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Waste & Environmental Services

Standard Operating Procedure

for **ROUTINE VALIDATION OF LC/MS/MS
PERCHLORATE ANALYTICAL DATA (SW-846 EPA
METHOD 6850)**

APPROVAL SIGNATURES:

Subject Matter Expert:	Organization	Signature	Date
Bill Hardesty	WES-EDA	Signature on file	4/29/08
Quality Assurance Specialist:	Organization	Signature	Date
Laura Ortega	QA-IQ	Signature on file	5/7/08
Responsible Line Manager:	Organization	Signature	Date
Craig Eberhart	WES-EDA	Signature on file	5/6/08

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1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating perchlorate 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 perchlorate 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 Perchlorate 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 LC/MS/MS Perchlorate 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 5191-1 Data Validation Cover Sheet (1 page)
Attachment 2 5191-2 LC/MS/MS Perchlorate Analytical Data Validation Checklist (3 pages)
Attachment 3 5191-3 Guidance for the Qualifier and Reason Code Application (12 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.

ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

5191-1

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 | |
- 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-5191, Revision 0.0

LOS ALAMOS
Environmental Restoration Project

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ATTACHMENT 2: LC/MS/MS PERCHLORATE ANALYTICAL DATA VALIDATION CHECKLIST

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LC/MS/MS Perchlorate 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 perchlorate RRT is outside the acceptance range of 0.98 to 1.02 seconds.	R, PE0	J, PE0
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. Required IS retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, PE0b	R, PE0b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. The IS area count is <25% of the expected value.	UJ, PE1a	J, PE1a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. The IS area count is <70% but >25% of the average of that obtained from the calibration standards.	UJ, PE1b	J, PE1b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. The IS area count is >130% of the average of that obtained from the calibration standards.	UJ, PE1c	J, PE1c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. Required IS information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, PE1d	R, PE1d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. The sample result is ≤ 5X the concentration of the related analyte in the method blank.	N/A	U, PE4
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5X.	N/A	J+, PE4a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. The sample result is ≤5X the concentration of the related analyte in the trip blank, rinsate blank, and/or equipment blank.	U, PE4d	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, PE4e	R, PE4e

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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 affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.	UJ, PE7	J, PE7
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	12. The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD criteria and/or the associated multipoint calibration correlation coefficient is <0.99.	UJ, R, PE7a	J, PE7a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	13. The ICV and/or CCV were recovered outside the method limits.	UJ, R, PE7c	J, PE7c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	14. The ICV and/or CCV were not analyzed at the appropriate method frequency.	UJ, R, PE7d	J, PE7d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15. Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.	R, PE7f	R, PE7f
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	16. The affected analyte is considered not detected because ion abundance ratios did not meet specifications.	N/A	R, PE8
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	17. The ion ratio documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	N/A	R, PE8a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	18. The holding time was >1 and ≤2 times the applicable holding time requirement.	UJ PE9	J-, PE9
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	19. The holding time was > 2 times the applicable holding time requirement.	R, PE9a	J-, PE9a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	20. The LCS percent recovery was <10%. Follow the external laboratory limits.	R, PE12	J-, PE12
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	21. The LCS percent recovery was < the Lower Acceptance Limit but >10%. Follow the external laboratory limits.	UJ, PE12a	J-, PE12a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	22. The LCS percent recovery was > the Upper Acceptance Limit. Follow the external laboratory limits.	N/A	J+, PE12b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	23. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, PE12c	R, PE12c

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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"/>	24. The MS/MSD percent recovery was <10%	R, PE12d	R, PE12d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	25. The MS/MSD percent recovery was >10% but <75%	UJ, PE12e	J, PE12e
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	26. The MS/MSD percent recovery was >125%.	N/A	J+, PE12f
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	27. The MS/MSD relative percent difference was >20%.	UJ, PE12g	J, PE12g
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	28. 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, PE15	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	29. The sample was diluted because target analytes were > the initial verification calibration.	UJ, PE15a	J, PE15a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	30. The Contract Required Detection Limit check standard (CRI) sample did not pass method-acceptance limits.	UJ, R, PE16	J, PE16
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	31. The Interference Check Sample was not within ±20% of the known value.	UJ, PE16a	J, PE16a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	32. The required CRI sample information is missing. Contact the SMO or external laboratory for information.	R, PE16c	R, PE16c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	33. 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, PE19	J, R, PE19
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	34. Duplicate, dilution, or reanalysis.	UJ, PE88	J, PE88

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ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

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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	R	J	PE0	The perchlorate RRT is outside the acceptance range of 0.98 to 1.02 seconds.
2	R	R	PE0b	Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
3	UJ	J	PE1a	This IS area count is <25% of the expected value. If the internal standard is used only as a Retention Time (RT) check (perchlorate analysis), the Relative Retention Time (RRT) of the internal standard must fall within the acceptance range of 0.98 to 1.02, and the internal standard recovery should be evaluated using the surrogate criteria. If recovery acceptance limits are not reported in the data package, recovery should be evaluated based on reported Matrix Spike acceptance limits.
4	UJ	J	PE1b	The internal standard area could 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. If the internal standard is used only as a RT check (perchlorate analysis), the RRT of the internal standard must fall within the acceptance range of 0.98 to 1.02, and the internal standard recovery should be evaluated using the surrogate criteria. If recovery acceptance limits are not reported in the data package, recovery should be evaluated based on reported Matrix Spike acceptance limits.
5	UJ	J	PE1c	If the internal standard 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. If the internal standard is used only as a RT check (perchlorate analysis), the RRT of the internal standard must fall within the acceptance range of 0.98 to 1.02, and the internal standard recovery should be evaluated using the surrogate criteria. If recovery acceptance limits are not reported in the data package, recovery should be

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
				evaluated based on reported Matrix Spike acceptance limits.
6	R	R	PE1d	Required Internal Standard information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
7	U	N/A	PE4	The sample result is $\leq 5X$ the concentration of the related analyte in the method blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.
8	N/A	J+	PE4a	The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was $>5X$.
9	U	N/A	PE4d	The sample result is $\leq 5X$ 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.
10	R	R	PE4e	Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
11	UJ	J	PE7	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 curve 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.
12	UJ, R	J	PE7a	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 $>15\%$ but $\leq 40\%$, 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 $\leq 60\%$, qualify all associated detects as J and all associated non-detects as UJ.

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
				<p>If the %RSD for any target analyte is >60%, qualify all associated detects as J and all associated non-detects as R.</p> <p>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.</p> <p>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.</p> <p>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 MDL, qualify all associated detects <3X the intercept as J+ as R.</p>
13	UJ, R	J	PE7c	<p>The ICV and/or CCV were recovered outside the method limits. The %D between the ICV and CCV standard concentrations and their true values must be $\leq 15\%$. 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.</p> <ol style="list-style-type: none"> 1. If the %D between a measured ICV and/or CCV concentration and its true value for any analyte is >15%, qualify all associated detects as J+. 2. If the %D between a measured ICV and/or CCV concentration and its true value for any analyte is >15% but $\leq 40\%$ 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 $\leq 60\%$ 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.

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
14	UJ, R	J	PE7d	<p>The ICV and/or CCV were not analyzed at the appropriate method frequency.</p> <p>An ICV standard is analyzed immediately following an initial calibration. 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 %D and CCV %D infractions occur, the worst infraction should be evaluated for result qualification.</p> <p>A CCV must be analyzed in the following instances:</p> <ul style="list-style-type: none"> • at the beginning of each analytical run; • at least once every 10 samples; and • at the end of each analytical run. <p>If multiple CCVs were analyzed to obtain a passing CCV, the calibration is not verified and the calibration frequency is not met.</p> <p>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.</p> <p>If all required ICVs and CCVs were not analyzed, qualify all associated detects as J and all associated non-detects as R.</p>
15	R	R	PE7f	<p>Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.</p>

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
16	N/A	R	PE8	<p>The affected analyte is considered not detected because ion abundance ratios did not meet specifications. The natural isotopic abundances for the chlorine isotopes give a $^{35}\text{Cl}/^{37}\text{Cl}$ ratio of approximately 3.08. Laboratories must statistically derive isotope ratio acceptance criteria to be used as an additional confirmation of analyte identity.</p> <p>When the laboratory does not specify acceptance criteria, the mean of the ration population shall not deviate by more than 10% from the 3.08 theoretical value and the standard deviation shall not significantly exceed 0.2. Between the MDL and the PQL, the individual sample isotope acceptance limits shall be near the population mean $\pm 20\%$ (approximately 3 sigma). Above the PQL, the individual sample isotope ratio acceptance limits shall be near the population mean $\pm 15\%$ (approximately 2 sigma).</p> <p>When isotope ratio acceptance criteria are not met, the laboratory must provide supporting data and explanatory case narrative comments in the data package.</p> <p>If the isotope ratios were not reported, calculate the ratio if the raw data were supplied or request an amended report from the laboratory if the raw data were not supplied. If an isotope ratio is outside the acceptance limits, qualify the detect results as J or R based on professional judgment.</p>
17	N/A	R	PE8a	The ion ratio documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
18	UJ	J-	PE9	The extraction/analytical holding time is exceeded by less than 2X the published method for holding times.
19	R	J-	PE9a	The extraction/analytical holding time is exceeded by less than 2X the published method for holding times.
20	R	J-	PE12	<p>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.</p> <p>The LCS must meet all sample acceptance criteria and all method-specific LCS requirements. The LCS for perchlorate must meet laboratory-derived acceptance criteria. If 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 85% to 115% (perchlorate limits) should be used as the criteria.</p> <p>The LCS percent recovery was <10%. Qualify detected results as J- and not detected results as R.</p>

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
21	UJ	J-	PE12a	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.
22	N/A	J+	PE12b	The LCS percent recovery was > the Upper Acceptance Limit. Follow the external laboratory limits. Qualify detected results as J+.
23	R	R	PE12c	The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
24	R	R	PE12d	<p>The MS/MSD percent recovery was <10%.</p> <p>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.</p> <p>For perchlorate, the MS/MSD recovery acceptance criteria are 75% to 125% with an RPD of $\leq 20\%$. For solid and waste samples, it may be appropriate to accept an RPD of up to 30% based on professional judgment.</p> <p>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 othe 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 results for all 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.</p> <p>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.</p>
25	J	UJ	PE12e	The MS/MSD percent recovery was >10% but <75%. Qualify all detects as J and all non-detects as UJ.
26	N/A	J+	PE12f	The MS/MSD percent recovery was >125%. Qualify all associated detects as J+.
27	J	UJ	PE12g	The MS/MSD relative percent difference was >20%. If the acceptance criteria are not reported, recovery limits of 75% to 125% and an RPD of 20% should be used as the criteria. For solid and waste samples, it may be appropriate to accept an RPD of up to 30% based on professional judgment.

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
28	UJ, R	N/A	PE15	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.
29	UJ	J	PE15a	<p>The sample was diluted because target analytes were > the initial verification calibration.</p> <p>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.</p> <p>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.</p> <p>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.</p> <p>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.</p> <p>If any target analyte exceeded the calibration range and the original undiluted sample analysis was not reported, request this information from the laboratory.</p> <p>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.</p>

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
30.	UJ, R	J	PE16	<p>The Contract Required Detection Limit check standard (CRI) sample did not pass method-acceptance criteria. CRI analysis recoveries for perchlorate analysis must be within limits specified by the Laboratory. If acceptance criteria are not reported, the recovery acceptance range shall be 70% to 130%.</p> <ol style="list-style-type: none"> If frequency criteria were not met, qualify all detects <5X the PQL as J and all non-detects as UJ. If the recovery is > the upper acceptance limit, qualify all associated detects <5X the PQL as J+. 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. <p>If the recovery is <30%, qualify all associated detects <5X the PQL as J- and all associated non-detects as R.</p>
31.	UJ	J	PE16a	<p>The Interference Check Sample recovery was not within ±20% of the known value.</p> <p>The laboratory shall analyze an Interference Check Sample from a matrix containing 500 ppm each of chloride, sulfate, carbonate, and bicarbonate in every batch. The concentration of this standard will be at the PQL.</p> <p>To determine that perchlorate is adequately isolated and recovered under the specific conditions used, this standard should recover within ±20% of the known value.</p> <p>If frequency criteria were not met, note the deficiency in the data validation report. If the recovery is not within ±20% of the known value, note the deficiency in the data validation report. Qualify not detected results as UJ and detected results as J.</p>
32.	R	R	PE16c	The required CRI sample information is missing. Contact the SMO or external laboratory for information.
33.	UJ, R	J, R	PE19	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.
34.	UJ	J	PE88	Duplicate, dilution, or reanalysis.

CONTROLLED DOCUMENT

Users are responsible for ensuring they work to the latest approved revision.
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