29	R	J-	I9a	The extraction holding time was exceeded by >2X the published method for holding times.
30.	R	R	I9b	The affected analytes are regarded as rejected because the analytical holding time was exceeded.
31.	U	J, NQ	U_LAB, J_LAB, NQ	Qualification of data via data validation does not occur based on Quality Control requirements in this procedure. Adhere to the external laboratory qualifier found within the Form I analytical data summary sheets generated by the external laboratory.

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Title: <b>Routine Validation of LC/MS/MS</b>	No.: SOP-5167	Page 11 of 11
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Section 16.1 Attachment 3 - Procedure Change Request

Procedure Change Request				
Section #1- Type of Request				
Manual/Procedure No. (if known): SOP-5167			Revision: 0	
Title: Routine Validation of General Chemistry Analytical Data				
Detailed description of requested change (Attach additional sheets if needed. Number additional sheets):				
New Procedure				
Requestor Signature: <i>Ellena Martinez</i>		Print Name: Ellena Martinez	Phone: 665-2751	Date: 4/18/08
Section #2- Procedure Owner Supervisor Approval For Processing				
<input checked="" type="checkbox"/> New Procedure	<input type="checkbox"/> Major Revision	<input type="checkbox"/> Minor Revision	<input type="checkbox"/> Special Procedure	
<input type="checkbox"/> IPC	<input type="checkbox"/> Deactivation	<input type="checkbox"/> Cancellation	<input type="checkbox"/> IPC Rollup	
<input checked="" type="checkbox"/> Approved <input type="checkbox"/> Disapproved (Return to originator)			Priority: High	
Procedure Owner Supervisor Signature: <i>Nita Patel</i>		Print Name: Nita Patel		Date: 4/21/08
Section #3 - Review and Concurrence				
IPC # N/A	IPCs Incorporated: N/A		Affected Pages: N/A sc 6/30/08	
Other affected facilities or N/A: N/A Obtain Concurrence all facilities/organizations affected by this change				
Review and Concurrence: Review organizations (N/A if not required), document additional review organizations, if needed on continuation sheet. CSE approval required for all technical procedures except minor revisions, IPC Rollup, and non-AB related cancellations/deactivations. CSE approval always required for changes affecting safety basis steps.				
Department:	Print Name:	Signature:	Date:	
WES-EDA	Bill Hardesty	<i>Bill Hardesty</i>	4/21/08	
WES-EDA	Craig Eberhart	<i>Craig Eberhart</i>	4/21/2008	
QA-IQ	Laura Ortega	<i>Laura Ortega</i>	5/19/08	
CT-DTS	Pam Flores	<i>See Below</i>		
CSE USQ Number (as applicable): N/A # 4/21/08 Val Rhodes, 699-4529	ADC: <input checked="" type="checkbox"/> Unclassified <input type="checkbox"/> OOU <input type="checkbox"/> UCNI <input type="checkbox"/> Classified	Print Name: S. Milln	Signature: <i>S. Milln</i>	
Section #4 - Final Approval By Procedure Owner				
Validation Required? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	Document is Authorized to serve as Part 1 of the IWD <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No		Periodic Review Requirements Satisfied? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	
Training Required: <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	<input type="checkbox"/> Classroom/Briefing <input type="checkbox"/> On the Job	<input type="checkbox"/> Just-in-Time <input checked="" type="checkbox"/> Required Reading	<input type="checkbox"/> Hold for Completion of Training <input type="checkbox"/> Release Procedure to field	
Approval Signature: <i>Nita Patel</i>	Print Name: Nita Patel	Z Number: 153003	Date: 4/21/08	Phone: 665-9273

Training Review Completed Cause # assigned  
Pam Flores 5/20/08 46143