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Los Alamos NATIONAL LABORATORY EST. 1943

Waste & Environmental Services

Standard Operating Procedure

for ROUTINE VALIDATION OF LC/MS/MS HIGH EXPLOSIVE ANALYTICAL DATA

APPROVAL SIGNATURES:

Subject Matter Expert:	Organization	Signature	Date
Bill Hardesty	WES-EDA	Bill Hardesty	4/24/2008
Quality Assurance Specialist:	**Organization**	**Signature**	**Date**
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Responsible Line Manager:	**Organization**	**Signature**	**Date**
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1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine explosives by LC/MS/MS organics 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, LC/MS/MS High Explosive Analytical Data Validation Checklist, during data validation.
	3.	Refer to the following attachments for additional guidance: • Attachment 3, Guidance for the Qualifier and Reason Code Application;

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 LC/MS/MS High Explosive 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 5168-1 Data Validation Cover Sheet (1 page)

Attachment 2 5168-2 LC/MS/MS High Explosive Analytical Data Validation Checklist (4 pages)

Attachment 3 5168-3 Guidance for the Qualifier and Reason Code Application (11 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	7/1/08	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.

ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

5168-1

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: _____

SOP-5168, Revision 0.0	LOS ALAMOS Environmental Restoration Project

ATTACHMENT 2: LC/MS/MS HIGH EXPLOSIVE ANALYTICAL DATA VALIDATION CHECKLIST

5168-2

LC/MS/MS High Explosive Analytical Data Validation Checklist

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Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	1. The IS retention time has shifted by more than 30 seconds.	R, UJ, HE0	J, HE0
☐	☐	☐	2. Required IS retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, HE0b	R, HE0b
☐	☐	☐	3. The quantitating IS area count is <25% of the expected value, which indicates increased potential for false negative results and other possible problems with sample quantitation. Follow the method-specific windows.	R, HE1a	J, HE1a
☐	☐	☐	4. The IS area count for the quantitating IS is <70% but >25% of the average of that obtained from the calibration standards.	UJ, HE1b	J+, HE1b
☐	☐	☐	5. The IS area count for the quantitating IS is >130% of the average of that obtained from the calibration standards.	UJ, HE1c	J-, HE1c
☐	☐	☐	6. Required IS information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, HE1d	R, HE1d
☐	☐	☐	7. The surrogate is <10%R. Follow the external laboratory limits.	R, HE3	J-, HE3
☐	☐	☐	8. The surrogate is < the Lower Acceptance Limit but ≥10% recovery. Follow the external laboratory limits.	UJ, HE3a	J-, HE3a
☐	☐	☐	9. The surrogate %R value is > the Upper Acceptance Limit. Follow the external laboratory limits.	N/A	J+, HE3b

Yes	No	N/A (Check One)		Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
☐	☐	☐	10. At least one surrogate is > the Upper Acceptance Limit and one surrogate is < the Lower Acceptance Limit. Follow the external laboratory limits.	UJ, HE3c	J, HE3c
☐	☐	☐	11. Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, HE3d	R, HE3d
☐	☐	☐	12. The sample result is ≤5 times the concentration of the related analyte in the method blank.	N/A	U, HE4
☐	☐	☐	13. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5x.	N/A	J, HE4a
☐	☐	☐	14. The sample result is ≤5 times the concentration of the related analyte in the trip blank, rinsate blank, and/or equipment blank.	U, HE4d	N/A
☐	☐	☐	15. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, HE4e	R, HE4e
☐	☐	☐	16. The absence of sample carry-over must be determined and verified.	N/A	R, N, HE4f
☐	☐	☐	17. The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.	UJ, HE7	J, HE7
☐	☐	☐	18. The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD criteria and/or the associated multipoint calibration correlation coefficient is less < 0.99.	UJ, R, HE7a	J, HE7a
☐	☐	☐	19. The affected analytes were analyzed with a RRF of <0.05 in the initial calibration and/or CCV.	UJ, R, HE7b	J, HE7b
☐	☐	☐	20. The ICV and/or CCV were recovered outside the method limits.	UJ, R, HE7c	J, HE7c
☐	☐	☐	21. The ICV and/or CCV were not analyzed at the appropriate method frequency.	UJ, R, HE7d	J, HE7d
☐	☐	☐	22. Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.	R, HE7f	R, HE7f

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	23. The mass spectral documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, HE8a	R, HE8a
☐	☐	☐	24. The holding time was >1 and ≤2 times the applicable holding time requirement.	UJ, HE9	J-, HE9
☐	☐	☐	25. The holding time was >2 times the applicable holding time requirement.	R, HE9a	J-, HE9a
☐	☐	☐	26. The LCS percent recovery was <10%. Follow the external laboratory limits.	R, HE12	J-, HE12
☐	☐	☐	27. The LCS percent recovery was < the Lower Acceptance Limit but >10%. Follow the external laboratory limits.	UJ, HE12a	J-, HE12a
☐	☐	☐	28. The LCS percent recovery was > the Upper Acceptance Limit. Follow the external laboratory limits.	N/A	J+, HE12b
☐	☐	☐	29. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, HE12c	R, HE12c
☐	☐	☐	30. The MS/MSD percent recovery was <10%.	R, HE12d	R, HE12d
☐	☐	☐	31. The MS/MSD percent recovery was >10% but <70%.	UJ, HE12e	J, HE12e
☐	☐	☐	32. The MS/MSD percent recover was >130%.	N/A	J+, HE12f
☐	☐	☐	33. The MS/MSD relative percent difference was >30%.	UJ, HE12g	J, HE12g
☐	☐	☐	34. The affected analytes are considered suspect 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 matrix interference.)	UJ, R, HE15	R, HE15
☐	☐	☐	35. The sample was diluted because target analytes were > the initial verification calibration.	UJ, HE15a	J, HE15a
☐	☐	☐	36. The Contract Required Detection Limit Check Standard (CRI) sample did not pass method acceptance criteria.	UJ, R, HE16	J, HE16

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	**37. The required CRI sample information is missing. Contact the SMO or external laboratory for information.**	**R, HE16c**	**R, HE16c**
☐	☐	☐	**38. 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, HE19**	**J, R, HE19**
☐	☐	☐	**39. Duplicate, dilution, or reanalysis.**	**UJ, HE88**	**J, HE88**

ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

5168-3

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, UJ	J	HE0	The IS retention time has shifted by >30 seconds.
2	R	R	HE0b	Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO and external laboratory for information.
3	R	J	HE1a	The quantitating IS area count is <25% of the expected value, which indicates increased potential for false negative results and other possible problems with sample quantitation. Follow the method specific windows. Qualify data as R if the IS area count is <25%.
4	UJ	J+	HE1b	If the internal standard was used for quantification and its area count is <70% but >25% of the average of that obtained from the calibration standards, qualify all associated detects as J+ and all associated non-detects as UJ.
5	UJ	J-	HE1c	The internal standard area counts must not vary by >70% to 130% from the average of those obtained from the calibration standards or from the mid-level calibration standard. If the internal standard was used for quantification and its area count is >130% of the average of that obtained from the calibration standards, qualify all associated detects as J- and all associated non-detects as UJ.

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
6	**R**	**R**	**HE1d**	**Required IS information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**
7	**R**	**J-**	**HE3**	**The surrogate is <10% recovery, which indicates the potential for a severely low bias in the results. Follow the external laboratory limits. Qualify non-detected results as R and detected results as J-.** **Also, if an initial dilution was performed on any sample and surrogate recovery is <10% recovery and all results are non-detect, qualify all sample results as R.**
8	**UJ**	**J-**	**HE3a**	**The surrogate is < the Lower Acceptance Limit but ≥10% recovery, which indicates the potential for a low bias in the results. Follow the external laboratory limits. Qualify non-detected results as UJ and detected results as J-.** **Also, if an initial dilution was performed on any sample and at least one surrogate recover is < the Lower Acceptance Limit, but ≥10%, or all surrogate recoveries are <10% and the results for one or more compounds are > the PQL, qualify non-detected results as UJ and detected results as J-.**
9	**N/A**	**J+**	**HE3b**	**The surrogate % recovery value is > the Upper Acceptance Limit, which indicates the potential for a high bias in the results and a potential for false positive results. Follow the external laboratory limits.**
10	**UJ**	**J**	**HE3c**	**At least one surrogate is > the Upper Acceptance Limit and one surrogate is < the Lower Acceptance Limit, which indicates a > normal degree of uncertainty in the result. Follow the external laboratory limits.**
11	**R**	**R**	**HE3d**	**Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.** **Sample and blank surrogate recoveries must be within limits specified by the laboratory. Surrogate compound recoveries shall be calculated using the procedure described in SW-846 EPA Method 8000B. Reported recoveries shall be accompanied by the applicable acceptance limits.** **Results from spiked or replicate QC samples that have surrogate recoveries <10% cannot be used to evaluate associated sample results. Associated sample results**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
				should be qualified for lack of accuracy and/or precision data, as applicable. **In the case of a blank analysis with surrogates out of specification, special consideration must be given to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical.** **If one or more samples in the batch show acceptable surrogate recoveries, the blank problem may be considered to be an isolated occurrence. However, even if this judgment allows some us of the affected data, analytical problems remain that must be corrected by the laboratory.** **If the surrogate recovery acceptance criteria were not reported in the data package, request amended data from the laboratory.** **If, based on professional judgment, the laboratory's internal acceptance criteria are excessively wide or biased, notify the program manager.**
12	**U**	**N/A**	**HE4**	**The sample result is ≤5 times the concentration of the related analyte in the method blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.** **The concentration of each target analyte found in the blank must be < the associated MDL. The sample results must not be corrected by subtracting any blank value. If QC problems exist with any blank, all data associated with the case must be carefully evaluated to determine whether there is an inherent variability in the data for the case or if the problem is an isolated occurrence not affecting other data.** **If a compound found in a blank is also found in a field sample, qualify the sample result for that compound in accordance with the scenarios given below.** **If gross contamination exists, qualify results for all affected compounds as R due to interference.** **If inordinate numbers of other target compounds are found at low levels in a blank, discuss the presence of those compounds in the data validation report as it may be indicative of a problem at the laboratory.** **Attachment 5 provides examples of application of the blank qualification guidelines.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
				Certain circumstances may warrant deviations from these guidelines.
13	**N/A**	**J**	**HE4a**	**The affected analytes are considered estimates and biased high because this analyte was identified in the method blank but was >5x.**
14	**U**	**N/A**	**HE4d**	**The sample result is ≤5 times the concentration of the related analyte in the trip blank, rinsate blank, and equipment blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.**
15	**R**	**R**	**HE4e**	**Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**
16	**N/A**	**R, N**	**HE4f**	**The absence of sample carry-over must be determined and verified. If examination of the run logs indicates that any samples in the analytical run of interest required dilution and there is no documentation of a rinse or blank analysis immediately following the original undiluted analysis, then sample carry-over may be suspected in the subsequent sample.** **If any target analyte found in the sample requiring dilution exceeded the high calibration standard and was also found in the following sample at a concentration <5x the PQL, qualify the result for that analyte in the second sample as R.** **If no data are available for the sample that required dilution, the laboratory has not documented that carry-over was evaluated, and any analyte was also found in the following sample as a concentration <5x the PQL, qualify the result for that analyte in the second sample as N.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
17	**UJ**	**J**	**HE7**	**The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.** **LC/MS/MS instrument calibration shall be performed using a minimum of five (5) calibration standards. The lowest point of the curve must be at or below the reporting limit. If calibration curves are used, five (5) standards are required for a linear (first order) calibration model, six (6) standards are required for a quadratic (second order) model, and seven (7) standards are required for a third order polynomial. Higher order curves should not normally be used. If the laboratory uses a higher order equation to establish a calibration curve, it should be evaluated for the appropriate application. If an insufficient number of calibration standards was used, the PQLs were incorrect, or all points were not analyzed within a 24-hour period, qualify all associated detects as J and all associated non-detects as UJ.**
18	**UJ, R**	**J**	**HE7a**	**The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD or r^2.** **If the %RSD for any target analyte is >20% but ≤40%, qualify all associated detects as J and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as UJ.** **If the %RSD for any target analyte is >40% but ≤60%, qualify all associated detects as J and all associated non-detects as UJ.** **If the %RSD for any target analyte is >60%, qualify all associated detects as J and all associated non-detects as R.** **If the r^2 for any target analyte is <0.99 but ≥0.90, qualify all associated detects as J and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as UJ.** **If the r^2 for any target analyte is >0.90 but ≤0.80, qualify all associated detects as J and all associated non-detects as UJ.** **If the r^2 for any target analyte is <0.80, qualify all associated detects as J and all associated non-detects if the intercept for any target analyte is positive and >3x the intercept J+ as R.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
19	**UJ, R**	**J**	**HE7b**	**The affected analytes were analyzed with a RRF of <0.05 in the initial calibration and/or CCV.** **If the average RF for any target analyte is < the specified minimum RF, or <0.05 if no minimum is specified, qualify all associated detects as J and all associated non-detects as UJ if the RF is ≥0.01 and as R if the RF is <0.01.**
20	**UJ, R**	**J**	**HE7c**	**The ICV and/or CCV were recovered outside the method limits. The %D between the ICV and CCV standard concentrations and their true values shall be calculated according to the formula in Attachment 4, and must be ≤20%. The evaluation of CCV data applies to all CCVs that bracket samples of interest. If the %D was reported with the wrong sign (e.g., +%D for negative bias), document the occurrence in the data validation report and assess any infractions using the correct sign.** **1. If the %D between a measured ICV and/or CCV concentration and its true value for any analyte is >20%, qualify all associated detects as J+.** **2. If the %D between a measured ICV and/or CCV concentration and is true value for any analyte is >20% but ≤40% and negative (low bias), qualify all associated detects as J- and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as UJ.** **3. If the %D between a measured ICV and/or CCV concentration and its true value for any analyte is >40% but ≤60% and negative, qualify all associated detects as J- and all associated non-detects as UJ.** **4. If the %D between a measured ICV and/or CCV concentration and its true value for any analyte is >60% and is negative, qualify all associated detects as J- and all associated non-detects as R.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
21	**UJ, R**	**J**	**HE7d**	**The ICV and/or CCV were not analyzed at the appropriate method frequency.** **An ICV standard is analyzed immediately following an initial calibration. For high explosive analysis, the ICV standard analysis results are not required to be reported in the data package unless the samples in the SDG were analyzed after the initial calibration but before a CCV standard analysis was performed. In this case, the ICV %D is assessed according to the calibration verification criteria described below for the associated samples. If a CCV is analyzed prior to samples and ICV data are also reported in the package, both the ICV %D and the appropriate CCV %D are to be assessed as described below. If both ICV %D and CCV %D infractions occur, the worst infraction should be evaluated for result qualification.** **A CCV must be analyzed in the following instances:** • **at the beginning of each analytical run;** • **at least once every 10 samples; and** • **at the end of each analytical run.** **If multiple CCVs were analyzed to obtain a passing CCV, the calibration is not verified and the calibration frequency is not met.** **If the ICV and CCV standards were not analyzed at the proper frequency, or if either a required ICV or CCV was not analyzed, or if all target compounds were not present in any ICV or CCV standard, qualify all associated detects as J and all associated non-detects as UJ.** **If all required ICVs and CCVs were not analyzed, qualify all associated detects as J and all associated non-detects as R.**
22	**R**	**R**	**HE7f**	**Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.**
23	**R**	**R**	**HE8a**	**The mass spectral documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**
24	**UJ**	**J-**	**HE9**	**The extraction/analytical holding time is exceeded by <2x the published method for holding times.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
25	**R**	**J-**	**HE9a**	**The extraction/analytical holding time was exceeded by more than 2x the published method for holding times.**
26	**R**	**J-**	**HE12**	**An LCS should be analyzed at a frequency of once per data package, once per matrix, or once per 20 analytical samples, whichever is most frequent.** **The LCS must meet all sample acceptance criteria and all method-specific LCS requirements. The LCS for high explosives must meet laboratory-derived acceptance criteria. If surrogate and IS recovery acceptance criteria are not met for the LCS analysis, the LCS must be reanalyzed. If the recovery acceptance criteria are not reported in the analytical data package recovery limits of 70% to 130% should be used as the criteria.** **If, based on professional judgment, the laboratory's internal acceptance criteria are excessively wide or acceptable recoveries are significantly biased, notify the program manager.** **The LCS percent recovery was <10%. Qualify detected results as J- and not detected results as R.**
27	**UJ**	**J-**	**HE12a**	**The LCS percent recovery was < the Lower Acceptance Limit but >10%. Follow the external laboratory limits. Qualify detected results as J- and not detected results as UJ.**
28	**N/A**	**J+**	**HE12b**	**The LCS percent recovery was > the Upper Acceptance Limit. Follow the external laboratory limits. Qualify detected results as J+.**
29	**R**	**R**	**HE12c**	**The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or the external laboratory for information.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
30	**R**	**R**	**HE12d**	**The MS/MSD percent recovery was <10%. The MS/MSD data shall not be used to evaluate associate field sample results unless the MS/MSD sample was from the same client and of similar matrix. If the acceptance criteria are not reported, recovery limits are 70% to 130%.** **The MS and MSD %R must be within the limits unless the sample concentration is >4X the spike concentration. The MS and MSD results may be used in conjunction with other QC results to determine the need for qualification of the data. An effort to determine to what extent the results of the MS/MSD affect the associated data should first be made. This determination should be made considering the MS/MSD sample matrix, the surrogate and internal standard recoveries, and the LCS results.** **Professional judgment should be used to determine if MS/MSD failure warrants qualification of only the results for the failed compounds or if the compounds associated with the failed MS compound are affected. Generally, unless evidence exists to warrant qualification of other compounds, only the compounds in the MS spiking mixture shall be qualified.** **If the surrogate, internal standard, and LCS recoveries are within the required acceptance criteria and either the MS or MSD recovery for any target analyte is <10%, qualify results as R.**
31	**UJ**	**J**	**HE12e**	**If the MS/MSD percent recovery was >10%, but <70%, qualify all detects as J and all non-detects as UJ.**
32	**N/A**	**J+**	**HE12f**	**If the MS/MSD percent recovery was >130%, qualify all associated detects as J+.**
33	**J**	**UJ**	**HE12g**	**If the MS/MSD relative percent difference was >30%, and the acceptance criteria are not reported, recovery limits of 70% to 130% and an RPD of ≤30% should be used as the criteria. For solid and waste samples, it may be appropriate to accept an RPD of up to 40% based on professional judgment.**
34	**UJ, R**	**R**	**HE15**	**If the affected analytes are considered suspect 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 matrix interference.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
35	**UJ**	**J**	**HE15a**	**The Practical Quantitation Limits must be adjusted to reflect all sample dilutions, concentrations, splits, clean-up activities, and dry weight factors that are not accounted for by the method. Samples must be diluted and reanalyzed when any analyte exceeds the calibration range. Data from the original sample analysis should be included when any sample requires dilution due to one or more analytes exceeding the calibration range. The original undiluted results document the actual MDLs for non-detects.** **If the PQLs have not been properly adjusted, request an amended report from the laboratory. If an initial dilution was required because of expected high concentrations of non-target analytes or because one or more target analytes were expected to greatly exceed the instrument working range and the laboratory was not able to analyze the undiluted sample, note the dilution and elevated MDLs in the data validation report.** **If any target analyte exceeded the calibration range and the original undiluted sample result was reported, qualify all detects from the undiluted analysis that exceeded the calibration range as J.** **If any target analyte exceeded the calibration range and the sample was diluted and reanalyzed and the diluted sample data were reported, qualify all non-detects from the diluted analysis as UJ.** **If any target analyte exceeded the calibration range and the original undiluted sample analysis was not reported, request this information from the laboratory.** **If data from the original sample analysis are unavailable, refer to HEXP3 and HEXP3a for assessment of initially diluted samples with low surrogate recovery.** **The laboratory shall strive to make dilutions in such a way that the final concentration is measured in the mid-range of the calibration curve, and that results are not reported from measurements below the lowest concentration standard. If the instrument response (reported result/dilution factor) for a diluted sample is < that of the lowest concentration standard, qualify all associated detects from the diluted analysis as J.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
36	**UJ, R**	**J**	**HE16**	**The Contract Required Detection Limit check standard (CRI) sample did not pass method-acceptance criteria.** **CRI analysis recoveries for high explosives analysis must be within limits specified by the Laboratory. If acceptance criteria are not reported, the recovery acceptance range shall be 70% to 130%.** **1. If frequency criteria were not met, qualify all detects <5X the PQL as J and all non-detects as UJ.** **2. If the recovery is > the upper acceptance limit, qualify all associated detects <5X the PQL as J+.** **3. If the recovery is < the lower acceptance limit but ≥30%, qualify all associated detects <5X the PQL as J- and all associated non-detects as UJ.** **4. If the recovery is <30%, qualify all associated detects <5X the PQL as J- and all associated non-detects as R.**
37	**R**	**R**	**HE16c**	**The required CRI sample information is missing. Contact the SMO or the external laboratory for information.**
38	**UJ, R**	**J, R**	**HE19**	**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.**
39	**UJ**	**J**	**HE88**	**Duplicate, dilution, or reanalysis.**

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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-5168	Revision: 0

Title: Routine Validation of LC/MS/MS High Explosive Analytical Data

Detailed description of requested change (Attach additional sheets if needed. Number additional sheets):

New Procedure

Requestor Signature: Ellena Martinez	Print Name: Ellena Martinez	Phone: 665-2751	Date: 4/18/08

Section #2-Procedure Owner Supervisor Approval For Processing

☑ New Procedure ☐ Major Revision ☐ Minor Revision ☐ Special Procedure

☐ IPC ☐ Deactivation ☐ Cancellation ☐ IPC Rollup

☑ Approved ☐ Disapproved (Return to originator) Priority: High

Procedure Owner Supervisor Signature: Nita Patel	Print Name: Nita Patel	Date: 4/21/08

Section #3 -Review and Concurrence

IPC # N/A	IPCs Incorporated: N/A	Affected Pages: N/A 5/2/08

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	Bill Hardesty	4/21/2008
WES-EDA	Craig Eberhart	Craig Eberhart	4/24/2008
QA-IQ	Laura Ortega	Laura Ortega	4/30/08
CT-DTS	Pam Flores	Pam Flores	5/7/08

CSE USQ Number (as applicable): NA per 4/24/08 Val Rhodes, 699-4529

ADC: ☒ Unclassified ☐ OUO ☐ UCNI ☐ Classified

Print Name S. Millin Signature

Section #4 - Final Approval By Procedure Owner

Validation Required? ☐ Yes ☑ No	Document is Authorized to serve as Part 1 of the IWD ☐ Yes ☑ No	Periodic Review Requirements Satisfied? ☑ Yes ☐ No

Training Required: ☑ Yes ☐ No ☐ Classroom/Briefing ☐ On the Job ☐ Just-in-Time ☑ Required Reading ☐ Hold for Completion of Training ☐ Release Procedure to field

Approval Signature: Nita Patel	Print Name: Nita Patel	Z Number: 153003	Date: 4/21/08	Phone: 665-9273

Training Review Completed - Course # assigned - 46000

PF - 5/7/08