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Environment, Safety, and Health Directorate

OIO-DO

Technical Procedure

ROUTINE VALIDATION OF TOTAL PETROLEUM HYDROCARBONS GASOLINE RANGE ORGANIC/DIESEL RANGE ORGANICS ANALYTICAL DATA (METHOD 8015B)

Subject Matter Expert:

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Derivative Classifier: ☐ **Unclassified or** ☐ **DUSA ENVPRO**

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REVISION HISTORY

Document Number and Revision *[Include revision number, beginning with Revision 0]*	**Effective Date** *[Document Control Coordinator inserts effective date]*	**Description of Changes** *[List specific changes made since the previous revision]*
OIO-TP-5171, Rev. 0	8/5/2016	Changed Document type and Organization. Replacing SOP-5171 to OIO-TP-5171

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1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine total petroleum hydrocarbon (TPH) gasoline range organics (GRO) and diesel range organics (DRO) 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 total petroleum hydrocarbon analytical data for 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 National Nuclear Security Administration (NNSA) Model Data Validation Procedure. Data qualifiers and reason codes are assigned according to the specifications in this method-specific procedure.

2.2 Precautions

Nothing in this procedure precludes the data validator from going beyond the minimum requirements specified within this procedure. If additional directions are required, the data validator shall reference the 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 QUALIFICATIONS FOR DATA VALIDATORS

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 OR two (2) years of data validation experience.

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1 Routine Validation of TPH GRO/DRO Data

Data Validator

1. Complete Attachment 1, Data Validation Cover Sheet, and Attachment 2, TPH GRO and DRO Analytical Data Validation Checklist, during data validation.
2. Use Attachment 3, Guidelines for the Qualifier and Reason Code Application, for additional guidance.

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 TPH GRO and DRO 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: *5171-1 Example of a Data Validation Cover Sheet*
Attachment 2: *5171-2 TPH GRO and DRO Analytical Data Validation Checklist*
Attachment 3: *5171-3 Guidelines for the Qualifier and Reason Code Application*

ATTACHMENT 1 – 5171-1 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	☐ High Explosives	☐ Dioxin Furans	☐ LCMSMS Perchlorates
☐ TPH-DRO	☐ Metals	☐ PCB Congeners	☐ Organochlorine Pesticides/Polychlorinated Biphenyls
☐ General Chemistry	☐ Radiochemistry	☐ LCMSMS High Explosives	

☐ Other (Describe): _____

Section II. Completeness Check

Yes	No	N/A	(**Check One**)	Yes	No	N/A	(**Check One**)
☐	☐	☐	1. Chain-Of-Custody Form(S)	☐	☐	☐	6. RAW/BSS Data
☐	☐	☐	2. Case Narrative	☐	☐	☐	7. Quality Control Forms
☐	☐	☐	3. Sample Result Forms	☐	☐	☐	8. Quantitation Reports
☐	☐	☐	4. Sample Chromatograms	☐	☐	☐	9. TICS Forms
☐	☐	☐	5. Standard Chromatograms	☐	☐	☐	10. TICS Mass Spectra

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

Validator's Signature: _____ Date: _____

(Attach additional comment sheets as necessary)

ATTACHMENT 2 – 5171-2 TPH GRO AND DRO 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 retention time criteria were not met.	R, DR0 or GR0	R, DR0 or GR0
☐	☐	☐	2. Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, DR0b or GR0b	R, DR0b or GR0b
☐	☐	☐	3. The surrogate is less than 10%R. Follow external laboratory limits.	R, DR3 or GR3	J-, DR3 or GR3
☐	☐	☐	4. The surrogate is less than the Lower Acceptance Limit, but greater than or equal to 10%R. Follow external laboratory limits.	UJ, DR3a or GR3a	J-, DR3a or GR3a
☐	☐	☐	5. The surrogate %R value is greater than the Upper Acceptance Limit. Follow external laboratory limits.	N/A	J+, DR3b or GR3b
☐	☐	☐	6. Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, DR3d or GR3d	R, DR3d or GR3d
☐	☐	☐	7. The sample result is less than or equal to 5 times the concentration of the related analyte in the method blank.	N/A	U, DR4 or GR4
☐	☐	☐	8. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank, but was greater than 5x.	N/A	J+, DR4a or GR4a
☐	☐	☐	9. The sample result is less than or equal to 5 times the concentration of the related analyte in the trip blank, rinsate blank or equipment blank.	U, DR4d or GR4d	N/A
☐	☐	☐	10. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, DR4e or GR4e	R, DR4e or GR4e
☐	☐	☐	11. The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.	UJ or R, DR7 or GR7	J, DR7 or GR7
☐	☐	☐	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 less than 0.995.	UJ, DR7a or GR7a	J, DR7a or GR7a

ATTACHMENT 2 – 5171-2 TPH GRO AND DRO ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)

Page 2 of 2

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	13. The ICV and/or CCV were recovered outside the method specific limits.	UJ, DR7c or GR7c	J, DR7c or GR7c
☐	☐	☐	14. The ICV and/or CCV were not analyzed at the appropriate method frequency.	UJ, DR7d or GR7d	J, DR7d or GR7d
☐	☐	☐	15. Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.	R, DR7f or GR7f	R, DR7f or GR7f
☐	☐	☐	16. The extraction/analytical holding time is greater than 1x and less than or equal to 2 times the applicable holding time requirement.	UJ, DR9 or GR9	J-, DR9 or GR9
☐	☐	☐	17. The holding time was greater than 2 times the applicable holding time requirement.	R, DR9a or GR9a	J-, DR9a or GR9a
☐	☐	☐	18. The LCS percent recovery was less than 10%. Follow the external laboratory limits.	R, DR12 or GR12	J-, DR12 or GR12
☐	☐	☐	19. The LCS percent recovery was less than the Lower Acceptance Limit, but greater than or equal to 10%. Follow the external laboratory limits.	UJ, DR12a or GR12a	J-, DR12a or GR12a
☐	☐	☐	20. The LCS percent recovery was greater than the Upper Acceptance Limit. Follow the external laboratory limits.	N/A	J+, DR12b or GR12b
☐	☐	☐	21. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or the external laboratory for information.	R, DR12c or GR12c	R, DR12c or GR12c
☐	☐	☐	22. The MS/MSD percent recovery was less than 10%.	R, DR12d or GR12d	R, DR12d or Gr12d
☐	☐	☐	23. The MS/MSD percent recovery was greater than or equal to 10%, but less than 70%.	UJ, DR12e or GR12e	J, DR12e or GR12e
☐	☐	☐	24. The MS/MSD percent recovery was greater than 130%.	N/A	J+, DR12f or GR12f
☐	☐	☐	25. The MS/MSD relative percent difference was greater than 30%.	UJ, DR12g or GR12g	J, DR12g or GR12g

ATTACHMENT 2 – 5171-2 TPH GRO AND DRO ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)

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Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
☐	☐	☐	26. 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 matrix interference.)	UJ, R, DR15 or GR15	R, DR15 or GR15
☐	☐	☐	27. The LANL project chemist identified quality deficiencies in the reported data that requires further qualification. This code can only be used under advisement by the LANL project chemist.	UJ, R, DR19 or GR19	J, R, DR19 or GR19
☐	☐	☐	28. Duplicate, dilution, or reanalysis.	UJ, DR88 or GR88	J, DR88 or GR88

ATTACHMENT 3 – 5171-3 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

Page 1 of 2

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
1	R	R	DRO or GRO	The retention time criteria were not met.
2	R	R	DROb or GROb	Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
3	R	J-	DR3 or GR3	The surrogate is less than 10%R, which indicates the potential for a severely low bias in the results. Follow the external laboratory limits.
4	UJ	J-	DR3a or GR3a	The surrogate is less than the Lower Acceptance Limit, but greater than or equal to 10%R, which indicates the potential for a low bias in the results. Follow the external laboratory limits.
5	N/A	J+	DR3b or GR3b	The surrogate %R value is greater than the Upper Acceptance Limit, which indicates a potential for a high bias in the results and a potential for false positive results. Follow the external laboratory limits.
6	R	R	DR3d or GR3d	Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
7	N/A	U	DR4 or GR4	The sample result is less than or equal to 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.
8	N/A	J+	DR4a or GR4a	The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was greater than 5x.
9	U	N/A	DR4d or GR4d	The sample result is less than or equal to 5 times 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.
10	R	R	DR4e or GR4e	Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
11	UJ, R	J	DR7 or GR7	The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.
12	UJ	J	DR7a or GR7a	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 than 0.995.
13	UJ	J	DR7c or GR7c	The ICV and/or CCV were recovered outside the method specific limits.
14	UJ	J	DR7d or GR7d	The ICV and/or CCV were not analyzed at the appropriate method frequency.

ATTACHMENT 3 – 5171-3 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION (CONT)

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15	R	R	DR7f or GR7f	Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.
16	UJ	J-	DR9 or GR9	The extraction/analytical holding time is greater than 1x and less than or equal to 2 times the applicable holding time requirement.
17	R	J-	DR9a or GR9a	The extraction/analytical holding times were exceeded by more than 2x the published method for holding times.
18	R	J-	DR12 or GR12	The LCS percent recovery was less than 10%. Follow the external laboratory limits.
19	UJ	J-	DR12a or GR12a	The LCS percent recovery was less than the Lower Acceptance Limit but greater than or equal to 10%. Follow the external laboratory limits.
20	N/A	J+	DR12b or GR12b	The LCS percent recovery was greater than the Upper Acceptance Limit. Follow the external laboratory limits.
21	R	R	DR12c or GR12c	The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
22	R	R	DR12d or GR12d	The MS/MSD percent recovery was less than 10%.
23	UJ	J	DR12e or GR12e	The MS/MSD percent recovery was greater than or equal to 10% but less than 70%.
24	N/A	J+	DR12f or GR12f	The MS/MSD percent recovery was greater than 130%.
25	UJ	J	DR12g or GR12g	The MS/MSD relative percent difference was greater than 30%.
26	UJ, R	R	DR15 or GR15	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 matrix interference.)
27	UJ, R	J, R	DR19 or GR19	The project chemist identified quality deficiencies in the reported data that requires further qualification. This code can only be used under advisement by the project chemist.
28	UJ	J	DR88 or GR88	Duplicate, dilution, or reanalysis.

From: Hollis, Diana J
Sent: Tuesday, August 23, 2016 11:08 AM
To: Maestas, Pamela Therese
Subject: RE: DC review of ADESH procedures

Hi Pamela,

All of the procedures are covered by at least one DUSA, so they're approved for posting.

Diana

From: Maestas, Pamela Therese
Sent: Tuesday, August 23, 2016 11:01 AM
To: Hollis, Diana J <dhollis@lanl.gov>
Subject: DC review of ADESH procedures

Hi Diana,
At your leisure, can you please DC review the attached ADESH procedures (EPC-CP-TPP-MetM, EPC-ES-TP-013, OIO-TP-5166, OIO-TP-5171, and OIO-TP-5191). These need to be posted to the EPRR because they are referenced in ADEM/ADESH deliverables.
Thank you.

Pamela T. Maestas
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