



**DATA VALIDATION REPORT**

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

**SDG: 23C0146  
APPL, INC.**

Prepared by  
**ENVIRONMENTAL DATA SERVICES, LTD.**

Prepared for  
**AECOM Environmental**

**Released: 4/04/23**

**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:** 23C0146

**Laboratory:** APPL, Inc.

**Site:** Red Hill Bulk Storage Facility, CV 23F0104

**Sampling dates:** 3/15/2023

**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’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
RHSF-PUMP-PR-01-031523-N	23C0146-01	aqueous	S4VEM
RHS-EF-TRAIN-01-031523-N	23C0146-02	aqueous	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 exception.*

*The observed recovery for PFPeS was outside of acceptance limits for one CCV associated with all samples in this sample delivery group (SDG). The results reported for the impacted analyte in the associated samples has been qualified estimated "J+" or "UJ" as appropriate 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  $\pm 30\%$ . 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 PFEESA, 9CL-PF3ONS, and 5:3FTCA 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 "UJ" on this basis.*

*The observed recovery for 11CL-PF3OUDS was greater than 150% 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 greater than 150% but the reported results were non-detected 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 exceptions. PFOS and PFOSA were positively detected in the method blank associated with all samples in this SDG. Positive results reported for PFOS and PFOSA in the associated samples have been evaluated and qualified per validation guidance on this basis.*

### **B) Instrument blank contamination:**

*No problems were found for this criterion.*

### **B) Field/Equipment blank contamination:**

*No samples were submitted as a field blank in association with this SDG.*

## 6. EXTRACTED INTERNAL STANDARDS

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

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

*The observed isotope dilution standard recovery for 13C2-8:2FTS was greater than the upper acceptance limit during the analyses of samples RHS-EF-TRAIN-01-031523-N. The result reported for associated target analytes have been qualified "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  $\pm 0.4$  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 ratio for PFBS in sample RHS-EF-TRAIN-01-031523-N 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 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. No anomalies were identified.*

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

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

*No samples were submitted for MS/MSD and/or matrix duplicate evaluation in association with this SDG.*

**11. FIELD DUPLICATES/ TRIPLICATES**

**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. 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 exceptions.*

*The observed recovery for PFHXA was greater than the upper acceptance limit for the low level LCS associated with all samples in this SDG. The positive results reported for the impacted analytes in the associated sample have 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.**

*Samples were re-extracted and/or reanalyzed in several cases to confirm quality control results. Upon review, the laboratory reported the best and final result.*

#### **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 / 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**

<b>Data Qualifier</b>	<b>Definition</b>
<b>U</b>	The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.
<b>J</b>	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
<b>J+</b>	The result is an estimated quantity, but the result may be biased high.
<b>J-</b>	The result is an estimated quantity, but the result may be biased low.
<b>UJ</b>	The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
<b>X</b>	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.
<b>R</b>	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**

<b>NO</b>	<b>CAS #</b>	<b>Target Name</b>	<b>Target Abbreviation</b>
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

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

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





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)
23C0146-01	PFBA	0.1892	2	581.1	1	0.651178799	0.65



**Low standard Calculation**

Sample calculation for results in Column G

Sample ID	RHSF-PUMP-PR-01-031523-N
Compound	PFBA
Low standard conc. (ng/ml)	0.4
Sample volume (L) [reported as grams by lab]*	0.5811
Extraction Volume (ml)	2
Dilution	1
AECOM calculated conc. (ng/L)	1.377 %D=
Lab reported conc. (ng/L)	1.4 -1.7

\*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-WGN01F-2212W1			
COMPOUND	CONC. of Low Cal Std and ISC Std (ng/ml)	LOQ (ng/L)	Calculated LOQ (ng/L)
PFBA	0.40	1.4	1.377
PFPEA	0.20	0.69	0.688
PFHXA	0.10	0.34	0.344
PFHPA	0.10	0.34	0.344
PFOA	0.10	0.34	0.344
PFNA	0.10	0.34	0.344
PFDA	0.10	0.34	0.344
PFUnA	0.10	0.34	0.344
PFDOA	0.10	0.34	0.344
PFTDA	0.10	0.34	0.344
PFTEDA	0.10	0.34	0.344
PFBS	0.0885	0.34	0.305
PFPEs	0.0938	0.34	0.323
PFHXS	0.0911	0.34	0.314
PFHPS	0.0951	0.34	0.327
PFOS	0.0927	0.34	0.319
PFNS	0.0960	0.34	0.330
PFDS	0.0963	0.34	0.331
PFDOS	0.0970	0.34	0.334
4:2FTS	0.3738	1.4	1.287
6:2FTS	0.3874	1.4	1.333
8:2FTS	0.3833	1.4	1.319
PFOSA	0.10	0.34	0.344
NMeFOSA	0.40	1.4	1.377
NEiFOSA	0.40	1.4	1.377
NMeFOSAA	0.10	0.34	0.344
NEiFOSAA	0.10	0.34	0.344
NMeFOSE	0.40	1.4	1.377
NEiFOSE	0.40	1.4	1.377
HFPO-DA	0.20	0.69	0.688
ADONA	0.1885	0.69	0.649
PFEEsA	0.1785	0.69	0.614
PFMPA	0.20	0.69	0.688
PFMBA	0.20	0.69	0.688
NFDHA	0.20	0.69	0.688
9CL-PF3ONS	0.1867	0.69	0.643
11CL-PF3OUDS	0.1886	0.69	0.649
3:3FTCA	0.40	1.4	1.377
5:3FTCA	0.40	1.4	1.377
7:3FTCA	0.40	1.4	1.377

## **Data Validation Worksheet**

# DATA VALIDATION PFAS

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

Validator: DM

Reviewer: GAP

Date Validated: 3/31/2023

Reviewed: 4/3/2023

Project: Red Hill

SDG: 23C0146

LAB: APPL

Samples Collected: 3/15/2023

2 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 3/16

### 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 3/15/23

Extracted 3/17

Analyzed 3/21

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)

RHSF-PUMP-PR-01-031523-N (23C0146-01 )

13C2-4:2FTS 1296↑ ND no Q

13C2-8:2FTS 209↑ ND no Q

RHS-EF-TRAIN-01-031523-N (23C0146-02 )

13C2-4:2FTS 217↑ ND no Q

13C2-8:2FTS 179↑ J-

#### Non-Extracted Internal STANDARDS

- ✓ Used to quantify EIS
- ✓ If low area 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 LLCS %R is > upper limit; qualify detects J+; no action on non-detected
- ✓ If LCS or LLCS %R is < lower limit; qualify detected J- and non-detected X

Use 40-150 to evaluate  
All 40 compounds included.

BCC0177-BS1 all ok

**MRL Check (BCC0177-MRL1) all ok except**

PFHXA ↑ **positive J+; no action ND**

### **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 40-150% to evaluate

Sample: **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 $\leq$ 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

FB/EBs  
None

Blank (BCC0177-BLK1)

PFOS .0941J sample 2 flag U <LOD  
PFOSA .295J no Q

ICBs/CCBs see below

### MASS CALIBRATION

- ✓ Verified to be  $\pm 0.2$  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 Saphira

3/7/2023

all %RSE <20%

Initial Cal Blank SC00916-ICB1 S2023-03-07A (9) 03/07/23 17:09 all ND  
Secondary Cal Check SC00916-SCV1 S2023-03-07A (10) 03/07/23 17:22 ok  
Calibration Blank SC01124-CCB1 S2023-03-21A (1) 03/21/23 13:57  
Low Cal Check SC01124-LCV1 S2023-03-21A (2) 03/21/23 14:10  
PFEEESA 31.2↑ FLAG UJ  
9CL-PF3ONS 30.5↑ FLAG UJ  
11CL-PF3OUDS 51.8% (>150%R) UJ (based on professional judgment instead of X)  
5:3FTCA -30.3↓ FLAG UJ  
Calibration Check SC01124-CCV2 S2023-03-21A (11) 03/21/23 16:06  
PFPEES %R 136↑ FLAG J+  
Calibration Blank SC01124-CCB3 S2023-03-21A (12) 03/21/23 16:19  
Samples 1, 2  
Calibration Check SC01124-CCV3 S2023-03-21A (36) 03/21/23 21:28  
Calibration Blank SC01124-CCB4 S2023-03-21A (37) 03/21/23 21:41

## COMPOUND IDENTIFICATION

- ✓ RT within  $\pm 0.4$  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

RHS-EF-TRAIN-01-031523-N

PFBS FLAG J

#### 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*

*All ok*

SEE FIELD DUPLICATE WORKSHEET

**Data Validation Report for 23C0146**

Facility: RH Fire Suppression System  
 Event: AFFF Assessment Sampling GAC 2023 March  
 SDG: 23C0146  
 Guidance Document: RHS PFAS UFP-QAPP  
 Prime Contractor: AECOM, Honolulu, HI  
 Project Manager:  
 Contract Laboratory(ies): Agriculture & Priority Pollutants Laboratories, Inc.  
 Data Review Contractor:  
 Data Review Level:  
 Primary Data Reviewer: ,  
 Date Submitted:

Field Sample ID	Lab Sample ID	Matrix	Type/Type Code	E1633DR
RHS-EF-TRAIN-01-031523-N	23C0146-02	Water	Field Sample/N	X
RHSF-PUMP-PR-01-031523-N	23C0146-01	Water	Field Sample/N	X

## Data Validation Report for 23C0146

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 Agriculture & Priority Pollutants Laboratories, Inc. and were reported under sample delivery group (SDG) 23C0146. 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.

- Extracted Internal Standard
- Lab Blank
- LCS 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 14 results (17.50%) out of the 80 results (sample and field QC samples) reported are qualified based on review and 0 results (0.00%) 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 23C0146

Narrative Comments

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Analytical Method	Data Reviewer Comment
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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 23C0146

### 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
LABQC (BS)	13C2-4:2 Fluorotelomer sulfonate (13C2- 4:2 FTS)	217	20 - 150	10 - 150	percent	J/None	Y5	No Qualifiers Applied
LABQC (LB)	13C2-4:2 Fluorotelomer sulfonate (13C2- 4:2 FTS)	231	20 - 150	10 - 150	percent	J/None	Y5	No Qualifiers Applied
LABQC (BS)	Perfluoro-n- [1,2,3,4- 13C4]heptanoic acid (13C4- PFHpA)	160	20 - 150	10 - 150	percent	J/None	Y5	No Qualifiers Applied
LABQC (LB)	Perfluoro-n- [1,2,3,4- 13C4]heptanoic acid (13C4- PFHpA)	169	20 - 150	10 - 150	percent	J/None	Y5	No Qualifiers Applied
RHS-EF-TRAIN-01-031523-N (N)	13C2-4:2 Fluorotelomer sulfonate (13C2- 4:2 FTS)	216	20 - 150	10 - 150	percent	J/None	Y5	
RHS-EF-TRAIN-01-031523-N (N)	13C2-8:2 Fluorotelomer sulfonate (13C2- 8:2 FTS)	179	20 - 150	10 - 150	percent	J/None	Y5	
RHSF-PUMP-PR-01-031523-N (N)	13C2-4:2 Fluorotelomer sulfonate (13C2- 4:2 FTS)	195	20 - 150	10 - 150	percent	J/None	Y5	
RHSF-PUMP-PR-01-031523-N (N)	13C2-8:2 Fluorotelomer sulfonate (13C2- 8:2 FTS)	208	20 - 150	10 - 150	percent	J/None	Y5	

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
RHS-EF-TRAIN-01- 031523-N 23C0146-02	N	8:2 Fluorotelomer sulfonic acid (8:2 FTS)	1.40	0.140 FJ	0.140 J	-	ng/l	TR/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 23C0146

### Quality Control Outliers for test method E1633DR, Lab Blank

The purpose of laboratory blanks is to determine the existence and magnitude of cross-contamination problems resulting from laboratory activities. Reported results were evaluated to determine compliance with the required acceptance criteria. Summary forms were evaluated and compared to electronic data deliverables. Findings of this review, and contaminants found in laboratory blanks are listed below along with any associated qualified results.

Sample ID/ Lab Sample ID	Analyte	Result	Warning Limits	Control Limits	Units	Qualifier	Reason Code	Comment
BCC0177-BLK1 (LB)	Perfluorooctanesul fonamide (PFOSA)	0.295	< 0.1	< 0.4	ng/l	U/None*	L	
BCC0177-BLK1 (LB)	Perfluorooctanesul fonic acid (PFOS)	0.0941	< 0.064	< 0.4	ng/l	U/None*	L	

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.

\*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 LOD or LOQ will be qualified based on the validation guidance assigned in the project setup.

### Qualified Results associated with the Lab Blank for E1633DR

FieldSample ID	Type	Analyte	LOQ	Lab Result	Qualified Result	Bias	Units	Reason
RHS-EF-TRAIN-01- 031523-N 23C0146-02	N	Perfluorooctanesulfonic acid (PFOS)	0.350	0.110 FJ	0.180 U		ng/l	L

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 23C0146

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
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	0.710	0.350 U	0.350 UJ		ng/l	V6
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	2H,2H,3H,3H-Perfluorooctanoic acid (5:3FTCA)	1.40	0.710 U	0.710 UJ		ng/l	V6
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	8:2 Fluorotelomer sulfonic acid (8:2 FTS)	1.40	0.140 FJ	0.140 J	-	ng/l	TR/Y5
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	0.710	0.350 U	0.350 UJ		ng/l	V6
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	0.710	0.350 U	0.350 UJ		ng/l	V6
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	Perfluorobutanesulfonic acid	0.350	0.0370 FI J	0.0370 J		ng/l	TR/Z5
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	Perfluorooctanesulfonic acid (PFOS)	0.350	0.110 FJ	0.180 U		ng/l	L
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	Perfluoropentanesulfonic acid (PFPeS)	0.350	0.180 U	0.180 UJ		ng/l	V2
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	0.690	0.340 U	0.340 UJ		ng/l	V6
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	2H,2H,3H,3H-Perfluorooctanoic acid (5:3FTCA)	1.40	0.690 U	0.690 UJ		ng/l	V6
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	0.690	0.340 U	0.340 UJ		ng/l	V6
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	0.690	0.340 U	0.340 UJ		ng/l	V6
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	Perfluorohexanoic acid (PFHxA)	0.340	0.720	0.720 J	+	ng/l	C
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	Perfluoropentanesulfonic acid (PFPeS)	0.340	0.0930 FJ	0.0930 J	+	ng/l	TR/V2

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 23C0146

### 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
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	0.710	0.350 U	0.350 U	0.350 UJ	V6
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	2H,2H,3H,3H-Perfluorooctanoic acid (5:3FTCA)	1.40	0.710 U	0.710 U	0.710 UJ	V6
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	0.710	0.350 U	0.350 U	0.350 UJ	V6
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	0.710	0.350 U	0.350 U	0.350 UJ	V6
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	Perfluorobutanesulfonic acid	0.350	0.0370 FI J	0.0370 J	0.0370 J	TR/Z5
RHS-EF-TRAIN-01-031523-N 23C0146-02	N	Perfluoropentanesulfonic acid (PFPeS)	0.350	0.180 U	0.180 U	0.180 UJ	V2
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	0.690	0.340 U	0.340 U	0.340 UJ	V6
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	2H,2H,3H,3H-Perfluorooctanoic acid (5:3FTCA)	1.40	0.690 U	0.690 U	0.690 UJ	V6
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	0.690	0.340 U	0.340 U	0.340 UJ	V6
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA)	0.690	0.340 U	0.340 U	0.340 UJ	V6
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	Perfluorohexanoic acid (PFHxA)	0.340	0.720	0.720	0.720 J	C
RHSF-PUMP-PR-01-031523-N 23C0146-01	N	Perfluoropentanesulfonic acid (PFPeS)	0.340	0.0930 FJ	0.0930 J	0.0930 J	TR/V2

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
L	Lab Blank
TR	Trace Level Detect
V2	CCV
V6	Low Level Calibration Verification
Y5	Extracted Internal Standard
Z5	Ion Ratio



## Data Validation Report for 23C0146

### 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.
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 23C0146

## Review Questions

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