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Waste & Environmental Services

Standard Operating Procedure

for **ROUTINE VALIDATION OF HIGH EXPLOSIVE (HE) 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

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1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine Explosives 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 Explosives 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 Organic 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 Organic 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, High Explosive (HE) 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 High Explosive (HE) 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 5164-1 Data Validation Cover Sheet (1 page)
Attachment 2 5164-2 High Explosive (HE) Analytical Data Validation Checklist (3 pages)
Attachment 3 5164-3 Guidance for the Qualifier and Reason Code Application (4 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		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

5164-1

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):

- | | | | |
|--|--|---|--|
| <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: _____

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LOS ALAMOS
Environmental Restoration Project

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ATTACHMENT 2: HIGH EXPLOSIVE (HE) ANALYTICAL DATA VALIDATION CHECKLIST

5164-2

High Explosive (HE) Analytical Data Validation Checklist

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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"/>	1. The holding time was >1 and ≤2 times the applicable holding time requirement.	UJ, H9	J-, H9
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. The holding time was >2 times the applicable holding time requirement.	R, H9a	J-, H9a
<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, H9b	R, H9b
<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, H7	J, H7
<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 multi-point calibration correlation coefficient is <0.995.	UJ, H7a	J, H7a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. The Initial Calibration Verification (ICV) and/or Continuing Calibration Verification (CCV) were recovered outside the method limits.	UJ, H7c	J, H7c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. The ICV and/or CCV were not analyzed at the appropriate method frequency.	UJ, H7d	J, H7d
<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, H7f	R, H7f
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. The sample result is ≤5 times the concentration of the related analyte in the method blank.	N/A	U, H4
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5x.	N/A	J+, H4a

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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"/>	11. The sample result is ≤ 5 times the concentration of the related analyte in the trip blank, rinsate blank, and/or equipment blank.	N/A	U, H4d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	12. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, H4e	R, H4e
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	13. The analyte RT shifted by more than 0.05 minutes from the mid-level standard of the initial calibration.	R, H0	J, H0
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	14. Analyte is positively confirmed but outside the IS retention time window; however, spectral matches must be provided (hexp – diode array detector).	N/A	J+, H0a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15. Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, H0b	R, H0b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	16. The surrogate is $< 10\%R$. Follow the external laboratory limits located within the associated data package.	R, H3	J-, H3
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	17. The surrogate is $<$ the Lower Acceptance Limit (LAL) but $\geq 10\%R$. Follow the external laboratory limits located within the associated data package.	UJ, H3a	J-, H3a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	18. The surrogate %R value is $>$ the Upper Acceptance Limit (UAL). Follow the external laboratory limits located within the associated data package.	N/A	J+, H3b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	19. At least one surrogate is $>$ the UAL and one surrogate is $<$ the LAL. Follow the external laboratory limits located within the associated data package.	UJ, H3c	J, H3c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	20. Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, H3d	R, H3d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	21. The LCS percent recover was $< 10\%$. Follow the external laboratory limits located within the associated data package.	R, H12	R, H12

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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"/>	22. The LCS percent recovery was < the LAL but >10%. Follow the external laboratory limits located within the associated data package.	UJ, H12a	J-, H12a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	23. The LCS percent recovery was > the UAL. Follow the external laboratory limits located within the associated data package.	N/A	J+, H12b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	24. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, H12c	R, H12c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	25. The analyte was not confirmed on a second dissimilar column [R] or diode array spectrum does not match the library (U).	N/A	R, U, H8
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	26. The second dissimilar column documentation is missing or diode array spectrums are missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, H8a	R, H8a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	27. Duplicate, dilution, or reanalysis.	UJ, H88	J, H88
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	28. The affected analytes have elevated detection limits and may not meet project DQOs because the sample was diluted without any target analytes identified due to matrix interference. Qualify as Reject if the analytical laboratory cannot provide proof for cleanup or matrix interference.	UJ, R, H15	R, H15
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	29. 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	30. The LANL project chemist identified quality deficiencies in the reported data that requires further qualification. This code can only be used and/or under advisement by the LANL project chemist.	UJ, R, H19	J, R, H19

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ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION**5164-3****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	R	J	H0	The analyte RT shifted by more than 0.05 minutes from the mid-level standard of the initial calibration. Reject nondetects for HPLC.
2	N/A	J	H0a	Analyte is positively confirmed but outside the retention time window; however, spectral matches must be provided (hexp – diode array detector).
3	R	R	H0b	Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
4	R	R	H12	The LCS percent recovery was <10%. Follow external laboratory limits located within the associated data package.
5	UJ	J-	H12a	The LCS percent recovery was < the Lower Acceptance Limit (LAL) but >10%. Follow external laboratory limits located within the associated data package.
6	N/A	J+	H12b	The LCS percent recovery was > than the Upper Acceptance Limit (UAL). Follow the external laboratory limits located within the associated data package.
7	R	R	H12c	The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
8	UJ, R	R	H15	The affected analytes have elevated detection limits and may not meet project DQOs because the sample was diluted without any target analytes identified due to matrix interference. Qualify as Reject if the analytical laboratory cannot provide proof for cleanup or matrix interference.
9	UJ, R	J, R	H19	The LANL project chemist identified quality deficiencies in the reported data that requires further qualification. This code can ONLY be used and/or under advisement by the project chemist.
10	R	J-	H3	The surrogate is <10%R, which indicates the potential for a severely low bias in the results. Follow external laboratory limits located within the associated data package.
11	UJ	J-	H3a	The surrogate is < the LAL but ≥10%R, which indicates the potential for a low bias in the results. Follow the external laboratory limits located within the associated data package.
12	N/A	J+	H3b	The surrogate %R value is > the UAL, which indicates a potential for a high bias in the results and a potential for false positive results. Follow the external laboratory limits located within the associated data package.
13	UJ	J	H3c	At least one surrogate is > the UAL and one surrogate is < the LAL, which indicates a greater than normal degree of uncertainty in the result. Follow external laboratory limits located within the associated data package.
14	R	R	H3d	Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
15	N/A	U	H4	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.
16	N/A	J	H4a	The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5X.

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
17	H4d	U	H4d	The sample result is $\leq 5X$ 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.
18	R	R	H4e	Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
19	UJ, R	J	H7	The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.
20	UJ	J	H7a	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 .
21	UJ	J	H7c	The Initial Calibration Verification (ICV) and/or Continuing Calibration Verification (CCV) were recovered outside the method-specific limits.
22	UJ	J	H7d	The ICV and/or CCV were not analyzed at the appropriate method frequency.
23	R	R	H7f	Required calibration information is missing or Samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.
24	N/A	R, U	H8	The analyte was not confirmed on a second dissimilar column or diode array spectrums do not match library.
25	UJ	J	H88	Duplicate, dilution, or reanalysis.
26	R	R	H8a	The required second dissimilar column or diode array documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
27	UJ	J-	H9	The extraction/analytical holding time was exceeded by $< 2X$ the published method for holding times.
28	R	J-	H9a	The extraction/analytical holding time was exceeded by $> 2X$ the published method for holding times.

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
29	R	R	H9b	The affected analytes are regarded as rejected because the analytical holding time was exceeded.
30	U	J, NQ	U_LAB, J_LAB, NQ	Qualification of data via data validation did not occur based on Quality Control requirements in this procedure. Adhere to the external laboratory qualifiers found with the Form 1 analytical data summary sheets generated by the external laboratory.

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