Appendix L

Data Validation Reports

EPA
United States
Environmental Protection
Agency

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2024 Clean Harbors PFAS Tests
Other Test Method 45 Data Validation Report

Office of Research and Development
Center For Environmental Solutions and Emergency Response

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2024 Clean Harbors PFAS Tests
Other Test Method 45 Data Validation Report

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Abbreviations

CCC	calibration check compound
CCV	continuing calibration verification
COC	Chain of Custody
DI	deionized
DOD	Department of Defense
DQO	data quality objective
EPA	Environmental Protection Agency
H2O	dihydrogen monoxide (water)
ICV	initial calibration verification
LCMSMS	liquid chromatography / tandem mass spectrometry
LCS	laboratory control standard
LCSD	laboratory control standard duplicate
MDL	method detection limit
MeOH	methanol
MS	matrix spike
MSD	matrix spike duplicate
NH4OH	ammonium hydroxide
OTM	Other Test Method
PFAS	per and polyfluoroalkyl substances
QSM	Quality Systems Manual
RF	response factor
RFA	Request for Analysis
RPD	relative percent difference
RL	reporting limit
RSD	percent relative standard deviation
RT	retention time
SOP	standard operating procedure

1 Summary

Samples submitted for validation were collected during the week of November 11th, 2024, as part of the Per and Polyfluoroalkyl Substances (PFAS) Test Program being conducted at the Clean Harbors Aragonite Hazardous Waste Incinerator in Grantsville, Utah. The samples were analyzed by the Eurofins Environment Testing USA Laboratory located in Knoxville, Tennessee. The types of samples analyzed are noted below:

- Other Test Method 45 (OTM-45) Measurement of Selected Per- and Polyfluorinated Alkyl Substances from Stationary Sources (OTM 45).

The results of these analyses have been reviewed for a data quality assessment based on the laboratory's analytical method procedures, and the requirements outlined by the United States Department of Defense (DOD) Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by Quality Systems Manual for Environmental Laboratories (QSM) Table B-15.

2 PER AND POLYFLUORINATED ALKYL SUBSTANCES

2.1 Samples

The analytical data for the stack gas sample fractions that were analyzed for Per and Polyfluorinated Alkyl Substances (PFAS) were received for validation as a single data package. The stack gas samples were analyzed as required by OTM 45 via liquid chromatography / tandem mass spectrometry (LCMSMS) based on Method 537 and 537.1 (Modified). All applicable compliance areas have been reviewed, and any significant findings are discussed below. Section 2.1 provides a list of the primary data quality objectives (DQOs) evaluated during this review. Section 2.2 summarizes the significant findings of the evaluation.

The following samples are included in this validation package:

Job ID 140-39576-1 List of Samples

- S-2001,2002 R1A OTM-45 FH
- S-2003,2004, R1A OTM-45 BH
- S-2005,2006 R1A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE
- S-2007 R1A OTM-45 BT XAD
- S-2008,2009 R2A OTM-45 FH
- S-2010,2011 R2A OTM-45 BH
- S-2012,2013 R2A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE
- S-2014 R2A OTM-45 BT XAD
- S-2015,2016 R3A OTM-45 FH
- S-2017,2018 R3A OTM-45 BH
- S-2019,2020 R3A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE
- S-2021 R3A OTM-45 BT XAD
- S-2022 QA OTM-45 GLASSWARE RINSE PB
- S-2023 QA OTM-45 FILTER MEDIA BLANK
- S-2024 QA OTM-45 XAD MEDIA BLANK
- S-2025 QA OTM-45 MEOH/5% NH4OH MEDIA BLANK
- S-2026 QA OTM-45 DI WATER MEDIA BLANK
- S-2027,2028 QA OTM-45 FH PB
- S-2029,2030, QA OTM-45 BH PB
- S-2031,2032 QA OTM-45 IMPINGERS CONTENTS, IMPINGER SOLVENT RINSE PB
- S-2033 QA OTM-45 BT XAD PB
- A-2499 OTM-45 MEDIA CHECK XAD
- A-2501 OTM-45 MEDIA CHECK FILTER.

2.2 Applicable Data Quality Objectives

2.2.1 Contract Compliance Monitoring

Requirements: The laboratory was requested to follow the analytical guidelines as presented in Revision 1 of OTM-45. The Eurofins laboratory had an existing SOP for OTM-45 at the time that this project was collecting samples in the field. The update changes to OTM-45 were in progress at the time the field sampling was proceeding. To be compliant with Revision 1 of OTM-45, the Laboratory was asked to follow the update to the method.

Laboratory sample data reports have been evaluated to determine if all required quality assurance and quality control measurements were performed appropriately.

Data packages have been reviewed to determine if all required reporting has been completed, and to verify that sufficient documentation has been provided as backup.

Data packages have also been reviewed to determine if the analytical procedures required by the project requirements were followed.

2.2.2 Sample Handling Criteria

Holding Time and Preservation Requirements: OTM 45 sample preservation requirements are that samples should be chilled at 6^{o}C or less from the time of sample collection to extraction. Samples are to be stored no more than 28 days from the time of sampling to the extraction processes are commenced, and 28 days from extractions to analysis if samples are stored at room temperature after extraction and one year if sample extracts are chilled. The Eurofins Laboratory SOP requires that samples be chilled between 0^{o}C and 6^{o}C from the time of sample collection to extraction and extracts be stored chilled between 0^{o}C and 6^{o}C and analyzed within 40 days.

Job ID 140-39576-1 Findings: All samples were received at the laboratory at the appropriate storage temperatures which were between 0.3^{o} and 1.5^{o}C in the coolers received. All samples were extracted within the holding time sample storage window that ranged between 20 to 27 days from sample collection. Sample extracts were analyzed within the extract holding time which was between 17 to 27 days from extraction to the time of analysis. The Run 2A sample fractions were initially analyzed at a dilution due to concentrations of native analytes. Per client request, these samples were reanalyzed without dilution 43 days following extraction. OTM 45 allows for chilled samples to be stored for one year after extraction. Therefore, samples were all properly preserved, and all holding times were met for these samples.

The Test Plan and Request for Analyses / Chain of Custody (RFA/COCs) documentation was evaluated against the field samples collected and received at the laboratory for this project. All samples scheduled for collection in the test plan were collected and received at the laboratory for analysis. Additionally, media check samples for Filter Media and XAD-2 were set aside in the laboratory at the time of media preparation. The media checks were batched with the field samples at laboratory check-in and analyzed with the field samples. Media checks were not sent to the site but remained at the laboratory until the field samples arrived.

2.2.3 Instrument Performance Criteria

General Requirements: . OTM 45 requires Internal standard spikes placed onto each continuing calibration verification (CCV) are required to exhibit an area count within 50% to 150% of the initial calibration. The laboratory SOP requires Internal standard spikes placed onto each CCV are required to exhibit a retention time (RT) within 0.2 minutes and an area count within 50% to 150% of the initial calibration.

Subsequently, OTM 45 requires Internal standard spikes placed onto each CCV are required to exhibit an area count within 50% to 150% of the associated continuing calibration check (CCC). The laboratory SOP requires Internal standard spikes placed onto each CCV are required to exhibit a RT within 0.2 minutes and an area count within 50% to 150% of the CCC.

Job ID 140-39576-1 Findings: All internal standard RT and area counts within the required criteria.

2.2.4 Initial and Continuing Calibrations

Requirements: OTM 45 requires a minimum of five (5) calibration points for the initial calibration with quadratic or linear regression allowed for curve evaluation. Each calibration standard should be within 90% - 110% of the true value.

The laboratory SOP requires a minimum of five to six calibration points for the initial calibration with average response factor (RF) relative standard deviation (RSD), quadratic, or linear regression allowed for curve evaluation. RSDs reported for compounds quantitated against an identically labeled analog must be $\leq$ 35% and RSDs reported for compounds quantitated against a closely related labeled analog must be $\leq$ 50%. Curves using linear regression are required to have a correlation coefficient (r) $\geq$ 0.995 and curves using a quadratic equation are required to have a coefficient of determination (r2) $\geq$ 0.990.

OTM 45 requires that CCCs be analyzed at the beginning, after every tenth samples, and at the end of each analysis batch. CCCs must be within 70% - -130% of the true value or 50% - 150% pf the true value for low level CCCs.

The Laboratory SOP requires an initial calibration verification (ICV) before samples are analyzed and that CCCs be analyzed at the beginning, after every tenth samples, and at the end of each analysis batch. All native analytes quantitated against an identically labeled analog must be within or equal to 60% to 140% of the true value and all native analytes quantitated against a closely related labeled analog must be between 50% to 150% of the true value.

Job ID 140-39576-1 Findings: The Initial calibrations, ICVs, and CCVs were analyzed as required at the beginning, every 10 samples, and at the end of the analysis batch.

All internal standards met the %RSD, or curve regression requirements. Two compounds, Perfluorobutanesulfonic acid (PFBS) and PES, reported concentrations more than 10% difference from the true value. Each standard reported outside 90% to 110% was reported slightly below 90% difference. This criterion alone does not require qualification of the data but may indicate that ongoing QC criteria may not be met.

All continuing calibration met the laboratory SOP requirements. A few target compounds were slightly outside the OTM 45 70% - 130% criteria (<35%D) and were not low level CCCs. All associated samples reported non-detect results and only two of the target compounds reported low recoveries. 8:2 FTCA

reported a -34.1%D associated with sample S-2003,2004, R1A OTM-45 BH and PFECA B reported a -34.2%D associated with the reanalysis of samples S-2008,2009 R2A OTM-45 FH, S-2010,2011 R2A OTM-45 BH, and S-2012,2013 R2A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE. No qualifiers have been applied based on this slight variation from the OTM 45 requirement as the laboratory SOP requirements were met.

2.2.5 Precision Objectives

Requirements: The laboratory SOP includes the requirement for laboratory control sample/laboratory control sample duplicate (LCS/LCSD) spikes when no matrix spike / matrix spike duplicate (MS/MSD) analysis is possible as is the case with OTM 45 sampling and analysis projects. The Relative Percent Differences (RPDs) should be within the laboratory SOP limits based on historical data. The project Test Work plan and DOD Validation Guidelines require that LCS/LCSD and / or MS/MSD RPDs for all analytes be less than 30%.

Job ID 140-39576-1 Findings: As expected, there were no MS/MSD analyses performed as there was an inadequate amount of sample to perform these analyses due to the sampling method. Each train fraction is used in its entirety as a sample composite. No additional sample portion is available on which to perform matrix spikes. Therefore, the LCS/LCSD analyses were performed to demonstrate precision for these OTM 45 samples. The LCS/LCSD analyses were performed for each of the three (3) fractions of the OTM 45 sampling train. RPDs for all spiked analytes were reported within the laboratory specified limits, test plan, and the DOD required limits except for the RPD for 10:2 FTCA (36%) associated with the back half and secondary XAD-2 resin fractions. No qualifiers were applied as all associated sample results are reported as non-detects.

2.2.6 Accuracy Objectives

Requirements: The laboratory SOP requirement for an LCS and an LCSD be performed for sampling train fractions since the sampling media from the sampling train is completely used in the extraction of the train fractions. Recoveries for the spiked samples are to be within the laboratory control limits based on historical data. The project Work Plan requires LCS recoveries to be within 60% - 140% and DOD Validation Guidelines specify that an LCS be performed and is to meet the QSM Appendix A requirements (40% - 150%) if project requirements are not specified.

The laboratory SOP also requires isotope dilution standards spikes be processed on each sample and surrogate sampling spikes standard be applied to the XAD- resins. Recoveries are to be within the laboratory required limits that are based on historical data. Internal standards should also be spiked into all samples with responses within 50 – 200% of the corresponding calibration and the retention times should be within 30 seconds of the corresponding calibration. The DOD Validation Guidelines require isotope dilution standard spikes processed on each sample with recoveries within 20% - -150% if no inhouse limits are specified.

Job ID 140-39576-1 Findings: All internal standard responses and retention times are reported within the required limits.

There were several isotope dilution standards with recoveries reported outside the laboratory specified limits of 25% - 150%. Positive results associated with recoveries greater than 150% Have J+ qualifier applied. All results associated with recoveries of less than 25% have J- or UJ- qualifiers applied to the data. Sample results with qualifiers applied by the Validator are listed below

Isotope Dilution Standards Outside the Required Criteria

N-ethylperfluoro-1-octanesulfonamide (UJ-)

S-2003,2004, R1A OTM-45 BH

d-N-MeFOSA-M	24%
NMeFOSA	(UJ-)

S-2017,2018 R3A OTM-45 BH

d-N-EtFOSA-M	24%
N-ethylperfluoro-1-octanesulfonamide (UJ-)	

S-2024 QA OTM-45 XAD MEDIA BLANK

3C4 PFOS	24%
Perfluorooctanesulfonic acid (PFOS) (J-)	

S-2014 R2A OTM-45 BT XAD
January 21, 2025 Analysis

13C2 PFDoA	23%
PFDoA	(J-)
PFTriA	(UJ-)
13C-10:2 FTCA	22%
10:2 FTCA	(UJ-)

LCS 140-93945/2-B (FH Filter)

13C-8:2 FTUCA	168%
8:2 FTUCA	(J+)

LCSD 140-93945/3-B (FH Filter)

13C-8:2 FTUCA	171%
*:2 FTUCA	(J+)
13C-6:2 FTUCA	158%
6:2 FTUCA	(J+)

MB 140-93949/1-B (BH XAD)

13C2 PFDoA	21%
PFDoA	(UJ-)
PFTriA	(UJ-)
d9-N-EtFOSE-M	23%

LCS 140-93949/2-B (BH XAD)

13C2 PFDoA	19%
PFDoA	(J-)
PFTriA	(J-)
d-N-EtFOSA-M	19%
N-ethylperfluoro-1-octanesulfonamide (J-)	
d-N-MeFOSA-M	22%
NMeFOSA	(J-)
2-(N-methylperfluoro-1-octanesulfonamido) ethanol (J-)	
Perfluorododecanesulfonic acid (PFDoS) (J-)	
Perfluorotridecanoic acid (PFTriA) (J-)	
13C-8:2 FTUCA	165%
8:2 FTUCA	(J+)
13C-6:2 FTUCA	179%
6:2 FTUCA	(J+)
13C-10:2 FTCA	21%
10:2 FTCA	(J-)

LCSD 140-93949/3-B (BH XAD)

13C2 PFDoA	23%
PFDoA	(J-)
PFTriA	(J-)
13C4 PFOS	24%
Perfluorooctanesulfonic acid (PFOS) (J-)	
d-N-EtFOSA-M	23%
N-ethylperfluoro-1-octanesulfonamide (J-)	
13C-8:2 FTUCA	180%
8:2 FTUCA	(J+)
13C-6:2 FTUCA	178%
6:2 FTUCA	(J+)

Two sampling surrogates reported 0% to 0.05% recovery for the condensate/impinger samples for Runs R1A, R2A, and R3A. Surrogates were not added to these samples. The surrogates were analyzed to evaluate breakthrough.

The LCS/LCSD recoveries were within Test Plan and laboratory specified limits except as listed below. Positive sample results associated with LCS recoveries reported below the criteria have a “J-“ qualifier applied by the Validator and non-detect results have a “UJ-” qualifier, applied by the Validator. Positive sample associated with LCS recoveries reported exceeding the criteria have a “J+“ qualifier applied by

the Validator.

Analytes	LCS %R	LCSD %R	Acceptance Criteria
LCS/LCSD 140-93798 (Condensate/Impinger)			
Perfluorooctadecanoic acid	66%	**53%**	60% - 140%
3:3 FTCA	**145%**	136%	60% - 140%
LCS/LCSD 140-93945 (FH/Filter)			
Perfluorotridecanoic acid (PFTriA)	129%	**148%**	60% - 140%
LCS/LCSD 140-93949 (BH/XAD)			
Perfluoroheptanesulfonic acid (PFHpS)	**177%**	**185%**	60% - 140%
Perfluorononanesulfonic acid (PFNS)	**51%**	**57%**	60% - 140%
Perfluorodecanesulfonic acid (PFDS)	**47%**	**56%**	60% - 140%
Perfluorododecanesulfonic acid (PFDoS)	131%	**149%**	60% - 140%
DONA	**354%**	**375%**	60% - 140%
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	**59%**	65%	60% - 140%
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	**51%**	64%	60% - 140%
5:3 FTCA	**170%**	**185**%	60% - 140%
3:3 FTCA	**144%**	**148%**	60% - 140%

2.2.7 Blanks

Requirements: The analysis of one method blank per preparatory batch of samples is required by the laboratory SOP and DOD validation guidance. The DOD Validation Guidance requires that positive blank results be compared to positive sample results and qualified as specified in the guidance.

The PFAS Test Program Test Plan required that a blank train (proof blank) and media blanks be collected and analyzed for contamination.

Job ID 140-39576-1 Findings: A method blank (laboratory blank) was extracted with the samples for each OTM 45 sample matrix type (i.e., filter, XAD-2 resin, and Condensate). Additionally, a complete OTM 45 blank train (proof blank) and OTM 45 media blanks were collected and processed by the laboratory for analysis. The following blank samples reported positive results.

S-2024 QA OTM-45 XAD MEDIA BLANK
S-2026 QA OTM-45 DI WATER MEDIA BLANK
S-2027,2028 QA OTM-45 FH PB
S-2029,2030, QA OTM-45 BH PB
S-2031,2032 QA OTM-45 IMPINGERS CONTENTS, IMPINGER SOLVENT RINSE PB
S-2033 QA OTM-45 BT XAD PB
A-2499 OTM-45 MEDIA CHECK XAD
A-2501 OTM-45 MEDIA CHECK FILTER.
MB 140-93798/1-B (Impinger)
MB 140-93945/1-B (Front Half)

Associated sample results were compared to the blank results. Associated sample results reported between the method detection limit (MDL) and the reporting limit (RL) had "U" qualifiers applied by the Validator. Associated sample results reported above the RL and less than 5X the blank result had "J+" qualifiers applied by the Validator. Qualified data are listed below.

S-2001,2002 R1A OTM-45 FH

Perfluoroheptanoic acid (PFHpA) (J+)
Perfluorohexanoic acid (PFHxA) (J+)
Perfluorooctanoic acid (PFOA) (J+)
Perfluorononanoic acid (PFNA) (U)
Perfluorodecanoic acid (PFDA) (U)

S-2003,2004, R1A OTM-45 BH

Perfluorooctanoic acid (PFOA) (J+)
Perfluorododecanoic acid (PFDoA) (U)
Perfluorobutanesulfonic acid (PFBS) (J+)
Perfluorooctanesulfonic acid (PFOS) (U)

S-2007 R1A OTM-45 BT XAD

Perfluorooctanoic acid (PFOA) (U)
Perfluorododecanoic acid (PFDoA) (U)
Perfluorobutanesulfonic acid (PFBS) (J+)
Perfluorooctanesulfonic acid (PFOS) (U)

S-2008,2009 R2A OTM-45 FH (DL1)

Perfluoroheptanoic acid (PFHpA) (U)
Perfluorohexanoic acid (PFHxA) (U)

S-2010,2011 R2A OTM-45 BH

Perfluorododecanoic acid (PFDoA) (U)
Perfluorobutanesulfonic acid (PFBS) (J+)
Perfluorooctanesulfonic acid (PFOS) (U)

S-2014 R2A OTM-45 BT XAD

Perfluorododecanoic acid (PFDoA) (U)
Perfluorobutanesulfonic acid (PFBS) (J+)
Perfluorooctanesulfonic acid (PFOS) (U)

S-2017,2018 R3A OTM-45 BH

Perfluorooctanoic acid (PFOA) (U)
Perfluorododecanoic acid (PFDoA) (U)
Perfluorobutanesulfonic acid (PFBS) (J+)
Perfluorooctanesulfonic acid (PFOS) (U)

S-2021 R3A OTM-45 BT XAD

Perfluorododecanoic acid (PFDoA) (U)
Perfluorobutanesulfonic acid (PFBS) (J+)
Perfluorooctanesulfonic acid (PFOS) (U)

2.2.8 Qualitative and Quantitative Results

Requirement: The data package has been evaluated to determine if any transcription or calculation errors exist. The items related to the quality of the data that could lead to inaccuracies and have not been previously discussed are evaluated and discussed in this section.

Method OTM 45 requires breakthrough to be calculated by dividing the mass in the breakthrough XAD-2 fraction (fraction 4) result by the sum of the masses in first three fractions and multiplying by 100%. If a breakthrough result is reported as 10% or greater, the mass result of the breakthrough XAD-2 resin should be included in the total sample mass for emissions calculations. If breakthrough is reported as 30% or greater, the data should be accessed for impact on the results. The breakthrough calculation is not required when the fraction 4 target compound mass is below three times (3X) its MDL (see OTM-45 Section 9.1.6).

Job ID 140-39576-1 Findings: There are no transcription errors that have been found during the review of this data package.

The target analyte results reported below the MDL as "ND" (non-detects) have a "U" qualifier applied to

them by the Validator. Target analytes reported between the MDL and the RL will retain the "J" qualifier applied by the laboratory.

HFPO-DA was reported as exceeding the calibration range for the undiluted analyses of samples S-2008,2009 R2A OTM-45 FH, S-2010,2011 R2A OTM-45 BH, and S-2012,2013 R2A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE and have a "J" qualifier applied. Results from the diluted analyses should be reported for HFPO-DA.

Breakthrough was calculated for each analyte for which the breakthrough XAD-2 reported positive sample results. For train R2A, the DL1 (1/21/25) sample results were used for the breakthrough evaluation. Below are the breakthrough percentages and the action taken for breakthrough. Results associated with breakthrough less than 10% required no action. Results associated with breakthrough between 10% and 30% should have the concentration of the breakthrough XAD-2 added to the total sample mass for emissions calculations. Results associated with breakthrough greater than 30% should have the second XAD-2 added to the total sample mass for emissions calculations and have a "UJ" or "J" qualifier added by the Validator to all train fractions when the XAD-2 results is less than the reporting limit (RL) and "UJ-" or "J-" when the XAD-2 results are greater than the RL.

Breakthrough Analysis Results

Analyte	Breakthrough (%)	Action taken
R1A Sample Train		
Perfluorobutanesulfonic acid (PFBS)	57.1%	XAD-2 result > RL. Breakthrough XAD-2 result added to total sample mass. All R1A train fractions are qualified as J- or UJ-.
R2A Sample Train		
Perfluorobutanesulfonic acid (PFBS)	65.2%	XAD-2 result > RL. Breakthrough XAD-2 result added to total sample mass. All R1A train fractions are qualified as J- or UJ-.
HFPO-DA	0.2%	No action required, breakthrough <10%..
R3A Sample Train		
Perfluorobutanesulfonic acid (PFBS)	45.4%	XAD-2 result > RL. Breakthrough XAD-2 result added to total sample mass. All R1A train fractions are qualified as J- or UJ-.

2.3 Data Quality Summary

Overall, the data quality objectives for accuracy, precision, and completeness were met for the EPA OTM 45 data. There were data quality qualifiers applied to the data for the quality criteria described below. Data quality for the OTM 45 analysis is deemed acceptable for its intended use.

The target analyte results reported below the MDL as “ND” (non-detects) have a “U” qualifier applied to them by the Validator. Target analytes reported between the MDL and the RL will retain the “J” qualifier applied by the laboratory.

HFPO-DA was reported as exceeding the calibration range for the Dilution Factor 1 analyses of samples S-2008,2009 R2A OTM-45 FH, S-2010,2011 R2A OTM-45 BH, and S-2012,2013 R2A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE and have a “J” qualifier applied. Results for HFPO-DA should be reported from the Dilution Factor 5 analysis for sample S-2008,2009 R2A OTM-45 FH, the Dilution Factor 20 analysis for sample S-2010,2011 R2A OTM-45 BH, and the Dilution Factor 50 analysis for sample analyses S-2012,2013 R2A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE.

The following samples had qualifiers applied by the Validator for LCS/LCSD results outside the criteria.

Sample	Analyte and Qualifier Applied
S-2008,2009 R2A OTM-45 FH DL1 (1/21/25)	Perfluorotridecanoic acid (PFTriA) (J+)
S-2003,2004, R1A OTM-45 BH S-2010,2011 R2A OTM-45 BH DL20 (1/6/25) S-2010,2011 R2A OTM-45 BH DL1 (1/21/25) S-2017,2018 R3A OTM-45 BH S-2024 QA OTM-45 XAD MEDIA BLANK S-2029,2030, QA OTM-45 BH PB A-2499 OTM-45 MEDIA CHECK XAD S-2007 R1A OTM-45 BT XAD S-2014 R2A OTM-45 BT XAD S-2014 R2A OTM-45 BT XAD DL 1 (1/6/25) S-2021 R3A OTM-45 BT XAD DL 1 (1/21/25) S-2033 QA OTM-45 BT XAD PB S-2022 QA OTM-45 GLASSWARE RINSE PB S-2025 QA OTM-45 MEOH/5% NH4OH MEDIA BLANK	Perfluorononanesulfonic acid (PFNS) (UJ-) Perfluorodecanesulfonic acid (PFDS) (UJ-) 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (UJ-) 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (UJ-)
S-2003,2004, R1A OTM-45 BH S-2010,2011 R2A OTM-45 BH DL1 (1/21/25) S-2017,2018 R3A OTM-45 BH	5:3 FTCA (J+)
S-2005,2006 R1A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE S-2012,2013 R2A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE S-2019,2020 R3A OTM-45 CONDENSATE, IMPINGER SOLVENT RINSE S-2026 QA OTM-45 DI WATER MEDIA BLANK S-2031,2032 QA OTM-45 IMPINGERS CONTENTS, IMPINGER SOLVENT RINSE PB	Perfluorooctadecanoic acid (UJ-)
LCS 140-93798/2-B (Condensate/Impinger)	3:3 FTCA (J+)
LCSD 140-93798 (Condensate/Impinger)	Perfluorooctadecanoic acid (J-)
LCSD 140-93945 (FH/Filter)	Perfluorotridecanoic acid (PFTriA) (J+)
LCS 140-93949 (BH/XAD)	Perfluoroheptanesulfonic acid (PFHpS) (J+) Perfluorononanesulfonic acid (PFNS) (J-) Perfluorodecanesulfonic acid (PFDS) (J-)
LCSD 140-93949 (BH/XAD)	Perfluoroheptanesulfonic acid (PFHpS) (J+) Perfluorononanesulfonic acid (PFNS) (J-) Perfluorodecanesulfonic acid (PFDS) (J-)

The following samples had qualifiers applied by the Validator for low Isotope dilution standard recoveries

Sample	Analyte and Qualifier Applied
S-2003,2004, R1A OTM-45 BH	NMeFOSA (UJ-)
S-2017,2018 R3A OTM-45 BH	N-ethylperfluoro-1-octanesulfonamide (UJ-)
S-2024 QA OTM-45 XAD MEDIA BLANK	Perfluorooctanesulfonic acid (PFOS) (UJ-)
S-2014 R2A OTM-45 BT XAD DL1 (1/21/25)	PFDoA (J-), PFTriA (UJ-), 10:2 FTCA (UJ-)
LCS 140-93945/2-B (FH / Filter)	8:2 FTUCA (J+)
LCSD 140-93945/3-B (FH / Filter)	8:2 FTUCA (J+), 6:2 FTUCA (J+)
MB 140-93949/1-B (BH / XAD)	PFDoA (UJ-), PFTriA (UJ-), N-ethylperfluoro-1-octanesulfonamide (UJ-)
LCSD 140-93949/3-B (BH / XAD)	PFDoA (J-), PFTriA (J-), 8:2 FTUCA (J+), 6:2 FTUCA (J+), Perfluorooctanesulfonic acid (PFOS) (J-), N-ethylperfluoro-1-octanesulfonamide (J-)
LCS 140-93949/2-B (BH / XAD)	PFDoA (J-), PFTriA (J-), 8:2 FTUCA (J+), 6:2 FTUCA (J+), NMeFOSA (J-), 10:2 FTCA (J-), N-ethylperfluoro-1-octanesulfonamide (J-) 2-(N-methylperfluoro-1-octanesulfonamido) ethanol (J-) Perfluorododecanesulfonic acid (PFDoS) (J-) Perfluorotridecanoic acid (PFTriA) (J-)

The following samples had qualifiers applied by the Validator for blank contaminations

Sample	Analyte and Qualifier Applied
S-2001,2002 R1A OTM-45 FH	Perfluoroheptanoic acid (PFHpA) (J+) Perfluorohexanoic acid (PFHxA) (J+) Perfluorooctanoic acid (PFOA) (J+) Perfluorononanoic acid (PFNA) (U) Perfluorodecanoic acid (PFDA) (U)
S-2008,2009 R2A OTM-45 FH DL1 (1/21/25)	Perfluoroheptanoic acid (PFHpA) (U) Perfluorohexanoic acid (PFHxA) (U)
S-2003,2004, R1A OTM-45 BH S-2010,2011 R2A OTM-45 BH DL1 (1/21/25) S-2017,2018 R3A OTM-45 BH S-2007 R1A OTM-45 BT XAD S-2014 R2A OTM-45 BT XAD S-2021 R3A OTM-45 BT XAD	Perfluorododecanoic acid (PFDoA) (U) Perfluorooctanesulfonic acid (PFOS) (U) Perfluorobutanesulfonic acid (PFBS) (J+)
S-2003,2004, R1A OTM-45 BH	Perfluorooctanoic acid (PFOA) (J+)
S-2017,2018 R3A OTM-45 BH S-2007 R1A OTM-45 BT XAD	Perfluorooctanoic acid (PFOA) (U)

The following actions when determining the total sample mass and the following qualifiers were applied by the Validator Based on a Breakthrough analysis. Note: For train R2A, the DL1 (1/21/25) sample results were used for the breakthrough evaluation and qualifiers were applied to the undiluted sample results.

Analyte	Breakthrough (%)	Action taken
R1A Sample Train		
Perfluorobutanesulfonic acid (PFBS)	57.1%	XAD-2 result > RL. Breakthrough XAD-2 result added to total sample mass. All R1A train fractions are qualified as J- or UJ-.
R2A Sample Train		
Perfluorobutanesulfonic acid (PFBS)	65.2%	XAD-2 result > RL. Breakthrough XAD-2 result added to total sample mass. All R1A train fractions are qualified as J- or UJ-.
R3A Sample Train		
Perfluorobutanesulfonic acid (PFBS)	45.4%	XAD-2 result > RL. Breakthrough XAD-2 result added to total sample mass. All R1A train fractions are qualified as J- or UJ-.

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OTM-50 Data Validation Report

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2024 Clean Harbors PFAS Tests
OTM-50 Data Validation Report

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Abbreviations

ADP	Analytical Data Package
CCV	continuing calibration verification
CO2	carbon dioxide
COC	Chain of Custody
DOD	Department of Defense
GC/MS	gas chromatography/mass spectrometry
Hg	mercury
ICV	initial calibration verification
LCS	laboratory control standard
LCSD	laboratory control standard duplicate
MDL	method detection limit
MS	matrix spike
MSD	matrix spike duplicate
OTM	Other Test Method
PFAS	per and polyfluoroalkyl substances
RFA	Request for Analysis
RPD	relative percent difference
RRF	relative response factor
RSD	percent relative standard deviation
VFC	Volatile Fluorinated Compounds

1 Summary

Samples submitted for validation were collected during the week of November 11th, 2024 as part of the Per and Polyfluoroalkyl Substances (PFAS) Test Program being conducted at the Clean Harbors Aragonite Hazardous Waste Incinerator in Grantsville, Utah. The samples were analyzed by the Eurofins Environment Testing USA Laboratory located in Knoxville, Tennessee. The types of samples analyzed are noted below:

- Stack Gas Volatile Fluorinated Compounds - GC/MS (Method OTM-50)

The results of these analyses have been reviewed for a data quality assessment based on the laboratory's analytical method procedures, and the requirements outlined by the United States Department of Defense (DOD) Data Validation Guidelines Module 6: Data Validation Procedures for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-24.

2 STACK GAS SEMI-VOLATILE PRODUCTS OF INCOMPLETE COMBUSTION

2.1 Samples

The stack gas Volatile Fluorinated Compounds (VFC) sample analyses were received as a single data package. The stack gas samples were analyzed as required by Method OTM-50 via gas chromatography/ mass spectrometry (GC/MS) at two calibration levels. The samples were initially analyzed for all target analytes except Tetrafluoromethane (CF4). Tetrafluoromethane was analyzed separately at an elevated calibration level due to its extreme volatility. All applicable compliance areas have been reviewed, and any significant findings are discussed below. Section 2.1 provides a list of the primary data quality objectives evaluated during this review. Section 2.2 summarizes the significant findings of the evaluation.

The following samples are included in this validation package:

Job ID 140-39565-1 List of Samples

- S-2063 R1A OTM-50 SILCO LINED CANISTER
- S-2064 R1B OTM-50 SILCO LINED CANISTER
- S-2065 R1C OTM-50 SILCO LINED CANISTER
- S-2066 R2A OTM-50 SILCO LINED CANISTER
- S-2067 R2B OTM-50 SILCO LINED CANISTER
- S-2068 R2C OTM-50 SILCO LINED CANISTER
- S-2069 R3A OTM-50 SILCO LINED CANISTER
- S-2070 R3B OTM-50 SILCO LINED CANISTER
- S-2071 R3C OTM-50 SILCO LINED CANISTER
- S-2072 QA OTM-50 SILCO LINED CANISTER-PRE-TEST BACKGROUND
- S-2073 QA OTM-50 SILCO LINED CANISTER-POST TEST BACKGROUND.

2.2 Applicable Data Quality Objectives

2.2.1 Contract Compliance Monitoring

Requirements:

Laboratory sample data reports have been evaluated to determine if all required quality assurance and quality control measurements were performed appropriately.

Data packages have been reviewed to determine if all required reporting has been completed, and to verify that sufficient documentation has been provided as backup.

Data packages have also been reviewed to determine if the analytical procedures required by the project requirements were followed.

Job ID 140-39565-1 Findings: The Silco-lined canisters' beginning and ending measured vacuum

pressures were at variance to the recommended vacuum pressures listed in the OTM-50 guidance method. The vacuum measured at the start of a sampling event is recommended to be ~30 inches of Hg, and the ending vacuum after sample collection is recommended to be between 5 and 6 inches of Hg. This guidance is provided presumably to optimize the amount of sample placed in the canister. However, Section 11.5.2 of the Work Plan provides a note regarding the vacuum that is achievable for samples collected at high altitudes and under vacuum stack pressure conditions. The Clean Harbors Aragonite facility is at an altitude of approximately 4,300 feet above sea level, resulting in absolute atmospheric pressure that is 85% of the pressure at sea level. Therefore, the maximum vacuum that can be achieved at this altitude is 85% x 30 inches of Hg, resulting in a value of 25.6 inches Hg.

The actual vacuum pressure measurements are presented in the table below. The beginning values are taken from Alliance field measurements recorded at the Aragonite site, and ending values are taken by the laboratory on sample canisters received at the laboratory. Note that data quality is not affected by these variances from the guidance, and no data qualifiers have been applied to the data set. It is noteworthy that the elevation at the Aragonite sampling site in Utah is 4,300 feet above sea level which affects the measurement of pressure in the field relative to the laboratory in Knoxville, TN (886 feet).

Another factor that affected the amount of stack gas captured in the canister was the type of flow controllers that were provided. New flow controllers were purchased for this project. The sampling location at the Aragonite facility is a vertical downflow duct that was under approximately 15 inches of water column vacuum. A flow controller was used on the Eurofins sample canister to control the gas flow rate into the canister. However, the controller was not rated for the negative pressure that existed at the sampling location, and it did not function as planned. Upon discovery of the flow controller limitations, subsequent sampling was conducted using critical orifices rather than flow controllers.

Sample Number	Sample Description	Initial Vacuum (in Hg)	Receipt Vacuum (in Hg)
140-39565-a-1	S-2063 R1A OTM-50 SILCO LINED CANISTER	25.5	8.8
140-39565-a-2	S-2064 R1B OTM-50 SILCO LINED CANISTER	25.5	7.1
140-39565-a-3	S-2065 R1C OTM-50 SILCO LINED CANISTER	25.0	7.3
140-39565-a-4	S-2066 R2A OTM-50 SILCO LINED CANISTER	25.0	9.1
140-39565-a-5	S-2067 R2B OTM-50 SILCO LINED CANISTER	25.0	9.5
140-39565-a-6	S-2068 R2C OTM-50 SILCO LINED CANISTER	25.5	7.7
140-39565-a-7	S-2069 R3A OTM-50 SILCO LINED CANISTER	25.0	9.2
140-39565-a-8	S-2070 R3B OTM-50 SILCO LINED CANISTER	25.0	7.6
140-39565-a-9	S-2071 R3C OTM-50 SILCO LINED CANISTER	25.0	7.4
140-39565-a-10	S-2072 QA OTM-50 SILCO LINED CANISTER-PRE TEST BACKGROUND	25.5	22.1
140-39565-a-11	S-2073 QA OTM-50 SILCO LINED CANISTER-POST TEST BACKGROUND	25.5	8.3

OTM-50 recommends that a CO2 bias check be performed. This procedure was not performed by the laboratory. However, the laboratory did follow their in-house procedures outlined in the OTM-50 work instructions to dilute the OTM-50 canisters with dilutions of 2-4x due to the expected high levels of CO2. The laboratories use of an alternative procedure to prevent bias from high levels of CO2does not provide a negative effect on data quality. No qualifiers will be applied to the data.

OTM-50 recommends the use of a second source for calibration verification however, secondary source standards are not available for this analytical method. Calibration verifications were performed and are addressed in section 2.1.4 below.

There are two (2) calibration concentration levels for the OTM-50 canister sample analysis performed in association with these samples. All target analytes except for Tetrafluoromethane (CF4) are evaluated against the lower calibration level, and Tetrafluoromethane (CF4) is evaluated against the higher calibration concentration level due to its extreme volatility.

2.2.2 Sample Handling Criteria

Holding Time and Preservation Requirements: Samples are to be held no more than 30 days from the day of sampling to analysis.

Job ID 140-39565-1 Findings: All samples were analyzed between 13 to 16 days from sample collection. Therefore, all holding times were met for these samples.

The Test Plan and Request for Analyses / Chain of Custodies (RFA/COCs) documentation was evaluated against the sampling and analysis program for this project. The Test Plan, Table 2-3 describes three (3) test runs each having three (3) test conditions. Therefore, a total of nine (9) runs were planned. The three (3) test conditions are identified as A, B, and C. However, RFAs/COCs prepared prior to mobilization to the site only listed three samples for a single test condition (Run 1A, Run 2A, and Run 3A. The RFA/COC was edited in the field to add additional samples, and the edits have been properly noted on the original documentation. The sampling dates were included for each of the samples collected and custody/traceability was followed for all samples. All samples included on the RFA/COC were included with results in the analytical data package (ADP).

2.2.3 Instrument Performance Criteria

General Requirements: . A CO2 Bias check is included in the method and should be performed before analysis of field samples is commenced to evaluate any bias due to CO2. Each target compound should recover within + 30% of the actual standard value.

The laboratory OTM-50 work instructions require the analyst to dilute OTM-50 canisters targeting a dilution of 2-4x due to expected high levels of CO2.

Job ID 140-39565-1 Findings: The method required bias check was not performed by the laboratory prior to sample analysis. The laboratory did follow their work instruction requirement to perform dilution of canisters targeting 2-4x due to expected high levels of CO_2. Additionally, when questioned about the CO_2 bias check, the laboratory performed an independent carbon dioxide (CO_2) bias test was performed outside of the completion of these OTM-50 samples and provided information that the samples included CO_2 concentrations approximately 9% resulting in approximately 3% in the samples analyzed. The laboratory then ran analyzed two spiked tanks of gas, to determine bias. Tank 1/3 contained 5% CO_2 and tank 2 contained 10%CO2. These analyses included a limited list of analytes. The bias analyses showed Hexafluoro 16.9% recovery at a concentration of 20ppb and 11.9% recovery at a concentration of 1ppb. Tetrafluoroethane also reported 48.5% recovery at a concentration of 1ppb.

The deviation from the OTM-50 requirement for CO_2 bias check and the independent CO_2 bias test were reported by the laboratory. Based on the information provided concerning the additional bias testing

performed by the laboratory, all non-detect Hexafluoroethane results and non-detect Tetrafluoroethane results have had a UJ- qualifier, biased low, added to the data by the validator.

2.2.4 Initial and Continuing Calibrations

Requirements: A minimum of five calibration points are used for the initial calibration. The initial calibration relative standard deviations (RSDs) for the mean relative response factor (RRF) should not exceed 20% for target analytes.

The lowest calibration concentration confirmation sample should be analyzed during the initial calibration. The lowest calibration concentration should be > 3X the MDL and should be within + 30% of the target compound spike concentration.

An initial calibration verification (ICV) and continuing calibration verifications (CCVs) should be analyzed at the beginning of each sequence, after ten samples, and at the end of the sequence. Recovery of CCVs should be within + 20% of the theoretical concentrations.

Job ID 140-39565-1 Findings: The Initial calibrations met the RSD criteria for all analytes. Target analytes of the initial calibration standard were also compared to the theoretical value of the standard. There were three (3) analytes that were outside of the 20% target difference from the theoretical Value. These analytes and their nonconforming results are listed below:

Chlorotrifluoromethane: Theoretical value 0.5000 ppb (22.46%D)

Difluoropropane: Theoretical value 0.5000 ppb (20.92%D)

1,1,1-TTrifluoroethane: Theoretical value 0.5000 ppb (37.46%D)

An ICV was performed, however, a second source standard was not available. CCVs were analyzed as required. There were a few analytes that reported the CCV standard outside of the 20% difference allowed from the theoretical Value. These analytes and their nonconforming results are listed below.

Octafluoropropane Continuing Calibration 11/28/2024 at 08:35 (22.6%D)

Hexafluoropropylene Oxide Continuing Calibration 11/28/2024 at 08:35 (-23.6%D)

Hexafluoropropylene Oxide Continuing Calibration 11/29/2024 at 16:10 (-28.0%D)

Hexafluoropropylene Oxide Continuing Calibration 11/30/2024 at 08:01 (-32.8%D)

No qualifiers were applied to the data based on the initial calibration individual standards being outside 20% of the theoretical value because all RSDs were within + 20%. Also, no qualifiers were applied to the data based on the ICV variances because the ICV was performed and reported with acceptable results. However, Octafluoropropane results for all samples and method blanks (MB) except S-2063 R1A OTM-50 SILCO LINED CANISTER, S-2064 R1B OTM-50 SILCO LIN ED CANISTER, and S-2065 R1C OTM-50 SILCO LINED CANISTER were given “UJ” qualifiers by the validator, and all Hexafluoropropylene Oxide results were given “UJ” qualifiers by the validator based on CCV %D values that were greater than 20%.

2.2.5 Precision Objectives

Requirements: Method OTM-50 requires one duplicate analysis of a field sample to be analyzed with each sample sequence. If samples concentrations are > 3X the MDL, the duplicate results should be + 25% of one another.

PFAS Test Program Work Plan precision requirements specified that laboratory control sample / laboratory control sample duplicate (LCS / LCSD) and matrix spike / matrix spike duplicate (MS / MSD) relative percent difference (RPD) requirements apply as listed below:

LCS / LCSD RPDs should be < 25%D

MS / MSD RPDs should be < 25%D

Job ID 140-39565-1 Findings: Duplicates were reported for one field sample with all results for both the original and duplicate analysis reported and non-detects. There were no LCS/LCSD analyses, or MS/MSD analyses reported in association with these samples. All field sample results are reported as non-detects. Therefore, omission of LCS/LCSD and MS/MSD analyses does not affect the quality of the data. No qualifiers are being applied to the data based on this omission.

2.2.6 Accuracy Objectives

Requirements: Method Internal standard retention times must be within + 5 seconds of the most recent calibration. All internal standard responses must also be within + 40% of the average of the initial calibration or most recent continuing calibration.

PFAS Test Program Work Plan accuracy requirements specified LCS, MS and surrogate recovery requirements as listed below.

LCS recoveries should be within 70% to 130% recovery

MS recoveries should be within 10% to 130% recovery

4-Bromofluorobenzene surrogate recoveries for both the normal and the high calibration analyses should be between 60% and 140% recovery.

Job ID 140-39565-1 Findings: All internal standard and surrogate recoveries are within the required limits. There were no matrix spike or laboratory control samples analyzed in association with these field samples. No qualifiers are being applied to the data based on these omissions.

2.2.7 Blanks

Requirements: Analysis of one laboratory blank after the high initial calibration standard and at the beginning of each analytical sequence. Analytes should be <3 times the MDL or < 50% of the project required reporting limit, whichever is higher.

Analysis or one or more cleaned canisters from a given batch of clean canisters. Analytes should be <3 times the MDL or < 50% of the project required reporting limit, whichever is higher.

Job ID 140-39565-1 Findings: A method blank (laboratory blank) was performed prior to sample analysis at both the normal calibration level, and high calibration level sample analyses. All target analytes were reported as non-detects. Additionally, there were pre- and post-test background canister

samples collected prior to and following the field sample collections. Both the pre- and post- test background samples gave non-detect values during the analyses. There are a few low level tentatively identified compounds (TICs) reported in the pre- and post- background samples. No qualifiers have been added to the sample results based on these results as there are no regulatory levels established for these compounds.

2.2.8 Qualitative and Quantitative Results

Requirement: The data package has been evaluated to determine if transcription or calculation errors are present. The items related to the quality of the data that could lead to inaccuracies and have not been previously discussed are evaluated and discussed in this section.

Job ID 140-39565-1 Findings: There are no transcription errors that have been found during the review of this data package.

The target analyte results reported as “ND” (non-detects) have a “U” qualifier applied to them by the Validator.

2.3 Data Quality Summary

Overall, the data quality objectives for accuracy, precision, and completeness were met for the OTM-50 data. There are a few data quality qualifiers applied to the sample data for information provided concerning the independent CO2 bias test and CCV percent differences exceeding the 20% criteria.

Overall, the data quality for the OTN-50 analysis is deemed acceptable for its intended use.

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2024 Clean Harbors PFAS Tests
Method 0010 Data Validation Report

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Abbreviations

CCC	calibration check compound
CCV	continuing calibration verification
COC	Chain of Custody
DFTPP	decafluorotriphenylphosphine
DOD	Department of Defense
EPA	Environmental Protection Agency
GC/MS	gas chromatography/mass spectrometry
ICV	initial calibration verification
LCS	laboratory control standard
LCSD	laboratory control standard duplicate
LOQ	level of quantitation
MDL	method detection limit
MS	matrix spike
MSD	matrix spike duplicate
PFAS	per and polyfluoroalkyl substances
PICs	Product of Incomplete Combustion
QSM	Quality Systems Manual
RF	response factor
RFA	Request for Analysis
RPD	relative percent difference
RRF	relative response factor
RL	reporting limit
RSD	percent relative standard deviation
SOP	standard operating procedure
SPCC	system performance check compound
SVOC	semi-volatile organic analysis
TIC	tentatively identified compound

1 Summary

Samples submitted for validation were collected during the week of November 11th, 2024 as part of the Per and Polyfluoroalkyl Substances (PFAS) Test Program being conducted at the Clean Harbors Aragonite Hazardous Waste Incinerator in Grantsville, Utah. The samples were analyzed by the Eurofins Environment Testing USA Laboratory located in Knoxville, Tennessee. The types of samples analyzed are noted below:

- Stack Gas Semi-Volatile Fluorinated Compounds as Products of Incomplete Combustion - GC/MS (Method 0010)

The results of these analyses have been reviewed for a data quality assessment based on the laboratory's analytical method procedures, and the requirements outlined by the United States Department of Defense (DOD) Data Validation Guidelines Module 1: Data Validation Procedures for Organic Analysis by GC/MS Table B-24

2 STACK GAS SEMI-VOLATILE PRODUCTS OF INCOMPLETE COMBUSTION

2.1 Samples

The stack gas sample fractions to be analyzed for Nonpolar PFAS Semi-Volatile Organic Compounds (SVOC) were received for validation as a single data package. The stack gas samples were analyzed as required by SW-846 Method 8270 via gas chromatography/ mass spectrometry (GC/MS). All applicable compliance areas have been reviewed, and any significant findings are discussed below. Section 2.1 provides a list of the primary data quality objectives (DQOs) evaluated during this review. Section 2.2 summarizes the significant findings of the evaluation.

The following samples are included in this validation package:

Job ID 140-39573-1 List of Samples

- S-2034,2035 R1A M0010 FILTER, FH SOLVENT RINSE
- S-2036,2037 R1A M0010 XAD RESIN, BH SOLVENT RINSE
- S-2038,2039 R1A M0010 IMPINGER 1-4, IMPINGER 1-4 SOLVENT RINSE
- S-2040,2041 R2A M0010 FILTER, FH SOLVENT RINSE
- S-2042,2043 R2A M0010 XAD RESIN, BH SOLVENT RINSE
- S-2044,2045 R2A M0010 IMPINGER 1-4, IMPINGER 1-4 SOLVENT RINSE
- S-2046,2047 R3A M0010 FILTER, FH SOLVENT RINSE
- S-2048,2049 R3A M0010 XAD RESIN, BH SOLVENT RINSE
- S-2050,2051 R3A M0010 IMPINGER 1-4, IMPINGER 1-4 SOLVENT RINSE
- S-2052,2053 QA M0010 FILTER, FH SOLVENT RINSE PB
- S-2054,2055 QA M0010 XAD RESIN, BH SOLVENT RINSE PB
- S-2056,2057 QA M0010 IMPINGER 1-4, IMPINGER 1-4 SOLVENT RINSE
- S-2058 QA M0010 FILTER REAGENT BLANK
- S-2059 QA M0010 METHYLENE CHLORIDE REAGENT BLANK
- S-2060 QA M0010 ACETONE REAGENT BLANK
- S-2061 QA M0010 DI WATER REAGENT BLANK
- S-2062 QA M0010 XAD REAGENT BLANK
- A-2500 M0010 MEDIA CHECK XAD
- M0010 MEDIA CHECK FILTER.

2.2 Applicable Data Quality Objectives

2.2.1 Contract Compliance Monitoring

Requirements:

Laboratory sample data reports have been evaluated to determine if all required quality assurance and quality control measurements were performed appropriately.

Data packages have been reviewed to determine if all required reporting has been completed, and to verify that sufficient documentation has been provided as backup.

Data packages have also been reviewed to determine if the analytical procedures required by the project requirements were followed.

Job ID 140-39573-1 Findings: The analytical data was reviewed to determine if all requirements were met and there are no issues reported.

Method 0010 Sampling Trains are being utilized on this project to collect semi-volatile Products of Incomplete Combustion as the testing objective. The U.S.EPA provided guidance to the performing laboratory for the evaluation of unknown compounds in the stack gas. The emission characterizations are being conducted as non-targeted compounds evaluated as Tentatively Identified Compounds (TICs).

Several fluorinated compounds have been prepared as low-level single point calibration standards. These single point standards are used to evaluate the fragmentation patterns and retention times of some common fluorinated materials. An instrumental scan of the standard Method 8270 target analyte list was conducted, in addition to a scan of these fluorinated materials. If a fluorinated compound "hit" is observed, then it is "semi-quantified" and reported. The Method 8270 process is used to supply a quality assessment protocol for the analytical process. The standard surrogate and internal standard compounds are introduced at their usual times in the analytical procedure, and at normal concentration levels. They are used to evaluate the general data quality that will be projected onto the fluorinated "hits" as well as the tentatively identified compound (TIC) data which is evaluated completely without standards.

2.2.2 Sample Handling Criteria

Holding Time and Preservation Requirements: Sample preservation requirements are that samples should be chilled within the range of 0°C to 6°C from the time of sample collection to arrival at the laboratory. Samples are to be stored no more than 14 days from the time of sampling to the time that the extraction processes are commenced, and 40 days from extractions to analysis.

Job ID 140-39573-1 Findings: All samples were received at the laboratory at the appropriate storage temperatures which were between 0.3° and 1.5°C in the coolers received. All samples were extracted within the holding time sample storage window that ranged between 10 to 13 days from sample collection. Sample extracts were analyzed within the extract holding time which was between 33 to 37 days from extraction to the time of analysis. Therefore, samples were all properly preserved, and all holding times were met for these samples.

The Test Plan and Request for Analyses / Chain of Custody (RFA/CoCs) documentation was evaluated against the field samples collected and received at the laboratory for this project. All samples scheduled for collection in the test plan were collected and received at the laboratory for analysis. Additionally, media check samples for Filter Media and XAD-2 were set aside in the laboratory at the time of media preparation. The media checks were batched with the field samples at laboratory check-in and analyzed with the field samples. Media checks were not sent to the site but remained at the laboratory until the field samples arrived.

2.2.3 Instrument Performance Criteria

General Requirements: . An instrumental tune continuing calibration verification (CCV) is performed at the start of each 12-hour shift using decafluorotriphenylphosphine (DFTPP).

The Internal standard spikes placed onto each CCV are required to exhibit a retention time within 30 seconds of the initial calibration of the instrument, and an area count within 50% to 200% of the same initial calibration.

Subsequently, the internal standard spike placed onto each field sample or quality control sample is required to exhibit a retention time within 30 seconds of the CCV, and an area count that is within 50% to 200% of the CCV.

Job ID 140-39573-1 Findings: The DFTPP Instrument Performance Checks were performed as required at the start of each 12-hour shift. Internal standard spikes for CCVs and samples all exhibit retention times and area counts that are within the required criteria.

2.2.4 Initial and Continuing Calibrations

Requirements: A minimum of five (5) calibration points are established for the initial calibration. The Laboratory standard operating procedures (SOP) for EPA method 8270 requires that the initial calibration relative standard deviations (RSDs) for the mean relative response factor (RRF) do not exceed 30% for Calibration Check Compounds (CCCs). The minimum average response factor (RF) for System Performance Check Compounds (SPCCs) should be 0.050 or greater. A quadratic or linear curve fit may be used during the calibration establishment. A linear or non-linear curve is also allowed to be used. If a linear curve is used, the correlation coefficient must be greater than or equal to 0.990.

DOD Validation Guidelines require that analytes' initial calibration RSDs do not exceed 15%. A quadratic or linear curve may be used during calibration, and a linear or non-linear curve is allowed to be used. If a curve is used, the correlation coefficient must be greater than or equal to 0.990.

The Laboratory SOP requires an initial calibration verification (ICV) to be performed and continuing calibration verifications (CCVs) be processed at the beginning of each sequence. The recovery of ICVs and CCVs be within + 30% of the theoretical concentration for standard target analytes. The ICV and CCV CCCs RFs should be within a ± 20% difference of the initial calibration and the average RFs for SPCCs should be 0.050 or greater.

DOD Validation Guidelines require that an ICV should be reported and CCV should be reported prior to sample analysis and for every 12-hour period that samples are analyzed, and at the end of the sequence. The ICV and initial and continuing CCVs percent difference for all analytes and surrogates should be ± 20% or the initial calibration. The Closing CCV percent difference for all analytes and surrogates should be ± 50% or the initial calibration.

Job ID 140-39573-1 Findings: The Initial calibrations, ICVs, and CCVs for the standard lists of compounds met the laboratory SOP required criteria. The CCVs were performed at the beginning of each 12-hour period, but no closing CCVs were performed. Percent recoveries of all target analytes included in the multi-level calibrations were within + 20%.

The project specific fluorinated semi-volatile analytes are intentionally calibrated using a single point calibration. These analytes are reported with their method detection limits (MDLs) and reporting limits

(RLs) set at the level of the single point calibration. The single level calibration is prepared prior to each day's analysis of the Method 0010 samples. Field samples display non-detect results for all analytes included in the single point calibrations which is the objective for which they were processed. There are no negative effects on data quality and therefore, no data qualifiers have been assigned to this project specific list of analytes.

2.2.5 Precision Objectives

Requirements: The laboratory SOP for SW-846 Method 8270 includes the requirement for laboratory control sample spikes when no matrix spike / matrix spike duplicate (MS/MSD) analysis is possible as is the case with Method 0010 sampling and analysis projects. The Relative Percent Differences (RPDs) should be within the laboratory SOP limits based on historical data. DOD Validation Guidelines require that MS/MSD RPDs for all analytes be less than 20%.

Job ID 140-39573-1 Findings: As expected, there were no MS/MSD analyses performed because there is not redundant sample available for these analyses due to the sampling method. Each train fraction is used in its entirety as a sample composite. No additional sample portion is available on which to perform matrix spikes. Therefore, the LCS/LCSD analyses are performed to demonstrate precision for these Method 0010 samples. A representative set of semi-volatile compounds is used for the LCS/LCSD spikes. It is not a full analyte spike. The LCS/LCSD analyses are performed for each of the three (3) fractions of the Method 0010 sampling train. The RPDs for all spiked analytes were reported within the laboratory specified limits and the DOD required limits. No qualifiers are being applied to analytes that were not spiked since there were three (3) single point daily calibrations performed, one for each day that analyses were performed. These daily calibrations closely compared to each other, and all three (3) reported identical results. Sample results are reported as non-detects (NDs) for all fractions of all trains.

2.2.6 Accuracy Objectives

Requirements: The laboratory SOP for SW-846 Method 8270 includes a requirement for an LCS and an LCSD be performed for Method 0010 sampling train fractions since the sampling media from the sampling train is completely used in the extraction of the train fractions. Recoveries for the spiked samples are to be within the laboratory control limits based on historical data. The DOD Validation Guidelines also specify that an LCS be performed and is to meet the QSM Appendix C requirements if project requirements are not specified.

The laboratory SOP requires surrogate spikes and internal standard spikes be processed on each sample. Surrogate recoveries are to be within the laboratory required limits that are based on historical data. Internal standard responses should be within 50 – 200% of the corresponding calibration and the retention times should be within 30 seconds of the corresponding calibration.

Job ID 140-39573-1 Findings: All internal standard responses and retention times are reported within the required limits.

The LCS/LCSD recoveries associated with the Front-half and the Back-half fractions are all within laboratory specified limits. However, the LCS/LCSD recoveries associated with the condensate and Impinger content fractions are low for the target analytes listed below. The recoveries for all compounds spiked onto the LCS/LCSD samples were within the DOD Validation Guidelines. Not all spiked compounds have limits established in the DOD Validation Guidelines. The LCS results reported outside the required criteria and positive "hits" in Condensate samples associated with LCS recoveries

below have been flagged with a “J-“ qualifier, and the non-detect results have been flagged with a “UJ-” qualifier, applied by the Validator.

Analytes	LCS %R	LCSD %R	Acceptance Criteria
Aniline	**48%**	**46%**	62% - 104%
4-Chloroaniline	**61%**	**57%**	66% - 110%
2-Chlorophenol	**59%**	60% (acceptable)	60% - 100%
2,4-Dimethylphenol	**55%**	65% (acceptable)	57% - 110%
2-Nitrophenol	**63%**	**66%**	69% - 109%
Pyridine	**40%**	**40%**	49% - 95%

Five (5) of the six (6) surrogate recoveries for condensate and impinger contents samples were consistently within limits. However, the four (4) Condensate and impinger contents samples report 0% recoveries of the surrogate 2-Fluorophenol, the D.I. water reagent blank reports 44% recovery for the surrogate 2-Fluorophenol, and the method blank associated with these samples reports 26% recovery of the surrogate 2-Fluorophenol. The narrative for this data set indicates that these samples showed evidence of a matrix interferent. The interferent substance is likely derived from laboratory contamination since the method blank is also affected.

The surrogate 2-fluorophenol is referred to an “acid extractable.” The Condensate and Impinger Contents water samples are extracted twice with methylene chloride for complete removal of semi-volatile target analytes. Once after the aqueous matrix is made basic (pH>12) with sodium hydroxide (NaOH), and a second extraction after the aqueous matrix is made acidic (pH<2) with sulfuric acid (H_2SO_4). There are three (3) acid extractable surrogates, and three base-neutral surrogates used to evaluate the overall extraction efficiency of semi-volatile compounds. The compounds on the EPA Method 8270 target analyte list that are related to 2-Fluorophenol are all phenolic compounds like this surrogate. Since there are two (2) other phenolic compounds also spiked onto the samples as surrogates with the 2-fluorophenol that have not been affected by the laboratory interferent, positive target phenolic analytes results have “J-“ qualifiers applied and non-detect target phenolic analytes have “UJ-“ qualifiers applied by the Validator .

There are two (2) back-half samples that display surrogate recoveries below the laboratory target range for the surrogate acid extractable surrogate 2,4,6-Tribromophenol but within the DOD Validation Guidelines. The methylene chloride and the acetone method blank display recovery results for the base-neutral surrogate terphenyl-d14 above the laboratory required criteria but within the DOD Validation Guidelines. All sample results are reported as non-detects, therefore no qualifiers were applied to the data by the Validator.

Sample ID	Surrogates	%R
S-2036,2037 R1A M0010 XAD RESIN, BH	2,4,6-Tribromophenol	50%
S-2038,2039 R1A M0010 IMPINGER	2-Fluorophenol	0%
S-2044,2045 R2A M0010 IMPINGER	2-Fluorophenol	0%
S-2048,2049 R3A M0010 XAD RESIN, BH	2,4,6-Tribromophenol	50%
S-2050,2051 R3A M0010 IMPINGER	2-Fluorophenol	0%
S-2056,2057 QA M0010 IMPINGER	2-Fluorophenol	0%

S-2059 QA M0010 METHYLENE CHLORIDE RB	Terphenyl-d14	109%
S-2060 QA M0010 ACETONE RB	Terphenyl-d14	110%
S-2061 QA M0010 DI WATER RB	2-Fluorophenol	44%
MB 140-93560/1-B	2-Fluorophenol	26%

The sampling surrogate $^{13}C_6$-Naphthlene was applied to XAD-2 resin traps prior to sampling media being sent to the field for sample collection. Sampling surrogate recoveries for the back half fractions were reported within the laboratory criteria of 50% to 150% recovery indicating that analytes collected during sampling activities were not lost due to breakthrough, transportation to, and storage at the laboratory.

2.2.7 Blanks

Requirements: The analysis of one method blank (MB) per preparatory batch of samples is required by the laboratory and DoD. The DOD Validation Guidance requires that positive blank results be compared to positive sample results and qualified as specified in the guidance.

The PFAS Test Program Test Plan required that a blank train and reagent blanks be collected and analyzed for contamination.

Job ID 140-39573-1 Findings: A method blank (laboratory blank) was extracted with the samples for each Method 0010 sample matrix type (i.e., filter, XAD-2 resin, and Condensate).

Additionally, a complete Method 0010 blank train and Method 0010 reagent blanks were collected and processed by the laboratory for analysis. The Blank Train Back-half sample composite, (S-2054,2055 QA M0010 XAD RESIN, BH SOLVENT RINSE PB) reported Acetophenone at 10.8 µg/sample and Phenol at 3.71 J µg/sample. There were also several tentatively identified compounds (TICs) reported in the blank train samples and the reagent blanks.

There are two positive phenol results reported in association with the positive blank sample results. Samples S-2036,2037 R1A M0010 XAD RESIN, BH SOLVENT RINSE and S-2048,2049 R3A M0010 XAD RESIN, BH SOLVENT RINSE reports positive phenol results below the Reporting Limit (RL). DOD Guidance specifies that these sample results should be elevated to the RL with a “U” qualifier applied. However, since positive results in the associated blank indicate a high bias in the sample, the Validator has chosen to leave the positive results for these samples at the concentration reported and add a J+ qualifier (indicating a high bias) to the results like what is required to be added to the sample result if they are greater than the RL.

No qualifiers were added to the data based on the “small hits” reported in the Field Blank data sets.

2.2.8 Qualitative and Quantitative Results

Requirement: The data package has been evaluated to determine if transcription or calculation errors are present. The items related to the quality of the data that could lead to inaccuracies and have not been previously discussed are evaluated and discussed in this section.

Job ID 140-39573-1 Findings: There are no transcription errors that have been found during the review of this data package.

The target analyte results reported as “ND” (non-detects) have a “U” qualifier applied to them by the

Validator.

2.3 Data Quality Summary

Overall, the data quality objectives for accuracy, precision, and completeness were met for the EPA Method 0010 data. There are a few data quality qualifiers applied to the impinger fraction sample data for low surrogate recoveries of an acid extractable surrogate and low surrogate recoveries for specified analytes. There were also qualifiers applied to phenol results for two back-half fraction samples associated with low level phenol results reported in the blank train sample.

Overall, the data quality for the M0010 analysis is deemed acceptable for its intended use.

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