ENVIRONMENTAL DATA SERVICES, LTD.

DATA VALIDATION REPORT

RI PFAS Release
Red Hill Bulk Storage Facility
Joint Base Pearl Harbor-Hickam
Pearl Harbor HI FISC Site 30
CTO 23F0178

SDG: 25A0170
APPL, Inc.

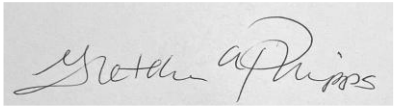
Prepared by
ENVIRONMENTAL DATA SERVICES, LTD.

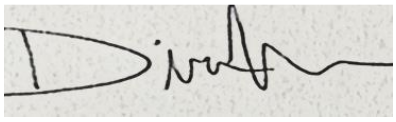
Prepared for
AECOM Environmental

Released: 03/18/25

Data Validators and Peer Reviewers:


Diane Waldschmidt


Gretchen Phipps


Dina Manov


Larry Lewis


Paloma Hoelzle

5 Brilliant Avenue, Pittsburgh, PA 15215
412.408.3288 I www.eds-pa.com

ENVIRONMENTAL DATA SERVICES, LTD.

EXECUTIVE NARRATIVE

Sample Delivery Group: 24A0170
Laboratory: APPL, Inc.
Site: RI PFAS Release, CTO 23F0178
Sampling dates: 01/28/2025
Number of Samples: 6
Test Method: USEPA Method 1633
Analysis: per- and polyfluoroalkyl substances (PFAS)

Quality Assurance Project Plan: Draft Final Remedial Investigation Work Plan Per- and Polyfluoroalkyl Substances Release Red Hill Bulk Fuel Storage Facility Joint Base Pearl Harbor-Hickam Oahu HI, Pearl Harbor HI FISC Site 30 (October 2024).

Validation Guidelines: United States Department of Defense Data Validation Guidelines Module 6: Data Validation Procedure for Per- and Polyfluoroalkyl Substances analysis by QSM Table B-24, Environmental Data Quality Workgroup, October 18, 2022; United States Department of Defense Data Validation Guidelines Modules 1, 2, 3, 4, and 6 Revised Table for Sample Qualification in the Presence of Blank Contamination, October 04, 2023; United States Department of Defense (DOD) Environmental Data Quality Workgroup (EDQW), General Validation Guidelines, November 2019.

Client Sample Identification	Laboratory Sample Identification	Matrix	Validation Stage
JV117	25A0170-01	aqueous	S4VM
JV118	25A0170-02	aqueous	S4VM
JV119	25A0170-03	aqueous	S4VM
JV120	25A0170-04	aqueous	S4VM
JV115	25A0170-05	aqueous	S4VM
JV116	25A0170-06	aqueous	S4VM

Table 1 provides a summary of the major and minor data quality issues identified in this data set. All data are acceptable except those results which have been qualified with “X”, rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an “X” flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. “X” values should not appear on any data tables even as a last resort. Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

DATA ASSESSMENT

1. NARRATIVE AND COMPLETENESS REVIEW

The case narrative was reviewed, and the data package was checked for completeness. No discrepancies were noted.

2. SAMPLE DELIVERY AND CONDITION

The samples arrived at the laboratory in acceptable condition. Proper custody was documented.

3. HOLDING TIME

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Proper sample handling and preservation also play a role in the chemical stability of analytes in the sample matrix. If samples are not collected and stored using proper containers and/or preservatives, data may not be valid.

No problems were found for this criterion.

4. CALIBRATION

Satisfactory instrument calibration is established to ensure that the instrument can produce acceptable quantitative data. An initial calibration demonstrates that the instrument can give acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance. Additionally, a continuing calibration is analyzed at the end of each 12-hour analytical sequence, denoted as a "closing" calibration verification and ascertains acceptable performance at the conclusion of the analytical sequence.

A) Initial Calibration

Percent relative standard deviation (%RSD) is calculated from the initial calibration and is used to indicate stability of a specific compound over the calibration range.

An RSD value outside the initial calibration limit indicates the potential for quantitation errors. For this reason, all positive and non-detected results are qualified as estimated. Severe performance failures (RSD >30%) requires rejection of all results. The following QC criteria have been applied for this project: The %RSD of initial calibration must be <20%.

No problems were found for this criterion.

B) Continuing Calibration

The Percent Recovery (%R) for all target analytes in the continuing calibration must be within 70-130%. All initial calibration verification (ICV) and continuing calibration verification (CCV) %Rs were with acceptance limits with the following exceptions.

No problems were found for this criterion.

C) Instrument Sensitivity Check

Prior to analysis an instrument sensitivity check (ISC) must be performed. The ISC must be at the limit of quantitation (LOQ). All analyte concentrations must be within ±30%.

No problems were found for this criterion.

5. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. When an equipment blank, or lab blank has an analyte detection, then all associated field samples are qualified per validation guidance as appropriate.

A) Method blank contamination:

No problems were found for this criterion with the following exceptions. PFOS and PFBA were positively identified in the method blank associated with all samples in this sample delivery group (SDG). The positive results reported for the impacted analytes in the associated samples have been evaluated and qualified as appropriate per validation guidance.

B) Instrument blank contamination:

No problems requiring result qualification were found for this criterion.

B) Field/Equipment blank contamination:

No samples were submitted as field / equipment blanks in association with this SDG.

6. EXTRACTED INTERNAL STANDARDS

All samples are spiked with labeled standard compounds prior to sample preparation and analyses to evaluate overall laboratory performance and efficiency of the analytical technique. The reported project samples had observed surrogate recoveries within the established limits in all cases with the following exceptions.

No problems requiring qualification of sample results were found for this criterion.

7. NON-EXTRACTED INTERNAL STANDARDS

Non-extracted internal standard peak areas are used to quantify extracted internal standard recoveries. The reported project samples had non-extracted internal standard area counts within the established limits in all cases with the following exceptions.

No problems were found for this criterion.

8. COMPOUND IDENTIFICATION

The project target analyte compounds are identified on the LC/MS/MS by using the analytes retention time (RT). The retention time of each target analyte should be within ± 0.4 minutes of the predicted retention. Target analyte detections should display a signal-to-noise of ≥ 3:1, have proper peak integration, and display all ions at the correct retention times.

Target analyte detections should have passing ion ratios (50 - 150% of theoretical). Ion ratio failures could be caused by matrix interference and/or be the result of the presence of isomers in the sample at different ratios than the ratio of isomers present in the calibration standards.

Target compound identification was verified. No anomalies were identified with the following exceptions.

The transition mass ratios for the analytes listed in the table were outside the established ratio limit during the analysis of the listed samples indicating some degree of uncertainty in the qualitative identification of the analytes. The impacted have been qualified "J" on this basis.

impacted sample	*analyte with ion ratio outside acceptance criteria*
JV115	*PFHpA*
JV117	*PFDA*
JV118	*PFHxS*
JV119	*PFPeS, PFHpA*

Manual integrations were reviewed at the Stage 4 level. No anomalies were identified.

9. COMPOUND QUANTIFICATION

Target compound quantitation was verified as part of the Level 4 data validation. No anomalies were identified.

10. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Matrix spike/matrix spike duplicate (MS/MSD) data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD data may be used in conjunction with other quality control criteria for additional qualification of data.

Sample JV115 was submitted for MS/MSD and/or matrix duplicate evaluation in association with this SDG. Upon evaluation all accuracy and precision indicators were acceptable or did not result in a need to qualify sample results.

11. FIELD DUPLICATES

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of ≤ 50% for the Relative Percent Difference (RPD) for water samples and ≤ 100% RPD for solid samples, shall be used when original and duplicate sample values are greater than or equal to the sample specific LOQ. Per project requirements validation action was not taken on this basis but a finding of the field duplicate evaluation are provided below.

No samples were submitted as a field duplicate pair in association with this SDG.

12. LABORATORY CONTROL SAMPLES

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. The LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Note: in addition to the standard LCS the laboratory has also provided a second LCS referred to as the MRL check in the laboratory report. The validator has determined that the MRL check in the laboratory's report is equivalent to the required low level LCS.

No problems were found for this criterion.

13. DILUTIONS, RE-EXTRACTIONS & REANALYSIS

Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are used.

Samples were analyzed at dilutions as necessary to bring analytical signals into the instrument's linear calibration range.

14. SYSTEM PERFORMANCE AND OVERALL ASSESSMENT

Overall, the laboratory data generated met the project goals and quality control criteria, with the exceptions identified in this report and as summarized in Table 1.

Table 1
Review Elements Summary

	Were acceptance criteria met?		
	Yes	No	
Per-fluorinated Compounds		Major	Minor
Holding Time/Sample Handling	x		
Method Blanks			x
Instrument Blanks	x		
Field Blanks	NA		
Calibration Percent Relative Standard Deviation and Percent Difference	x		
Instrument Sensitivity Check	x		
Extracted Internal Standards	x		
Non-Extracted Internal Standards	x		
Compound Identification			x
Matrix Spike/Matrix Spike Duplicate	x		
Laboratory Control Sample	x		
Other Quality Control Data out of Specification	x		
Field Duplicate	NA		

Major= Major data quality issue identified resulting in rejection of data.
Minor= Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.
NA = Not applicable

Table 2
Data Validation Qualifiers

Data Qualifier	Definition
U	**The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.**
J	**The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.**
J+	**The result is an estimated quantity, but the result may be biased high.**
J-	**The result is an estimated quantity, but the result may be biased low.**
UJ	**The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.**
X	**The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided.**

Table 3
PFAS Definitions Table

NO	CAS #	Target Name	Target Abbreviation
1	763051-92-9	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS
2	914637-49-3	2H,2H,3H,3H-Perfluorooctanoic acid	5:3FTCA
3	812-70-4	3-Perfluoroheptyl propanoic acid	7:3FTCA
4	356-02-5	3-Perfluoropropyl propanoic acid	3:3FTCA
5	919005-14-4	4,8-Dioxa-3H-perfluorononanoic acid	ADONA
6	757124-72-4	4:2 Fluorotelomer sulfonic acid	4:2 FTS
7	27619-97-2	6:2 Fluorotelomer sulfonic acid	6:2 FTS
8	39108-34-4	8:2 Fluorotelomer sulfonic acid	8:2 FTS
9	756426-58-1	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS
10	13252-13-6	Hexafluoropropylene oxide dimer acid	HFPO-DA
11	4151-50-2	N-Ethyl perfluorooctanesulfonamide	NEtFOSA
12	2991-50-6	N-Ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA
13	1691-99-2	N-Ethyl perfluorooctanesulfonamidoethanol	NEtFOSE
14	31506-32-8	N-Methyl heptadecafluorooctanesulfonamide	NMeFOSA
15	2355-31-9	N-Methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA
16	24448-09-7	N-Methyl perfluorooctanesulfonamidoethanol	NMeFOSE
17	151772-58-6	Nonafluoro-3,6-dioxaheptanoic acid	NFDHA
18	113507-82-7	Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA
19	377-73-1	Perfluoro-3-methoxypropanoic acid	PFMPA
20	863090-89-5	Perfluoro-4-methoxybutanoic acid	PFMBA
21	375-73-5	Perfluorobutanesulfonic acid	PFBASA
22	375-22-4	Perfluorobutanoic acid	PFBA
23	335-77-3	Perfluorodecanesulfonic acid	PFDS
24	335-76-2	Perfluorodecanoic acid	PFDA
25	79780-39-5	Perfluorododecanesulfonic acid	PFDoS
26	307-55-1	Perfluorododecanoic acid	PFDoA
27	375-92-8	Perfluoroheptanesulfonic acid	PFHpS
28	375-85-9	Perfluoroheptanoic acid	PFHpA
29	355-46-4	Perfluorohexanesulfonic acid	PFHXSA
30	307-24-4	Perfluorohexanoic acid	PFHxA
31	68259-12-1	Perfluorononanesulfonic acid	PFNS
32	375-95-1	Perfluorononanoic acid	PFNA
33	754-91-6	Perfluorooctanesulfonamide	PFOSA
34	1763-23-1	Perfluorooctanesulfonic acid	PFOS
35	335-67-1	Perfluorooctanoic acid	PFOA
36	2706-91-4	Perfluoropentanesulfonic acid	PFPeS
37	2706-90-3	Perfluoropentanoic acid	PFPeA
38	376-06-7	Perfluorotetradecanoic acid	PFTeDA
39	72629-94-8	Perfluorotridecanoic acid	PFTrDA
40	2058-94-8	Perfluoroundecanoic acid	PFUnA

Qualified Sample Result Summaries

Summary of Qualified Results

Sample	Lab ID	Analyte	Result	lab_qualifiers	validator_qualifiers	interpreted_qualifiers	result_unit	Reason_code
JV115	25A0170-05	Perfluoroheptanoic Acid (PFHpA)	0.053	NG_L	[2]	J	J	V
JV115	25A0170-05	Perfluorobutanoic Acid (PFBA)	0.45	NG_L	MI6 J	J+	J+	B
JV115	25A0170-05	Perfluorooctanesulfonic acid (PFOS)	0.072	NG_L	MI2 J	J+	J+	B
JV117	25A0170-01	Perfluorodecanoic Acid (PFDA)	0.14	NG_L	IR2 J	J	J	V
JV118	25A0170-02	Perfluorobutanoic Acid (PFBA)	0.61	NG_L	MI6 J	J+	J+	B
JV118	25A0170-02	Perfluorooctanesulfonic acid (PFOS)	0.17	NG_L	MI2 J	J+	J+	B
JV118	25A0170-02	Perfluorohexanesulfonic Acid (PFHxS)	0.042	NG_L	IR2 J	J	J	V
JV119	25A0170-03	Perfluoro-1-pentanesulfonate (PFPeS)	0.16	NG_L	[1]	J	J	V
JV119	25A0170-03	Perfluoroheptanoic Acid (PFHpA)	0.076	NG_L	IR1 J	J	J	V
JV119	25A0170-03	Perfluorobutanoic Acid (PFBA)	0.64	NG_L	MI6 J	J+	J+	B
JV119	25A0170-03	Perfluorooctanesulfonic acid (PFOS)	0.23	NG_L	MI2 J	J+	J+	B
JV120	25A0170-04	Perfluorooctanesulfonic acid (PFOS)	0.34	NG_L	MI2 J	J+	J+	B

Table II: Qualification Code Reference Table

Qualifier	Organics	Inorganics
H	Holding times were exceeded.	Holding times were exceeded.
S	Surrogate recovery was outside QC limits.	The sequence or number of standards used for the calibration was incorrect.
C	Calibration %RSD, r, r_2 or %D were noncompliant	Correlation coefficient is <0.995.
R	Calibration RRF was <0.05.	%R for calibration is not within control limits
B	Presumed contamination from preparation (method blank)	Presumed contamination from preparation (method) blank or calibration blank
L	Laboratory Control Sample/Laboratory Control Sample Duplicate %R or RPD was not within control limits	Laboratory Control Sample/Laboratory Control Sample Duplicate %R or RPD was not within control limits
Q	MS/MSD recovery was poor	MS/MSD recovery was poor.
E	MS/MSD or Duplicate RPD was high.	MS/MSD or Duplicate RPD or difference was high.
I	Internal standard performance was unsatisfactory	ICP ICS results were unsatisfactory.
A	Not applicable.	ICP Serial Dilution %D were not within control limits
M	Instrument Performance Check (BFB or DFTPP) was noncompliant	Not applicable.
T	Presumed contamination from trip blank.	Not applicable.
F	Presumed contamination from FB or ER.	Presumed contamination from FB or ER.
D	The analysis with this flag should not be used because another more technically sound analysis is available.	The analysis with this flag should not be used because another more technically sound analysis is available.
P	Instrument performance for pesticides was poor	Post Digestion Spike recovery was not within control limits
V	Unusual problems found with the data that have been described in the validation report where a description of the problem can be found.	Unusual problems found with the data that have been described in the validation report where a description of the problem can be found.

Calculation Documentation

Internal Standard Initial Calibration and Calculation Worksheet

Lab: APPL
Method: 1633
Instrument: Vhagar
Curve Date: 2/3/2025
Compound: PFOS
Internal Standard: 13C8-PFOS

Initial Calibration Model Worksheet

Compound Area Ax	ISTD Area Ais	Compound Conc Cx	ISTD Conc Cis	Y-Values Ax/Ais	X-Values Cx/Cis	X^2 $(Cx/Cis)^2$	RF (Ax*Cis)/(Ais*Cx)
45189	730615	0.1	2	0.061850633	0.05	0.0025	1.237
209352	739493	0.5	2	0.283102071	0.25	0.0625	1.132
383993	794734	1	2	0.483171728	0.5	0.25	0.966
834344	777969	2	2	1.072464327	1	1	1.072
1839304	739870	5	2	2.485982673	2.5	6.25	0.994
3711453	675724	10	2	5.492557612	5	25	1.099
7440586	691737	20	2	10.75637995	10	100	1.076
17468679	616636	50	2	28.32899636	25	625	1.133
SUM OF EACH COLUMN :				**48.9645**	**44.3**	**757.565**	**8.7099**

CALIBRATION MODELS:

Average Response Factor:
Cx = Ax*Cis/Ais/RF

Average RF	1.089	**AVERAGE(RF)**	1.17749
RSD	7.8%	**STDEV(RF)/(AveRF)**	7.8

Linear Regression:
y = mx + b
Cx = (((Ax/Ais)-b)/m)*Cis

Weighting	Equal	1/X	$1/X^2$	Equation
Slope (m)	1.13169	1.10620	1.05840	**SLOPE(RatioY,RatioX)**
Intercept (b)	-0.14618	-0.00504	0.008749	**INTERCEPT(RatioY,RatioX)**
CC (R)	0.99977	0.99932	0.99817	**CORREL(RatioY,RatioX)**
COD (R^2)	0.99954	0.99863	0.99634	**POWER(R,2)**

Quadratic Regression:
$y = ax^2 + bx + c$
Cx=(SQRT(b^2-(4*a*(c-(Ax/Ais))))-b)/(2*a)*Cis

Weighting	Equal	1/X	$1/X^2$	Equation
x^2 Coefficient (a)	0.00339	0.00465	-0.00526	**LINEST(RatioY,RatioX:RatioX^2,1,1)**
x Coefficient (b)	1.04848	1.01758	1.19956	**INDEX(LINEST(RatioY,RatioX:RatioX^2,1,1),1,2)**
Intercept (c)	-0.00648	0.04542	-0.02355	**INDEX(LINEST(RatioY,RatioX:RatioX^2,1,1),1,3)**
COD (R^2)	0.99991			**INDEX(LINEST(RatioY,RatioX:RatioX^2,1,1),3,1)**

Sample Concentration Calculations

Sample ID	File ID	Compound Area Ax	ISTD Area Ais	ISTD Conc Cis	Ave RF On-column Conc	Linear Cal On-column Conc Equal Weighting	Linear Cal On-column Conc 1/X Weighting	Linear Cal On-column Conc $1/X^2$ Weighting	Quadratic Cal On-column Conc Equal Weighting	Quadratic Cal On-column Conc 1/X Weighting	Quadratic Cal On-column Conc $1/X^2$ Weighting		%D
Equations:					Ax*Cis/Ais/RF	((Ax/Ais-b)/m)*Cis			(SQRT(b^2-(4*a*(c-(Ax/Ais))))-b)/(2*a)*Cis				
SE00384-SCV1	V2025-02-03A (10)	1567165	772215	2	3.728	3.845	3.678	3.818	3.859	3.865	3.449	3.4577	1.881155
BEB0017-BLK1	V2025-02-06A (6)	8700	857251	2	0.019	0.276	0.027	0.003	0.032	-0.069	0.056	0.017	2.304841
BEB0017-BS1	V2025-02-06A (7)	495491	823100	2	1.106	1.322	1.097	1.121	1.158	1.091	1.045	1.026	1.872339
SE00428-CCV2	V2025-02-06A (23)	1733569	738446	2	4.312	4.407	4.254	4.420	4.458	4.479	3.988	4	1.879615
25A0170-05	V2025-02-06A (20)	10893	916906	2	0.022	0.279	0.031	0.006	0.035	-0.066	0.059	0.02	2.180198
25A0170-05 MS	V2025-02-06A (11)	500960	911346	2	1.010	1.230	1.003	1.022	1.059	0.989	0.958	0.937	1.86914
25A0170-05 MSD	V2025-02-06A (12)	504536	830226	2	1.116	1.332	1.108	1.132	1.169	1.102	1.055	1.035	1.890714
					#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!		
					#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
					#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
					#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
					#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		

Internal Standard Initial Calibration and Calculation Worksheet

Lab: APPL
Method: 1633
Instrument: Vhagar
Curve Date: 2/3/2025
Compound: 13C8-PFOS
Internal Standard: 13C4-PFOS

Initial Calibration Model Worksheet

Compound Area Ax	ISTD Area Ais	Compound Conc Cx	ISTD Conc Cis	Y-Values Ax/Ais	X-Values Cx/Cis	X^2 $(Cx/Cis)^2$	RF (Ax*Cis)/(Ais*Cx)
730615	309891	2	1	2.357651561	2	4	1.179
739493	309115	2	1	2.392290895	2	4	1.196
794734	311486	2	1	2.551427673	2	4	1.276
777969	277292	2	1	2.805594824	2	4	1.403
739870	298161	2	1	2.481444589	2	4	1.241
675724	284869	2	1	2.372051715	2	4	1.186
691737	286833	2	1	2.411636736	2	4	1.206
616636	245431	2	1	2.512461751	2	4	1.256
SUM OF EACH COLUMN :				19.8846	16	32	9.9423

CALIBRATION MODELS:

Average Response Factor:
Cx = Ax*Cis/Ais/RF

Average RF	1.243	AVERAGE(RF)	1.24278
RSD	5.9%	STDEV(RF)/(AveRF)	5.9

Linear Regression:

y = mx + b
Cx = (((Ax/Ais)-b)/m)*Cis

Weighting	Equal	1/X	$1/X^2$	Equation
Slope (m)	#DIV/0!	#DIV/0!	#DIV/0!	SLOPE(RatioY,RatioX)
Intercept (b)	#DIV/0!	#DIV/0!	#DIV/0!	INTERCEPT(RatioY,RatioX)
CC (R)	#DIV/0!	#DIV/0!	#DIV/0!	CORREL(RatioY,RatioX)
COD (R^2)	#DIV/0!	#DIV/0!	#DIV/0!	POWER(R,2)

Quadratic Regression:

$y = ax^2 + bx + c$
Cx=(SQRT(b^2-(4*a*(c-(Ax/Ais))))-b)/(2*a)*Cis

Weighting	Equal	1/X	$1/X^2$	Equation
x^2 Coefficient (a)	0.00000	#DIV/0!	#DIV/0!	LINEST(RatioY,RatioX:RatioX^2,1,1)
x Coefficient (b)	0.00000	#DIV/0!	#DIV/0!	INDEX(LINEST(RatioY,RatioX:RatioX^2,1,1),1,2)
Intercept (c)	2.48557	#DIV/0!	#DIV/0!	INDEX(LINEST(RatioY,RatioX:RatioX^2,1,1),1,3)
COD (R^2)	0.08905			INDEX(LINEST(RatioY,RatioX:RatioX^2,1,1),3,1)

Sample Concentration Calculations

Sample ID	File ID	Compound Area Ax	ISTD Area Ais	ISTD Conc Cis	Ave RF On-column Conc	Linear Cal On-column Conc Equal Weighting	Linear Cal On-column Conc 1/X Weighting	Linear Cal On-column Conc $1/X^2$ Weighting	Quadratic Cal On-column Conc Equal Weighting	Quadratic Cal On-column Conc 1/X Weighting	Quadratic Cal On-column Conc $1/X^2$ Weighting	
Equations:					Ax*Cis/Ais/RF	((Ax/Ais-b)/m)*Cis			(SQRT(b^2-(4*a*(c-(Ax/Ais))))-b)/(2*a)*Cis			
SE00384-SCV1	V2025-02-03A (10)	772215	300753	1	2.066	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	2.066
BEB0017-BLK1	V2025-02-06A (6)	857251	362295	1	1.904	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	1.904
BEB0017-BS1	V2025-02-06A (7)	823100	331487	1	1.998	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	1.998
SE00428-CCV2	V2025-02-06A (23)	738446	289646	1	2.051	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	2.051
25A0170-05	V2025-02-06A (20)	916906	358297	1	2.059	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	2.059
25A0170-05 MS	V2025-02-06A (11)	911346	381194	1	1.924	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	1.924
25A0170-05 MSD	V2025-02-06A (12)	830226	346772	1	1.926	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	1.926
					#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#DIV/0!	#DIV/0!	
					#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
					#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
					#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	
					#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	

Final Sample Result Calculation
PFAS
method 1633
APPL

on column result (ng/ml) x final volume(ml)/initial sample amount (g) x 1 g/ 1 ml x 1000g/1 ml x dilution factor = calculated result

density of water = 1g/1ml

Sample	Analyte	On column results (ng/ml)	Final Prep Volume (ml)	Initial Sample amount (g)	Dilution Factor	Calculate result (ng/L)	Reported Result (ng/L)
25A0170-05	PFOS	0.02	2	560.503	1	0.071364471	0.072

Data Validation Worksheet

DATA VALIDATION PFAS

Module 6; PFAS by QSM Table 5-24; October 18, 2022

Validator: GAP

Reviewer: DM

Date Validated: 03/13/2025

Reviewed: 3/18/2025

Project: Red Hill

SDG: 25A0170

LAB: APPL

Samples Collected: 01/28/2025

8 aqueous

SAMPLE RECEIPT AND CASE NARRATIVE REVIEW

- ✓ Traffic reports, chain-of-custody forms or SDG narrative do not indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data.
- ✓ AFFF samples are to be shipped in HDPE containers with an unlined cap
- ✓ Shipment temp 0-6°C: recommended to freeze tissue samples upon receipt
- ✓ If temp upon receipt is greater than 6°C J/UJ all

  Received on 1/30 at 0.7 and 0.5°C

HOLDING TIMES

- ✓ Recommended storage temp is ≤ -20°C
- ✓ Per method 1633: aqueous samples may be held in the lab for up to 90 days when stored at recommended temp and protected from light; when stored at 0-6 °C and protected from light samples can be held for up to 28 days (see method for additional details)
- ✓ Per method 1633: solid samples may be held in the lab for up to 90 days when stored at recommended temp or 0-6 °C (see method for additional details)
- ✓ Per method 1633: biosolid samples may be held in the lab for up to 90 days when stored at recommended temp or 0-6 °C; however, freezing is recommended (see method for additional details)
- ✓ Samples extracts should be stored at 0-4°C protected from light and analyzed within 90 days

- ✓ If hold time is exceeded qualify J/UJ
- ✓ If hold time is grossly exceeded (2X hold time) J/X

244 Table II. Sample Storage and Holding Time Requirements

Matrix Type	Stored at 0 - 6°C, protected from light		Stored at ≤ -20°C, protected from light	
	Holding Time	Caveat	Holding Time	Caveat
Aqueous	28 days	Precursor degradation occurs after 7 days	90 days	None
Solid and Tissue	90 days	Should be prepared as soon as possible if NFDHA is a target analyte	90 days	Should be prepared as soon as possible if NFDHA is a target analyte
Biosolid	90 days	Not recommended due to the production of gases due to microbiological activity	90 days	None

Samples collected 1/28/2025
Extracted 02/04/2025
Analyzed 02/06/2025 and 02/07/2025

All ok

Extracted Internal STANDARDS

- ✓ Added to all QC and field samples
- ✓ Recoveries are within the limits as defined in QAPP; otherwise QSM criteria (20-150%) should be used
- ✓ Detected for analytes qualified using an EIS percent recovery >200% should be qualified J-. Nod-detects should not be qualified.
- ✓ If EIS recovery is <10%; associated detected and non-detects should be qualified X
- ✓ EIS retention times should be within 0.4 minutes of standard; use professional judgment to qualify

Per QAPP:

QC Sample	Number	Method/SOP QC Acceptance Limits
EIS	Every field sample, standard, blank, and QC sample.	Field and QC samples EIS compound recoveries must be within the acceptance limit specified for the matrix of the sample provided by the method (Tables 5, 6, 7, and 8). In addition to the requirements of EPA Method 1633, the following must be met for analytes not included in EPA Method 1633: 1) QC samples and field samples must recover within in-house limits. Preliminary in-house acceptance criteria of 20%–150% must be used until in-house limits are generated in accordance with Section 9.4 of EPA Method 1633. 2) The lower limit of inhouse acceptance criteria cannot be < 20%. 3) Must meet laboratory-derived limits.

Lab limits used to evaluate with the exception of lower limits <20%. 20% was used as lowest acceptance limit in those instances.

All ok except

JV116 (25A0170-06) 13C2-6:2FTS ↑ initial analysis

Reported from 100X; no action

Non-Extracted Internal STANDARDS

- ✓ Used to quantify EIS
- ✓ If low area counts are reported (<30%) detected and non-detected should be qualified X

All ok

Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (LLLCS) (MRL in APPL data package)

- ✓ LCMS Lab Control Recovery (Form III), Form I, prep log, run log
- ✓ LCS prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per SDG.
- ✓ Laboratory Control Samples were analyzed for all the target analytes that the samples are analyzed for.
- ✓ Use limits as defined in QAPP; otherwise lab limits or QSM criteria of 40-150%.
- ✓ If LCS or LLLCS %R is > upper limit; qualify detects J+; no action on non-detected
- ✓ If LCS or LLLCS %R is < lower limit; qualify detected J- and non-detected X

Use lab limits to evaluate

All 40 compounds included.

MRL Check (BEB0017-MRL1) all ok
LCS and LCSD (BEB0017-BS1) all ok

MS/MSD and Matrix Duplicate

- ✓ LCMS Matrix Spike Recovery (Form III)
- ✓ The Matrix Spike Samples were spiked and analyzed for all the target analytes that the samples are analyzed for (Same analytes as LCS).
- ✓ Per module 6: MS and MSD are applicable where the spike concentration is a least 3 times greater than the native analyte concentration **(3X rule)**
- ✓ Use limits as defined in QAPP; otherwise lab limits or QSM criteria of 40-150%.
- ✓ If MS or MSD %R is > upper limit; qualify detects J+; no action on non-detected
- ✓ If MS or MSD %R is < lower limit but >10%; qualify detected J- and non-detected UJ
- ✓ If MS or MSD %R is < 10%; qualify detected J- and non-detected X
- ✓ If MS/MSD RPD is out; qualify detected J and non-detected UJ
- ✓ For matrix duplicate; for concentrations of analytes that are equal to or greater than the LOQ, the RPD must be ≤30%; if out qualified detected J; no action on non-detects

Use lab limits to evaluate
Sample: **25A0170-05 JV115 MS/MSD all ok**

BLANKS

- ✓ LCMS Method Blank Summary (Form IV), method blank Form I, prep log, run log
- ✓ Frequency of Analysis: method blank has been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch.
- ✓ Continuing Calibration Blanks (Form I) and run log
- ✓ Frequency of Analysis: immediately following the highest standard analyzed and daily prior to sample analysis.
- ✓ Field/rinse blanks are non-detected for all analytes

United States Department of Defense Data Validation Guidelines Modules 1, 2, 3, 4, and 6 Revised Table for Sample Qualification in the Presence of Blank Contamination, October 04, 2023:

Table A: Sample Qualification in the Presence of Blank Contamination

	Sample		
Row Number	Result	Validated Result	Validation Qualifier
1	non-detect or detect ≤ LOD	Report at LOD	U
2	> LOD and ≤ 5x blank	Report at Sample Result	J+
3	> 5x blank	Report at Sample Result	None

LOD = Limit of Detection

FB/EBs
none

Blank (BEB0017-BLK1)
PFOS 0.0692 J ng/L results >LOD but <5X flag J+
PFBA 0.289 J ng/L results >LOD but <5X flag J+

ICBs/CCBs see below

MASS CALIBRATION

- ✓ Verified to be ±0.2 amu of true value

Bile Salt Interference Check and Qualitative Identification Standard

- ✓ Provided and requirements met
- ✓ See Module 6

acceptable

ICAL

- ✓ Initial Calibration Data Curve Evaluation (Form VI) and run log
- ✓ Lowest standard should be at or below LOQ
- ✓ %RSD <20% or relative standard error (RSE) <20%
- ✓ If %RSD > 20% but <30% J/UJ
- ✓ If %RSD >30% J/R

See below

INSTRUMENT PERFORMANCE CHECK PER DRAFT METHOD 1633 (LCV in APPL data package)

- ✓ Concentration equal to LOQ
- ✓ Analyzed after ICAL and daily before samples
- ✓ If not analyzed all associated data should be qualified X
- ✓ The %R for ICV and CCV 30%; if out >130% qualify positive J+ and nondetected UJ; if out <70% qualify positives J- and nondetects UJ
- ✓ Per module if gross exceedances of recoveries <50% or >150%; qualify all associate data X

CCAL

- ✓ Continuing Calibration Data (Form VII) and run log
- ✓ Continuing calibration standard analyzed on each working day, prior to sample analyses.
- ✓ Calibration verification/continuing calibration standard been analyzed after every 10 samples and at the end of each analytical sequence
- ✓ If not analyzed all associated data should be qualified X
- ✓ The %R for ICV and CCV 30%; if out >130% qualify positive J+ and nondetected UJ; if out <70% qualify positives J- and nondetects UJ
- ✓ Per module if gross exceedances of recoveries <50% or >150%; qualify all associate data X

LCV is the method required ISC
70-130%

Instrument Vhagar

02/03/25 all %RSE <20%

Initial Cal Blank SE00384-ICB1 V2025-02-03A (9) 02/03/25 20:45
Secondary Cal Check SE00384-SCV1 V2025-02-03A (10) 02/03/25 21:06

Calibration Blank SE00428-CCB1 V2025-02-06A (1) 02/06/25 12:00
Low Cal Check SE00428-LCV1 V2025-02-06A (2) 02/06/25 12:21
Calibration Check SE00428-CCV1 V2025-02-06A (3) 02/06/25 12:42
Performance Mix SE00428-PEM1 V2025-02-06A (4) 02/06/25 13:02
Calibration Blank SE00428-CCB2 V2025-02-06A (5) 02/06/25 13:23

Samples 1-6

Calibration Check SE00428-CCV2 V2025-02-06A (23) 02/06/25 19:37
Calibration Blank SE00428-CCB3 V2025-02-06A (24) 02/06/25 19:57

Calibration Blank SE00443-CCB1 V2025-02-07A (1) 02/07/25 14:06
Low Cal Check SE00443-LCV1 V2025-02-07A (2) 02/07/25 14:26
Calibration Check SE00443-CCV1 V2025-02-07A (3) 02/07/25 14:47
Performance Mix SE00443-PEM1 V2025-02-07A (4) 02/07/25 15:08
Calibration Blank SE00443-CCB2 V2025-02-07A (5) 02/07/25 15:29

PFBA 0.10 J ng/ml not reported in associated sample

Samples 6RE16RE2

Calibration Check SE00443-CCV2 V2025-02-07A (11) 02/07/25 17:33
Calibration Blank SE00443-CCB3 V2025-02-07A (12) 02/07/25 17:54
PFBA 0.10 J ng/ml not reported in associated sample

COMPOUND INDENTIFICATION

- ✓ RT within ±0.4 RRT units (review for Level 4)
- ✓ S/N ration 3:1 (review for Level 4)
- ✓ Ion response ratio with ±50% (review for Level 2B)
- ✓ If ion ratio is outside limit; qualify J

AECOM DVA SOP Reason Code: V

JV115	PFHpA	flag J
JV117	PFDA	flag J
JV118	PFHxS	flag J
JV119	PFPeS, PFHpA	flag J

FIELD DUPLICATES

- ✓ Use QAPP defined criteria
- ✓ If outside acceptance criteria qualify J/UJ (MODULE FLAGS NONDETECTS TOO)

Per QAPP: Do not qualify based on FD; note in report

RPD ≤50% water [c]
RPD ≤100% soil/sediment (judgmental) [c]

[c] Per Section II, *Data Validation Procedures* (DON 2015). For analytes measured above the LOQ, the MPC is 50%. Results below the LOQ or non-detected are estimates, and RPD exceedances at these levels do not significantly impact data quality. For field duplicates above the LOQ, if RPDs exceed 50%, no qualification is necessary, but RPDs and absolute differences should be noted in the data validation summary. Discussions of RPDs exceeding the MPC will be included in the data usability assessment as described in Worksheet #37. Assessment of field duplicate precision will be evaluated in the context of detected concentrations, reporting limits, and screening levels.

none