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: 24J0258
APPL, Inc.

Prepared by
ENVIRONMENTAL DATA SERVICES, LTD.

Prepared for
AECOM Environmental

Released: 12/13/24

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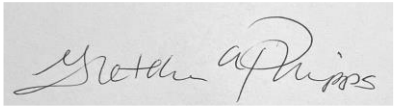
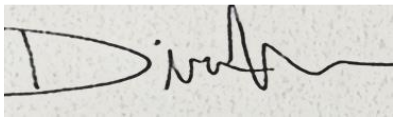
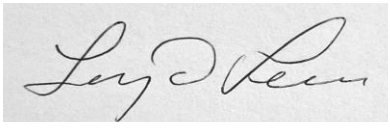
Diane Waldschmidt


Gretchen Phipps


Dina Manov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ENVIRONMENTAL DATA SERVICES, LTD.

EXECUTIVE NARRATIVE

Sample Delivery Group: 24J0258
Laboratory: APPL, Inc.
Site: RI PFAS Release, CTO 23F0178
Sampling dates: 10/18/2024, 10/21/2024, and 10/23/2024
Number of Samples: 10
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
JV035	24J0258-01	aqueous	S2BVM
JV036	24J0258-02	aqueous	S2BVM
JV037	24J0258-03	aqueous	S2BVM
JV062	24J0258-04	aqueous	S2BVM
JV038	24J0258-05	aqueous	S2BVM
JV039	24J0258-06	aqueous	S2BVM
JV042	24J0258-07	aqueous	S2BVM
JV045	24J0258-08	aqueous	S2BVM
JV043	24J0258-09	aqueous	S2BVM
JV044	24J0258-10	aqueous	S2BVM

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.

B) Instrument blank contamination:

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

B) Field/Equipment blank contamination:

No samples were submitted as field / equipment blanks in association with the samples in this sample delivery group (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 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 PFHxA and PFOS in sample JV035 were outside the established ratio limit indicating some degree of uncertainty in the qualitative identification of the analyte. The result reported for the impacted analytes in the aforementioned sample have been qualified "J" on this basis.

The transition mass ratios for PFHxA in sample JV037 was outside the established ratio limit indicating some degree of uncertainty in the qualitative identification of the analyte. The result reported for the impacted analyte in the aforementioned sample has been qualified "J" on this basis.

The transition mass ratio for PFOS in sample JV038 was outside the established ratio limit indicating some degree of uncertainty in the qualitative identification of the analyte. The result reported for the impacted analyte in the aforementioned sample has been qualified "J" on this basis.

The transition mass ratios for PFHxS in sample JV042 was outside the established ratio limit indicating some degree of uncertainty in the qualitative identification of the analyte. The result reported for the impacted analyte in the aforementioned sample has been qualified "J" on this basis.

Manual integrations were not reviewed at the Stage 2B level.

9. COMPOUND QUANTIFICATION

Target compound quantitation was not verified as part of the Level 2B data validation.

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.

No sample was submitted for MS/MSD and/or matrix duplicate evaluation in association with this SDG.

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.

Samples JV037 and JV062 were submitted as a field duplicate pair in association with this SDG. Upon evaluation, adequate field precision was demonstrated.

Samples JV038 and JV039 were submitted as a field duplicate pair in association with this SDG. Upon evaluation, adequate field precision was demonstrated.

Samples JV043 and JV044 were submitted as a field duplicate pair in association with this SDG. Upon evaluation, adequate field precision was demonstrated.

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.

No re-extractions, dilutions or reanalyses were provided for data review.

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	NA		
Laboratory Control Sample	x		
Other Quality Control Data out of Specification	x		
Field Duplicate	x		

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
JV035	24J0258-01	Perfluorooctanesulfonic acid (PFOS)	0.16	IR1 J	J	J	NG_L	V
JV035	24J0258-01	Perfluorohexanoic Acid (PFHxA)	0.083	IR2 J	J	J	NG_L	V
JV037	24J0258-03	Perfluorohexanoic Acid (PFHxA)	0.16	IR2 J	J	J	NG_L	V
JV038	24J0258-05	Perfluorooctanesulfonic acid (PFOS)	0.13	IR1 J	J	J	NG_L	V
JV042	24J0258-07	Perfluorohexanesulfonic Acid (PFHxS)	0.20	IR1 J	J	J	NG_L	V

Summary of Qualified Results

Sample	Lab ID	Analyte	Result	lab_qualifiers	validator_qualifiers	interpreted_qualifiers	result_unit	Reason_code

Summary of Qualified Results

Sample	Lab ID	Analyte	Result	lab_qualifiers	validator_qualifiers	interpreted_qualifiers	result_unit	Reason_code

Summary of Qualified Results

Sample	Lab ID	Analyte	Result	lab_qualifiers	validator_qualifiers	interpreted_qualifiers	result_unit	Reason_code

Summary of Qualified Results

Sample	Lab ID	Analyte	Result	lab_qualifiers	validator_qualifiers	interpreted_qualifiers	result_unit	Reason_code

Summary of Qualified Results

Sample	Lab ID	Analyte	Result	lab_qualifiers	validator_qualifiers	interpreted_qualifiers	result_unit	Reason_code

Summary of Qualified Results

Sample	Lab ID	Analyte	Result	lab_qualifiers	validator_qualifiers	interpreted_qualifiers	result_unit	Reason_code

Summary of Qualified Results

Sample	Lab ID	Analyte	Result	lab_qualifiers	validator_qualifiers	interpreted_qualifiers	result_unit	Reason_code

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

Field Duplicate Calculations
this pair was incorrect in the DVA

Parameters	Original Sample	Duplicate Sample			Flag
	JV038	JV039	RPD	LOQ	
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)			#DIV/0!	0.73	
1H,1H,2H,2H-perfluorohexane sulfonate (4:2 FTS)			#DIV/0!	1.5	
1H,1H,2H,2H-perfluorooctane sulfonate (6:2 FTS)	4.6	4.8	4.3	1.5	
3:3 Fluorotelomer carboxylic acid			#DIV/0!	1.5	
4,8-dioxa-3H-perfluorononanoic acid (ADONA)			#DIV/0!	0.73	
5:3 Fluorotelomer carboxylate			#DIV/0!	1.5	
7:3 Fluorotelomer carboxylate			#DIV/0!	1.5	
8:2 Fluorotelomer Sulfonate			#DIV/0!	1.5	
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)			#DIV/0!	0.73	
Hexafluoropropylene oxide dimer acid (HFPO-DA)			#DIV/0!	0.73	
N-Ethyl Perfluorooctane Sufonamidoacetic Acid (NEtFOSAA)			#DIV/0!	0.36	
N-Ethyl Perfluorooctane Sulfonamide (NEtFOSA)			#DIV/0!	1.5	
N-Ethyl Perfluorooctane Sulfonamidoethanol (NEtFOSE)			#DIV/0!	1.5	
N-Methyl Perfluorooctane Sulfonamide (NMeFOSA)			#DIV/0!	1.5	
N-Methyl Perfluorooctane Sulfonamidoacetic Acid			#DIV/0!	0.36	
N-Methyl Perfluorooctane Sulfonamidoethanol (NMeFOSE)			#DIV/0!	1.5	
Perfluoro(2-ethoxyethane)sulfonic acid			#DIV/0!	0.73	
Perfluoro-1-nonanesulfonic acid (PFNS)			#DIV/0!	0.36	
Perfluoro-1-pentanesulfonate (PFPeS)			#DIV/0!	0.36	
Perfluoro-3,6-dioxaheptanoic acid			#DIV/0!	0.73	
Perfluoro-3-methoxypropanoic acid			#DIV/0!	0.73	
Perfluoro-4-methoxybutanoic Acid			#DIV/0!	0.73	
Perfluorobutanesulfonic Acid (PFBS)			#DIV/0!	0.36	
Perfluorobutanoic Acid (PFBA)	0.84	0.81	3.6	1.5	
Perfluorodecanesulfonic Acid (PFDS)			#DIV/0!	0.36	
Perfluorodecanoic Acid (PFDA)			#DIV/0!	0.36	
PERFLUORODODECANESULFONIC ACID (PFDoDS)			#DIV/0!	0.36	
Perfluorododecanoic Acid (PFDoA)			#DIV/0!	0.36	
Perfluoroheptanesulfonic acid (PFHpS)			#DIV/0!	0.36	
Perfluoroheptanoic Acid (PFHpA)	0.22	0.23	4.4	0.36	
Perfluorohexanesulfonic Acid (PFHxS)		0.057	200.0	0.36	ok; results <LOQ; diff LOQ
Perfluorohexanoic Acid (PFHxA)	1.1	1.2	8.7	0.36	
Perfluorononanoic Acid (PFNA)			#DIV/0!	0.36	
Perfluorooctane Sulfonamide (PFOSA)			#DIV/0!	0.36	
Perfluorooctanesulfonic acid (PFOS)	0.13	0.27	70.0	0.36	ok; results <LOQ; diff LOQ
Perfluorooctanoic Acid (PFOA)			#DIV/0!	0.36	
Perfluoropentanoic Acid (PFPA) (PFPeA)	3.8	3.8	0.0	0.73	
Perfluorotetradecanoic Acid (PFTA)			#DIV/0!	0.36	
Perfluorotridecanoic Acid (PFTrDA)			#DIV/0!	0.36	
Perfluoroundecanoic Acid (PFUnA)			#DIV/0!	0.36	

Data Validation Worksheet

DATA VALIDATION PFAS

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

Validator: GAP

Reviewer: DLW

Date Validated: 12/10/2024

Reviewed: 12/13/24

Project: Red Hill

SDG: 24J0258

LAB: APPL

Samples Collected: 10/18/2024, 10/21/2024, and 10/23/2024

10 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 10/26 at 2.5, 2.2, and 3.2°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 10/18/2024, 10/21/2024, and 10/23/2024
Extracted 10/29/2024
Analyzed 10/31/2024

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

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 (BDJ0601-MRL1) all ok
LCS (BDJ0601-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: **None**

Analyte	MS	MSD	RPD	flag

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 (BDJ0601-BLK1)
ND

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 Fafnir

10/22/24 all %RSE <20%

Initial Cal Blank SD04358-ICB1 F2024-10-22A (9) 10/22/24 17:58
NMeFOSAA 0.04 J ND; no action
NEtFOSAA 0.04 J ND; no action

Secondary Cal Check SD04358-SCV1 F2024-10-22A (11) 10/22/24 18:40

Calibration Blank SD04486-CCB1 F2024-10-31A (1) 10/31/24 10:19
Low Cal Check SD04486-LCV1 F2024-10-31A (2) 10/31/24 10:40

Calibration Check SD04486-CCV2 F2024-10-31A (18) 10/31/24 17:25
Calibration Blank SD04486-CCB3 F2024-10-31A (19) 10/31/24 17:47

All samples

Calibration Check SD04486-CCV3 F2024-10-31A (31) 10/31/24 22:01
Calibration Blank SD04486-CCB4 F2024-10-31A (32) 10/31/24 22:22

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

All ok except

JV035 PFHxA J

PFOS J

JV037 PFHxA J

JV038 PFOS J

JV042 PFHxS 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.

JV043 and JV044 ok results <RL; diff <RL; no action

JV037 and JV062 ok results <RL; diff <RL; no action

JV038 and JV039 ok results <RL; diff <RL; no action