



DATA VALIDATION REPORT

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

**SDG: FC6649
SGS North America Inc - Orlando**

Prepared by
ENVIRONMENTAL DATA SERVICES, LTD.

Prepared for
AECOM Environmental

Released: 06/26/23

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 "Dina Manov".

Dina Manov

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

Larry Lewis

A handwritten signature in black ink, appearing to read "Paloma Hoelzle".

Paloma Hoelzle

EXECUTIVE NARRATIVE

Sample Delivery Group: FC6649

Laboratory: SGS North America Inc - Orlando

Site: Red Hill Bulk Storage Facility, CV 23F0104

Sampling dates: 06/02/2023

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'Ahū, 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-RHMW17D-WGN01LF-2305W5	FC6649-1	groundwater	S2BVEM
AF-RHMW17D-WQFB01-2305W5	FC6649-2	field blank	S2BVEM

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 $\pm 30\%$. Note: the laboratory reports refer to the ISC as Low-Concentration Calibration Verification (LCCV). The validator has determined that the low level CCV 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 MeFOSAA, PFDA, and PFEESA were outside of acceptance limits for the ISC associated with all samples in this sample delivery group (SDG). The results reported for the impacted analytes in the associated samples have been qualified estimated "UJ" as appropriate on this basis.

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 were found for this criterion.

B) Field/Equipment blank contamination:

Sample AF-RHMMW17D-WQFB01-2305W5 was submitted as a field blank in association with the samples in this SDG. No problems were found for this criterion.

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 with the following exception.

The observed isotope dilution standard recovery for ¹³C₄-PFBA was extremely low (less than 10%) during the analysis of sample AF-RHMW17D-WGN01LF-2305W5. The non-detected result reported for the impacted analyte has been qualified rejected "R" on this basis. It is the data validator's recommendation that this result be considered rejected "R" when using data as the recovery was extremely low rather than applying an "X" qualifier as the validation module instructs.

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.

9. COMPOUND QUANTIFICATION

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

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

10. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY / MATRIX DUPLICATE

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 AF-RHMW17D-WGN01LF-2305W5 was submitted for MS evaluation in association with this SDG. Upon evaluation all accuracy indicators were acceptable with the following exceptions. The observed MS recoveries for PFMPA and 3:3 FTCA were less than the lowest acceptance limit. The non-detected results reported for the impacted analytes in the parent sample have been qualified "UJ" on this basis.

11. FIELD DUPLICATES/TRIPPLICATES

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 $\leq 30\%$ for the Relative Percent Difference (RPD) for water samples and $\leq 50\%$ RPD for solid samples, shall be used when original and duplicate sample values are 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. A control limit of $\leq 35\%$ RSD was applied for field triplicate samples when original and triplicate sample values are greater than the sample specific LOQ. For field triplicate analyses that do not meet the technical criteria, the action was applied to only the parent sample, duplicate and triplicate.

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.

No problems were found for this criterion with the following exception. The observed recovery was less than the lower limit for 3:3 FTCA in the 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 validator's 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% in the LCS; rather than applying an "X" qualifier as the validation module instructs.

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 sample dilutions, re-extractions and/or reanalyses 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	x		
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/Matrix Duplicate			x
Laboratory Control Sample			x
Other Quality Control Data out of Specification	x		
Field Duplicate / Triplicate	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.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

**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.

Data Validation Worksheet

DATA VALIDATION PFAS

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

Validator: [GAP](#)

Reviewer: [DLW](#)

Date Validated: [6/23/23](#)

Reviewed: [6/26/23](#)

Project: Red Hill

SDG: FC6649

LAB: SGS North America Inc. - Orlando

Samples Collected: 6/2/2023

[1 GW, 1FB](#)

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 6/6 at 4.2C](#)

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

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 6/2/23

Extracted 6/9

Analyzed 6/12

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-. 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-RHMW17D-WGN01LF-2305W5 13C4-PFBA 4% (confirmed by MS on same sample) X per module;
recommend R since <10%

Non-Extracted Internal STANDARDS

- ✓ Used to quantify EIS
- ✓ If low are 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)

- ✓ 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.](#)

OP97275-LLBS all ok

OP97275-BS 3:3 FTCA 38%↓ associated with all samples

X per module recommend UJ as recovery is ↓ but >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:

MS none FC6649-1 AF-RHMW17D-WGN01LF-2305W5

PFMPA 23% UJ

3:3 FTCA 20% UJ

Matrix duplicate: none

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 ≤ LOD	Report at LOD	U
2	> LOQ but ≤ 5x blank	Report at Sample Result	J+
3	> LOQ and > 5x blank	Report at Sample Result	None

313 LOD = Limit of Detection

All instrument blanks ND

AF-RHMW17D-WQFB01-2305W5 ND

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

All 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

- ✓ 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

1.0LL CCV is the method required ISC

Instrument GCMS6Q

6/12/2023

all %RSD <20%

S6Q287-IBLK 6Q19248.D 06/12/23 17:40 n/a Instrument Blank
 S6Q287-ICV287 6Q19249.D 06/12/23 17:54 n/a Initial cal verification 4
 S6Q287-ICV287 6Q19250.D 06/12/23 18:08 n/a Initial cal verification 20

S6Q287-CC287 6Q19251.D 06/12/23 18:22 n/a Continuing cal 4
 S6Q287-CC287 6Q19252.D 06/12/23 18:36 n/a Continuing cal 1.0LL

MeFOSAA >30% UJ

PFDA >30% UJ

PFEESA >30% UJ

S6Q287-CC287 6Q19261.D 06/12/23 20:42 05:36 Continuing cal 4

S6Q287-ICCB 6Q19262.D 06/12/23 20:56 05:50 Continuing Calibration Blank

All samples

S6Q287-CC287 6Q19272.D 06/12/23 23:15 08:09 Continuing cal 4
 S6Q287-ICCB 6Q19273.D 06/12/23 23:29 08:23 Continuing Calibration Blank

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

Reason Code: Z5

All ok

FIELD DUPLICATES

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

For field triplicates use 35% RSD per Kristin's email on file from 12/14/22

None

FIELD DUPLICATE WORKSHEET

Data Validation Report for FC6649

Facility: RH Fire Suppression System
Event: AFFF Assessment Sampling GW 2023 May
SDG: FC6649
Guidance Document: RHS PFAS UFP-QAPP
Prime Contractor: AECOM, Honolulu, HI
Project Manager:
Contract Laboratory(ies): SGS North America, Inc., Orlando, FL
Data Review Contractor:
Data Review Level:
Primary Data Reviewer: ,
Date Submitted:

Field Sample ID	Lab Sample ID	Matrix	Type/Type Code	E1633DR
AF-RHMW17D-WGN01LF-2305W5	FC6649-1	Water	Field Sample/N	X
AF-RHMW17D-WQFB01-2305W5	FC6649-2	Water	Ambient Blank/AB	X

Data Validation Report for FC6649

This report assesses the analytical data quality associated with the analyses listed on the preceding cover page at data validation level. This assessment has been made through a combination of automated data review (ADR) and supplemental manual review, the details of which are described below. The approach taken in the review of this data set is consistent with the requirements contained in the RHS PFAS UFP-QAPP and the additional guidance documents incorporated by reference to the extent possible. Where definitive guidance is not provided, results have been evaluated in a conservative manner using professional judgment.

Sample collection was managed and directed by AECOM, Honolulu, HI; analyses were performed by SGS North America, Inc., Orlando, FL and were reported under sample delivery group (SDG) FC6649. Data have been evaluated electronically based on electronic data deliverables (EDDs) provided by the laboratory, and hard copy data summary forms have also been reviewed during this effort and compared to the automated review output by the reviewers whose signatures appear on the following page. Findings based on the automated data submission and manual data verification processes are detailed in the ADR narrative and throughout this report.

All quality control (QC) elements associated with this SDG have been reviewed by a project chemist in accordance with the requirements defined for the project. This review is documented in the attached Data Review Checklists. The QC elements listed below were supported by the electronic deliverable and were evaluated using ADR processes.

- Ambient Blank
- Extracted Internal Standard
- Lab Blank
- LCS Recovery
- MS Recovery
- Prep Hold Time
- Test Hold Time

Results of the ADR process were subsequently reviewed and updated as applicable by the data review chemists identified on the signature page. Quality control elements that were not included in the electronic deliverable were reviewed manually and findings are documented within this report. Summaries of findings and associated qualified results are documented throughout this report.

A total of 10 results (12.50%) out of the 80 results (sample and field QC samples) reported are qualified based on review and 1 results (1.25%) have been rejected or deemed a serious deficiency (X qualifier). Trace values, defined as results that are qualified as estimated because they fall between the detection limit and the reporting limit/limit of quantitation, are not counted as qualified results in the above count. The qualified results are detailed throughout this report and discussed in the narrative below, where appropriate.

Data Validation Report for FC6649

Narrative Comments

Analytical Method	Data Reviewer Comment
-------------------	-----------------------

Reviewed by , ,

As the Reviewer, I certify that I have performed a data review process in accordance with the requirements of the project guidance document, and have compared the electronic data to the laboratory's hard copy report and have verified the consistency of the reported sample results and method quality control data between the two deliverables.

Data Validation Report for FC6649

Quality Control Outliers for test method E1633DR, Extracted Internal Standard

Method performance for individual samples is demonstrated through spiking activities. All samples are spiked with internal standards compounds prior to sample preparation (EIS). The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Summary forms were evaluated and compared to electronic data deliverables. EIS results that were outside of the acceptance criteria are listed below.

Sample ID/ Lab Sample ID	Analyte	Result	Warning Limits	Control Limits	Units	Qualifier	Reason Code	Comment
AF-RHMW17D-WGN01LF-2305W5 (N)	Perfluoro-n-[13C4]butanoic acid (13C4-PFBA)	4.00	20 - 150	10 - 150	percent	X/X	Y5	
AF-RHMW17D-WGN01LF-2305W5 (MS)	Perfluoro-n-[13C4]butanoic acid (13C4-PFBA)	4.00	20 - 150	10 - 150	percent	X/X	Y5	No Qualifiers Applied

Where two qualifiers are listed, such as 'J/UJ', the first applies to positive results, and the second to non-detect results. Upper and Lower Warning and Control Limits are abbreviated UWL, LWL, UCL, and LCL in the Comment field.

Qualified Results associated with the Extracted Internal Standard for E1633DR

FieldSample ID	Type	Analyte	LOQ	Lab Result	Qualified Result	Bias	Units	Reason
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluorobutanoic acid (PFBA)	15.0	3.70 U	3.70 R		ng/l	Y5

Analytes not found in project samples are reported as not detected at the limit of detection (LOD) unless blank contamination occurs and then the sample may be reported as not detected at the (LOD) or (LOQ) based on the sample concentration and the validation guidance. In instances where no LOD is provided, results are reported down to the LOQ.

Data Validation Report for FC6649

Quality Control Outliers for test method E1633DR, LCS Recovery

The laboratory control sample/laboratory control sample duplicate (LCS/LCSD) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Reported results were evaluated to determine compliance with the required acceptance criteria, and summary forms were evaluated and compared to electronic data deliverables. Findings of this review, and any associated qualified results, are listed below.

Sample ID/ Lab Sample ID	Analyte	Result	Warning Limits	Control Limits	Units	Qualifier	Reason Code	Comment
OP97275-BS (BS)	3-Perfluoropropyl propanoic acid (3:3FTCA)	38.0	40 - 150	40 - 150	percent	J/X	C	

Where two qualifiers are listed, such as 'J/UJ', the first applies to positive results, and the second to non-detect results. Upper and Lower Warning and Control Limits are abbreviated UWL, LWL, UCL, and LCL in the Comment field.

Qualified Results associated with the LCS Recovery for E1633DR

FieldSample ID	Type	Analyte	LOQ	Lab Result	Qualified Result	Bias	Units	Reason
AF-RHMW17D- WGN01LF-2305W5 FC6649-1	N	3-Perfluoropropyl propanoic acid (3:3FTCA)	19.0	9.30 U	9.30 UJ		ng/l	C/M
AF-RHMW17D-WQFB01- 2305W5 FC6649-2	AB	3-Perfluoropropyl propanoic acid (3:3FTCA)	18.0	8.90 U	8.90 UJ		ng/l	C

Analytes not found in project samples are reported as not detected at the limit of detection (LOD) unless blank contamination occurs and then the sample may be reported as not detected at the (LOD) or (LOQ) based on the sample concentration and the validation guidance. In instances where no LOD is provided, results are reported down to the LOQ.

Data Validation Report for FC6649

Quality Control Outliers for test method E1633DR, MS Recovery

Data for matrix spikes/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. These data alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgment, MS/MSD data can be used in conjunction with other available QC information. Reported results were evaluated to determine compliance with the required acceptance criteria, and summary forms were evaluated and compared to electronic data deliverables. Findings of this review, and any associated qualified results, are listed below.

Sample ID/ Lab Sample ID	Analyte	Result	Warning Limits	Control Limits	Units	Qualifier	Reason Code	Comment
AF-RHMW17D-WGN01LF-2305W5 (MS)	3-Perfluoropropyl propanoic acid (3:3FTCA)	20.0	40 - 150	10 - 150	percent	J/UJ	M	
AF-RHMW17D-WGN01LF-2305W5 (MS)	Perfluoro-3-methoxypropanoic acid (PFMPA)	23.0	40 - 150	10 - 150	percent	J/UJ	M	

Where two qualifiers are listed, such as 'J/UJ', the first applies to positive results, and the second to non-detect results. Upper and Lower Warning and Control Limits are abbreviated UWL, LWL, UCL, and LCL in the Comment field.

Qualified Results associated with the MS Recovery for E1633DR

FieldSample ID	Type	Analyte	LOQ	Lab Result	Qualified Result	Bias	Units	Reason
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	3-Perfluoropropyl propanoic acid (3:3FTCA)	19.0	9.30 U	9.30 UJ		ng/l	C/M
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluoro-3-methoxypropanoic acid (PFMPA)	7.40	1.90 U	1.90 UJ		ng/l	M

Analytes not found in project samples are reported as not detected at the limit of detection (LOD) unless blank contamination occurs and then the sample may be reported as not detected at the (LOD) or (LOQ) based on the sample concentration and the validation guidance. In instances where no LOD is provided, results are reported down to the LOQ.

Data Validation Report for FC6649

Table of All Qualified Results

Test Method: E1633DR		Extraction Method: METHOD						
FieldSample ID / LabSample ID	Type	Analyte	LOQ	Lab Result	Qualified Result	Bias	Units	Reason
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	3-Perfluoropropyl propanoic acid (3:3FTCA)	19.0	9.30 U	9.30 UJ		ng/l	C/M
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	N-Methyl perfluorooctanesulfonamid oacetic acid (NMeFOSAA)	4.60	3.70 U	3.70 UJ		ng/l	V6
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	7.40	1.90 U	1.90 UJ		ng/l	V6
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluoro-3-methoxypropanoic acid (PFMPA)	7.40	1.90 U	1.90 UJ		ng/l	M
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluorobutanoic acid (PFBA)	15.0	3.70 U	3.70 R		ng/l	Y5
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluorodecanoic acid (PFDA)	3.70	1.90 U	1.90 UJ		ng/l	V6
AF-RHMW17D-WQFB01-2305W5 FC6649-2	AB	3-Perfluoropropyl propanoic acid (3:3FTCA)	18.0	8.90 U	8.90 UJ		ng/l	C
AF-RHMW17D-WQFB01-2305W5 FC6649-2	AB	N-Methyl perfluorooctanesulfonamid oacetic acid (NMeFOSAA)	4.50	3.60 U	3.60 UJ		ng/l	V6
AF-RHMW17D-WQFB01-2305W5 FC6649-2	AB	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	7.10	1.80 U	1.80 UJ		ng/l	V6
AF-RHMW17D-WQFB01-2305W5 FC6649-2	AB	Perfluorodecanoic acid (PFDA)	3.60	1.80 U	1.80 UJ		ng/l	V6

Analytes not found in project samples are reported as not detected at the limit of detection (LOD) unless blank contamination occurs and then the sample may be reported as not detected at the (LOQ) based on the sample concentration.

In instances where no LOD is provided, results are reported down to the LOQ.

Trace values are not included in the qualified results table unless additional reason codes are associated.

Data Validation Report for FC6649

Table of Results with Modified Qualifiers

Modified Qualifiers for test method E1633DR

FieldSample ID / LabSample ID	Type	Analyte	LOQ	Lab Result	ADR Result	Modified Result	Reason
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	3-Perfluoropropyl propanoic acid (3:3FTCA)	19.0	9.30 U	9.30 X	9.30 UJ	C/M
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	N-Methyl perfluorooctanesulfonamid oacetic acid (NMeFOSAA)	4.60	3.70 U	3.70 U	3.70 UJ	V6
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluoro(2-ethoxyethane)sulfonic acid (PFEEESA)	7.40	1.90 U	1.90 U	1.90 UJ	V6
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluorobutanoic acid (PFBA)	15.0	3.70 U	3.70 X	3.70 R	Y5
AF-RHMW17D-WGN01LF-2305W5 FC6649-1	N	Perfluorodecanoic acid (PFDA)	3.70	1.90 U	1.90 U	1.90 UJ	V6
AF-RHMW17D-WQFB01-2305W5 FC6649-2	AB	3-Perfluoropropyl propanoic acid (3:3FTCA)	18.0	8.90 U	8.90 X	8.90 UJ	C
AF-RHMW17D-WQFB01-2305W5 FC6649-2	AB	N-Methyl perfluorooctanesulfonamid oacetic acid (NMeFOSAA)	4.50	3.60 U	3.60 U	3.60 UJ	V6
AF-RHMW17D-WQFB01-2305W5 FC6649-2	AB	Perfluoro(2-ethoxyethane)sulfonic acid (PFEEESA)	7.10	1.80 U	1.80 U	1.80 UJ	V6
AF-RHMW17D-WQFB01-2305W5 FC6649-2	AB	Perfluorodecanoic acid (PFDA)	3.60	1.80 U	1.80 U	1.80 UJ	V6

Analytes not found in project samples are reported as not detected at the limit of detection (LOD) unless blank contamination occurs and then the sample may be reported as not detected at the (LOQ) based on the sample concentration.
 In instances where no LOD is provided, results are reported down to the LOQ.
 Trace values are not included in the qualified results table unless additional reason codes are associated.

Reason Code Definitions

Code	Definition
C	LCS Recovery
M	MS Recovery
TR	Trace Level Detect
V6	Low Level Calibration Verification
Y5	Extracted Internal Standard

Flag Code and Definitions

Flag	Definition
J	Estimated Value
N	The analysis indicates the presence of an analyte for which there was presumptive evidence to make a tentative identification.
NJ	The analyte has been tentatively identified or presumptively as present and the associated numerical value was the estimated concentration in the sample.
R	The data are rejected due to deficiencies in meeting QC criteria and may not be used for decision making.
U	Undetected: The analyte was analyzed for, but not detected.

Data Validation Report for FC6649

UJ	The analyte was not detected; however, the result is estimated due to discrepancies in meeting certain analyte-specific quality control criteria.
X	Result may require rejection; PDT attention required

Bias

-	The result may be biased low
+	The result may be biased high

Note - The bias field is a separate field; however, it is an integral part of the final flag (qualifier) on the sample result

Data Validation Report for FC6649

Review Questions
