



DATA VALIDATION REPORT

**Red Hill Bulk Fuel Storage Facility
Joint Base Pearl Harbor-Hickam
CV 23F0104**

**SDG: 22L0033
APPL, INC.**

Prepared by
ENVIRONMENTAL DATA SERVICES, LTD.

Prepared for
AECOM Environmental

Rev01 Released: 12/16/22

Data Validators and Peer Reviewers:

A handwritten signature in black ink, appearing to read "Diane Waldschmidt".

Diane Waldschmidt

A handwritten signature in black ink, appearing to read "Gretchen Phipps".

Gretchen Phipps

A handwritten signature in black ink, appearing to read "Joshua Ley".

Joshua Ley

A handwritten signature in black ink, appearing to read "Dina Manov".

Dina Manov

A handwritten signature in black ink, appearing to read "Larry Lewis".

Larry Lewis

EXECUTIVE NARRATIVE

Sample Delivery Group: 22L0033

Laboratory: APPL, Inc.

Site: Red Hill Bulk Storage Facility, CV 23F0104

Sampling dates: 12/5/2022

Number of Samples: 2

Test Method: USEPA Method 1633

Analysis: per- and polyfluoroalkyl substances (PFAS)

Quality Assurance Project Plan: Sampling and Analysis Plan, Investigation and Remediation of Releases and Groundwater Protection and Evaluation, Red Hill Bulk Fuel Storage Facility, Joint Base Pearl Harbor-Hickam, O’ahu, Hawai’i (Revision 01, April 2017); PFAS-Specific Sampling and Analysis plan, Red Hill Bulk Fuel Storage Facility, Adit 6, Joint Base Pearl Harbor-Hickam, O’Ahu, Hawai’i (November 30, 2022) (SAP).

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 (DOD) Environmental Data Quality Workgroup (EDQW), General Validation Guidelines, November 2019.

Client Sample Identification	Laboratory Sample Identification	Matrix	Validation Stage
AF-RHMW03-WGN01LF-2212W1	22L0033-01	groundwater	S4VEM
AF-RHMW02-WGN01LF-2212W1	22L0033-02	groundwater	S4VEM

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 with the following exceptions.

The observed recovery for NFDHA was outside of acceptance limits for a CCV associated with all samples in this sample delivery group (SDG). The results reported for the impacted analyte in the associated samples have been qualified estimated "UJ" on this basis.

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 $\pm 30\%$ of the true value. Note: the laboratory reports refer to the ISC as Low-Concentration Calibration Verification (LCCV). The validator has determined that the LCCV in the laboratory's report is equivalent to the method required ISC.

No problems were found for this criterion with the following exceptions.

The observed recoveries for NETFOSAA and 9CL-PF3ONS were outside of acceptance limits for the ISC associated with all samples in this SDG. The results reported for the impacted analytes in the associated samples have been qualified estimated "UJ" as appropriate on this basis.

The observed recovery for NFDHA was less than 50% for the ISC associated with all samples in this SDG. The non-detected results reported for the impacted analyte in the associated samples have been qualified "UJ" on this basis. It is the data validators recommendation that these results be considered estimated "UJ" when using data as the recovery was less than the lower acceptance limit but greater than 10% rather than applying an "X" qualifier as the validation module instructs.

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 exception. PFOSA was detected in the method blank associated with all samples in this SDG. Upon evaluation, all results reported for the impacted analyte were non-detected and validation action was not required on this basis.

B) Instrument blank contamination:

No problems 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 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 result qualification were found for this criterion with the following exception.

The 13C2-6:2FTS labeled isotope recovery observed during the analysis of sample AF-RHMMW02-WGN01LF-2212W1 was greater than the upper acceptance limit. The positive result reported for the associated target analyte has been qualified estimated low "J-" on this basis.

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 $\geq 3:1$, 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 positive PFNA and PFOS results in sample AF-RHMMW03-WGN01LF-2212W1 were outside the established ratio limit indicating some degree of uncertainty in the qualitative identification of these analytes. The results reported for the impacted analytes in the aforementioned sample have been qualified as estimated, "J" on this basis.

9. COMPOUND QUANTIFICATION

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

Manual integrations were reviewed at the Stage 4 level.

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 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 $\leq 50\%$ for the Relative Percent Difference (RPD) for water samples and $\leq 100\%$ RPD for solid samples, shall be used for original and duplicate sample values greater than or equal to the sample specific LOQ. For field duplicate analyses that do not meet the technical criteria, the action was applied to only the parent sample and its duplicate.

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 with the following exception.

The observed recoveries were less than the lower limit for PFMPA in the LCS, laboratory control duplicate sample (LCSD), and low level LCS associated with all samples in this SDG. The non-detected results reported for the impacted analyte in the associated samples have been qualified "UJ" on this basis. It is the data validators recommendation that these results be considered estimated "UJ" when using data as the recoveries were less than the lower acceptance limit but greater than 10% in the LCS, LCSD, and low level LCS rather than applying an "X" qualifier as the validation module instructs.

The observed recovery for PFBA was greater than the upper acceptance limit for the low level LCS associated with all samples in this SDG. The positive result reported for the impacted analyte has been qualified estimated high "J+" on this basis. Validation action was not required for non-detected results.

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 dilutions, re-extractions, and other re-analyses were provided by the laboratory for 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	
		Major	Minor
Per-fluorinated Compounds			
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	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	PFTTrDA
40	2058-94-8	Perfluoroundecanoic acid	PFUnA

Data Qualification Reason Codes	
Reason Code	Reason Code Description
A	Serial dilution
A1	Ambient Blank
B	The analyte was found in an associated blank as well as in the sample.
B2	CCB
B3	CCB - Neg
B4	Grinding Blank
C	LCS Recovery
C1	Reference Recovery
C2	Reference Recovery RPD
D	MS RPD
D1	Lab Replicate RPD
D2	No precision available
D3	Field Duplicate RPD
D4	Field Triplicate RSD
D5	Laboratory Triplicate RSD
F	Field Blank
F1	Hydrocarbon pattern does not match standard
G1	Initial Calibration RRF
G2	Initial Calibration RSD/r²/r
G3	ICV RRF
H1	Test Hold Time
H2	Prep Hold Time
I	Surrogate recovery outside project limits.
J	CRA/CRI Recovery
K	An analyte (non-common laboratory artifact) was detected in the sample at a concentration less than 5X the concentration detected in the associated method blank.
L	Lab Blank
L1	Lab Blank - Neg
M	MS Recovery
M2	Post Spike
N	Blank - No Action
O	ICS
P	Sample preservation/collection requirement not met.
P1	Column RPD
P2	Improper preparation/extraction
Q	Encore sample holding time exceeded by more than 2X.
Q1	Material Blank

Q2	Encore sample holding time exceeded by less than 2X.
R	Exceeds Linear Calibration Range
S	Internal standard
T	Trip Blank
TI	Tentatively Identified Compound
TR	Trace Level Detect
U	Receipt Temperature
V	Equipment Blank
V1	ICV
V2	CCV
V3	CCV RRF
V4	Sample Receipt Condition
V5	Ending Continuing Calibration Verification
V6	Low Level Calibration Verification
V7	Interference Check Sample A
V8	Interference Check Sample AB
V9	Interference Check Sample A - Negative
W	Column breakdown (pesticides/8270)
X	Raised reporting limit
Y	Cooler temperature greater than 10 degreeec C.
Y1	False Positive
Y2	Data rejected due to radiological anomolies
Y3	Non-accredited analyte/compound. Accreditation not offered at time of analyses for the analyte/compound by the stated method and matrix.
Y4	Performance Check - Degradation of DDT
Y5	Extracted Internal Standard
Y6	Analyte not confirmed on second column.
Y7	Signal to Noise Ratio not met
Z	LCS RPD
Z1	Non-accredited analyte/compound
Z1	Data rejected, more valid data available.
Z2	Detection Level not met uncertainty greater than DL
Z4	MDA Greater than RDL.
Z5	Ion Ratio
Z6	Samples were analyzed past the 12 hour time period from the Tune or opening CCV.

Calculation Documentation

Low standard Calculation
 Sample calculation for results in Column G
 Sample ID AF-RHMW03-WGN01LF-2212W1

Compound	PFBA	
Low standard conc. (ng/ml)		0.4
Sample volume (L) [reported as grams by lab]*		0.49632
Extraction Volume (ml)		2
Dilution		1
AECOM calculated conc. (ng/L)		1.612 %D=
Lab reported conc. (ng/L)		1.6 0.7

*The lab provides the sample weight in grams.
 This assumes that the density of the water sample is 1.0 g/ml.
 It should be noted that the actual density of the sample
 was not in the lab report. (Fresh water is most likely 1.0 g/ml)

AF-RHMW03-WGN01LF-2212W1			
COMPOUND	CONC. of Low Cal Std and ISC Std (ng/ml)	LOQ (ng/L)	Calculated LOQ (ng/L)
PFBA	0.40	1.6	1.612
PFPEA	0.20	0.81	0.806
PFHXA	0.10	0.40	0.403
PFHPA	0.10	0.40	0.403
PFOA	0.10	0.40	0.403
PFNA	0.10	0.40	0.403
PFDA	0.10	0.40	0.403
PFUnA	0.10	0.40	0.403
PFDOA	0.10	0.40	0.403
PFTDA	0.10	0.40	0.403
PFTEDA	0.10	0.40	0.403
PFBS	0.0885	0.40	0.357
PFPEs	0.0940	0.40	0.379
PFHXS	0.0915	0.40	0.369
PFHPS	0.0955	0.40	0.385
PFOS	0.0930	0.40	0.375
PFNS	0.0960	0.40	0.387
PFDS	0.0965	0.40	0.389
PFDOS	0.0970	0.40	0.391
4:2FTS	0.3750	1.6	1.511
6:2FTS	0.3800	1.6	1.531
8:2FTS	0.3834	1.6	1.545
PFOSA	0.10	0.40	0.403
NMeFOSA	0.40	1.6	1.612
NEiFOSA	0.40	1.6	1.612
NMeFOSAA	0.10	0.40	0.403
NEiFOSAA	0.10	0.40	0.403
NMeFOSE	0.40	1.6	1.612
NEiFOSE	0.40	1.6	1.612
HFPO-DA	0.20	0.81	0.806
ADONA	0.1890	0.81	0.762
PFEEsA	0.1780	0.81	0.717
PFMPA	0.20	0.81	0.806
PFMBA	0.20	0.81	0.806
NFDHA	0.20	0.81	0.806
9CL-PF3ONS	0.1870	0.81	0.754
11CL-PF3OUDS	0.1890	0.81	0.762
3:3FTCA	0.40	1.6	1.612
5:3FTCA	0.40	1.6	1.612
7:3FTCA	0.40	1.6	1.612

Final Sample Result Calculation
Red Hill
PFAS
method 1633
APPL

density of water = 1g/ml
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

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)
22L0033-01	PFBA	0.5312	2	496.32	1	2.140554481	2.1
22L0033-02	PFBA	0	2	569.72	1	0	0.70 U

Data Validation Worksheet

DATA VALIDATION PFAS

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

Validator: GAP

Reviewer: DLW

Date Validated: 12/12/22

Reviewed: 12/15/22

Project: Red Hill

SDG: 22L0033

LAB: APPL

Samples Collected: 12/5/2022

2 GW Samples

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 12/6 at 4C

HOLDING TIMES

- ✓ Recommended storage temp is $\leq -20^{\circ}\text{C}$
- ✓ 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

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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

all inside holding time

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-. Non-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

For Red Hill project(see Kristin's email on file in project folder 12/14/22 at 3:25pm)

For EIS %Rs >150% J- positive results, no action on non-detects

For EIS %Rs between lab limit of 20-150%; no action

For EIS %Rs <20% but >10%; J+ positive results, UJ non-detects

For EIS %Rs <10% X positive and non-detected (and recommend R of non-detected, J+ of positive results)

AF-RHMW03-WGN01LF-2212W1

Surrogate: 13C2-4:2FTS 187% associated with 4:2 FTS ND no action required

AF-RHMW02-WGN01LF-2212W1

Surrogate: 13C2-4:2FTS 206% associated with 4:2 FTS ND no action required

Surrogate: 13C2-6:2FTS 175% <200% associated with 6:2 FTS J-

Blank (BBL0133-BLK1) Blank (BBL0133-BLK1)

13C4-PFBA <10% no action taken on this basis

LCS (BBL0133-BS1)

13C4-PFBA <10% no action taken on this basis

LCS Dup (BBL0133-BSD1)

13C4-PFBA <10% no action taken on this basis

MRL Check (BBL0133-MRL1)

13C4-PFBA <10% no action taken on this basis

Non-Extracted Internal STANDARDS

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

Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (LLCS) (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 (40-150) to evaluate
All 40 compounds included.

LCS (BBL0133-BS1) PFMPA ↓ 18.4 nondetects in samples flag X, recommend UJ since %R>10%

LCS Dup (BBL0133-BSD1) PFMPA ↓ 18.5 nondetects in samples flag X, recommend UJ since %R>10%

BBL0133-MRL1 PFBA 833% ↑ flag J+
PFMPA 20.8% ↓ nondetects in samples flag X, recommend UJ since %R>10%

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

Dilution and RA not used for MS evaluation

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

312 **Table III: Sample Qualification in the Presence of Blank Contamination**

	Sample		
Row Number	Result	Validated Result	Validation Qualifier
1	Non-detect or detect \leq LOD	Report at LOD	U
2	> LOQ but \leq 5x blank	Report at Sample Result	J+
3	> LOQ and > 5x blank	Report at Sample Result	None

313 LOD = Limit of Detection

No FB/EBs

MB

BBL0133-BLK1

PFOSA 0.00223 J ug/L ND in samples; no action

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

[Instrument Saphira](#)

12/7/2022	all %RSE <20%
SB03724-ICB1 S2022-12-07A (9) 12/07/22 15:53	all ND
SB03724-SCV1 S2022-12-07A (10) 12/07/22 16:06	all ok
SB03753-CCB1 S2022-12-08B (1) 12/08/22 18:12	all ND
SB03753-LCV1 S2022-12-08B (2) 12/08/22 18:24	NETFOSAA %R<70% but>50% FLAG UJ NFDHA %R<50% FLAG X Recommend UJ since >10% 9CL-PF3ONS %D>130% FLAG UJ
SB03753-CCV1 S2022-12-08B (3) 12/08/22 18:37	all ok
SB03753-CCB2 S2022-12-08B (6) 12/08/22 19:15	all ND
All samples	
SB03753-CCV2 S2022-12-08B (25) 12/08/22 23:17	NFDHA %R<70% but >50% FLAG UJ
SB03753-CCB3 S2022-12-08B (26) 12/08/22 23:30	all ND

COMPOUND IDENTIFICATION

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

Use J flag for module 6

Ion ratio out for:
 AF-RHMW03-WGN01LF-2212W1
 PFNA flag J
 PFUnA ND no action
 PFOS flag J

FIELD DUPLICATES

- ✓ Use QAPP defined criteria
- ✓ If outside acceptance criteria qualify J/UJ

None

SEE FIELD DUPLICATE WORKSHEET

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Sample Summary								E1633DR	
Location	Field Sample ID	Date	Time	Sample Type	Matrix	SBD	SED		
RHMW03	AF-RHMW03-WGN01LF-2212W1	12-05-2022	2005	N	WG	0.00	0.00		X
RHMW02	AF-RHMW02-WGN01LF-2212W1	12-05-2022	1645	N	WG	0.00	0.00		X
Total								2	

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Batch Report

Test Method: E1633DR		Analysis Batch: SB03753								
Location	Matrix	Field Sample ID	Lab Sample ID	Calibration Ref	Run#/Dil'n	Collection Date/Time	Extraction Date/Time	Analysis Date/Time	Prep/Leach Batch	Sample Type
LABQC	WQ	LABQC	BBL0133-BLK1	2250016	1/1	12/7/2022 06:12	12/7/2022 06:12	12/8/2022 19:28	BBL0133/	LB
LABQC	WQ	LABQC	BBL0133-BS1	2250016	1/1	12/7/2022 06:12	12/7/2022 06:12	12/8/2022 19:41	BBL0133/	BS
LABQC	WQ	LABQC	BBL0133-BSD1	2250016	1/1	12/7/2022 06:12	12/7/2022 06:12	12/8/2022 19:53	BBL0133/	BD
RHMW03	WG	AF-RHMW03-WGN01LF-2212W1	22L0033-01	2250016	1/1	12/5/2022 20:05	12/7/2022 06:12	12/8/2022 20:44	BBL0133/	N
RHMW02	WG	AF-RHMW02-WGN01LF-2212W1	22L0033-02	2250016	1/1	12/5/2022 16:45	12/7/2022 06:12	12/8/2022 21:10	BBL0133/	N

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Field Batch Report

--No Records Found--

MS Mismatch Report

--No Records Found--

Section to identify Matrix Spike mismatches where parent sample differs from MS by dilution.

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QC Outlier Report

Test Method: E1633DR		Extraction Method: METHOD		Leach Method: NONE							
QC Element	Sample ID/ Lab Sample ID	Run#/ Dil'n	Analyte	Result (Units)	Qualifier	Warning Limits	Control Limits	Reason	Comment	Rule	Action Level
Extracted Internal Standard	AF-RHMW02-WGN01LF-2212W1 (N) / 22L0033-02	1 / 1.00	13C2-4:2 Fluorotelomer sulfonate (13C2-4:2 FTS)	206.0 (percent)	J/None	20 - 150	10 - 150	Y5			
Extracted Internal Standard	AF-RHMW02-WGN01LF-2212W1 (N) / 22L0033-02	1 / 1.00	13C2-6:2 Fluorotelomer sulfonate (13C2-6:2 FTS)	176.0 (percent)	J/None	20 - 150	10 - 150	Y5			
Extracted Internal Standard	AF-RHMW03-WGN01LF-2212W1 (N) / 22L0033-01	1 / 1.00	13C2-4:2 Fluorotelomer sulfonate (13C2-4:2 FTS)	187.0 (percent)	J/None	20 - 150	10 - 150	Y5			
Lab Blank	BBL0133-BLK1 (LB) / BBL0133-BLK1	1 / 1.00	Perfluorooctanesulfonamide (PFOSA)	0.0002230 (ug/l)	U/None*	< 0.0001	< 0.0004	L		5	0.0011 1
LCS Recovery	BBL0133-BS1 (BS) / BBL0133-BS1	1 / 1.00	Perfluoro-3-methoxypropanoic acid (PFMPA)	18.00 (percent)	J/X	40 - 150	40 - 150	C			
LCS Recovery	BBL0133-BSD1 (BD) / BBL0133-BSD1	1 / 1.00	Perfluoro-3-methoxypropanoic acid (PFMPA)	19.00 (percent)	J/X	40 - 150	40 - 150	C			

*Blank flags displayed in the above table identify qualification of the sample result when it is less than or equal to the LOQ/RL. Sample results above the LOQ will be qualified based on the validation type such as J+ at the sample result.

Rule is the multiplier used when blank contamination occurs to determine action level.

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Qualified Results

Test Method: E1633DR		Extraction Method: METHOD		Leach Method: NONE							
FieldSample ID	LabSample ID	Matrix	Type	Analyte	LOQ	Lab Result	Qualified Result	Bias	Units	Reason	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	6:2 Fluorotelomer sulfonic acid (6:2 FTS)	0.00140	0.00210 J	0.00210 J	-	ug/l	Y5	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	0.000700	0.000350 U	0.000350 UJ		ug/l	V6	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	N-Ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	0.000350	0.000180 U	0.000180 UJ		ug/l	V6	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	0.000700	0.000350 U	0.000350 UJ		ug/l	V2/V6	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	Perfluoro-3-methoxypropanoic acid (PFMPA)	0.000700	0.000350 UR	0.000350 UJ		ug/l	C	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	Perfluorohexanoic acid (PFHxA)	0.000350	0.000160 FJ	0.000160 J		ug/l	TR	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	Perfluorooctanesulfonic acid (PFOS)	0.000350	0.000130 FJ	0.000130 J		ug/l	TR	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	0.000810	0.000400 U	0.000400 UJ		ug/l	V6	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	N-Ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	0.000400	0.000200 U	0.000200 UJ		ug/l	V6	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	0.000810	0.000400 U	0.000400 UJ		ug/l	V2/V6	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	Perfluoro-3-methoxypropanoic acid (PFMPA)	0.000810	0.000400 UR	0.000400 UJ		ug/l	C	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	Perfluorobutanoic acid (PFBA)	0.00160	0.00210	0.00210 J	+	ug/l	C	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	Perfluorononanoic acid (PFNA)	0.000400	0.000170 FI J	0.000170 J		ug/l	TR/Z5	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	Perfluorooctanesulfonic acid (PFOS)	0.000400	0.0000690 FI J	0.0000690 J		ug/l	TR/Z5	

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Qualified Results

Test Method: E1633DR			Extraction Method: METHOD			Leach Method: NONE				
FieldSample ID	LabSample ID	Matrix	Type	Analyte	LOQ	Lab Result	Qualified Result	Bias	Units	Reason
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	Perfluorooctanoic acid (PFOA)	0.00040 0	0.000250 FJ	0.000250 J		ug/l	TR

Qualified analytes in samples are reported as estimated, not detected (UJ) at the Limit of Detection (LOD).

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Detected Results

Test Method: E1633DR		Extraction Method: METHOD			Leach Method: NONE						
FieldSample ID	LabSample ID	Matrix	Type	Dilution	Analyte	LOQ	Lab Result	Qualified Result	Units	Reason	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	1	6:2 Fluorotelomer sulfonic acid (6:2 FTS)	0.00140	0.00210 J	0.00210 J	ug/l	Y5	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	1	Perfluorohexanoic acid (PFHxA)	0.00035 0	0.000160 FJ	0.000160 J	ug/l	TR	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	1	Perfluorooctanesulfonic acid (PFOS)	0.00035 0	0.000130 FJ	0.000130 J	ug/l	TR	
AF-RHMW02-WGN01LF-2212W1	22L0033-02	W	N	1	Perfluoropentanoic acid (PFPeA)	0.00070 0	0.000920	0.000920	ug/l		
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	1	6:2 Fluorotelomer sulfonic acid (6:2 FTS)	0.00160	0.00800	0.00800	ug/l		
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	1	Perfluorobutanoic acid (PFBA)	0.00160	0.00210	0.00210 J	ug/l	C	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	1	Perfluoroheptanoic acid (PFHpA)	0.00040 0	0.000790	0.000790	ug/l		
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	1	Perfluorohexanoic acid (PFHxA)	0.00040 0	0.00120	0.00120	ug/l		
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	1	Perfluorononanoic acid (PFNA)	0.00040 0	0.000170 FI J	0.000170 J	ug/l	TR/Z5	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	1	Perfluorooctanesulfonic acid (PFOS)	0.00040 0	0.0000690 FI J	0.0000690 J	ug/l	TR/Z5	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	1	Perfluorooctanoic acid (PFOA)	0.00040 0	0.000250 FJ	0.000250 J	ug/l	TR	
AF-RHMW03-WGN01LF-2212W1	22L0033-01	W	N	1	Perfluoropentanoic acid (PFPeA)	0.00081 0	0.00310	0.00310	ug/l		

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Rejected Results

--No Records Found--

Anomalies Count

--No Records Found--

Reporting Anomalies

--No Records Found--

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Review Questions
