



## DATA VALIDATION REPORT

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

**SDG: 25C0006  
APPL, Inc.**

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

Prepared for  
**AECOM Environmental**

**Released: 04/04/25**

### **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

A handwritten signature in black ink, appearing to read "Miranda Maines".

Miranda Maines

### EXECUTIVE NARRATIVE

**Sample Delivery Group:** 25C0006

**Laboratory:** APPL, Inc.

**Site:** RI PFAS Release, CTO 23F0178

**Sampling dates:** 02/27/2025

**Number of Samples:** 4

**Test Method:** USEPA Method 1633

**Analysis:** per- and polyfluoroalkyl substances (PFAS)

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

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

| Client Sample Identification | Laboratory Sample Identification | Matrix  | Validation Stage |
|------------------------------|----------------------------------|---------|------------------|
| JV130                        | 25C0006-01                       | aqueous | S2BVM            |
| JV131                        | 25C0006-02                       | aqueous | S2BVM            |
| JV132                        | 25C0006-03                       | aqueous | S2BVM            |
| JV128                        | 25C0006-04                       | aqueous | S2BVM            |

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

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

## DATA ASSESSMENT

### 1. NARRATIVE AND COMPLETENESS REVIEW

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

### 2. SAMPLE DELIVERY AND CONDITION

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

### 3. HOLDING TIME

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

*No problems were found for this criterion.*

### 4. CALIBRATION

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

#### A) Initial Calibration

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

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

*No problems were found for this criterion.*

#### B) Continuing Calibration

The Percent Recovery (%R) for all target analytes in the continuing calibration must be within 70-130%. All initial calibration verification (ICV) and continuing calibration verification (CCV) %Rs were within 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  $\pm 30\%$ .

*No problems were found for this criterion.*

## **5. BLANK CONTAMINATION**

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

### **A) Method blank contamination:**

*No problems were found for this criterion.*

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

*No problems requiring result qualification were found for this criterion.*

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

*No samples were collected as field / equipment blanks in association with the samples in this sample delivery group (SDG).*

## **6. EXTRACTED INTERNAL STANDARDS**

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

*No problems requiring qualification of sample results were found for this criterion with the following exceptions. The observed recoveries for labeled standards 13C5-PFPEA and 13C5-PFHXA were less than the lowest acceptance limits during the initial analyses of samples JV131 and JV132. The results reported for the associated target analytes in the impacted samples have been qualified "UJ" 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.*

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

## 9. COMPOUND QUANTIFICATION

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

## 10. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

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

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

## 11. FIELD DUPLICATES

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

*Samples JV131 and JV132 were submitted as a field duplicate pair in association with this SDG. Adequate field precision was demonstrated.*

## 12. LABORATORY CONTROL SAMPLES

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

*No problems were found for this criterion.*

**13. DILUTIONS, RE-EXTRACTIONS & REANALYSIS**

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

*Sample reanalyses and dilutions were conducted as necessary due to high analyte concentration or to confirm quality control results.*

**14. SYSTEM PERFORMANCE AND OVERALL ASSESSMENT**

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

**Table 1**  
**Review Elements Summary**

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

Major= Major data quality issue identified resulting in rejection of data.

Minor= Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.

NA = Not applicable

**Table 2**  
**Data Validation Qualifiers**

| <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. |

**Table 3**  
**PFAS Definitions Table**

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

## **Qualified Sample Result Summaries**

## Summary of Qualified Results

| Sample | Lab ID     | Analyte                                | Result | lab_qualifiers | validator_qualifiers | interpreted_qualifiers | result_unit | Reason_code |
|--------|------------|----------------------------------------|--------|----------------|----------------------|------------------------|-------------|-------------|
| JV131  | 25C0006-02 | 3:3 Fluorotelomer carboxylic acid      |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV131  | 25C0006-02 | Perfluoro-3-methoxypropanoic acid      |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV131  | 25C0006-02 | 5:3 Fluorotelomer carboxylate          | 3.4    |                | J+                   | J+                     | NG_L        | V           |
| JV131  | 25C0006-02 | Perfluoro-4-methoxybutanoic Acid       |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV131  | 25C0006-02 | Perfluoro[2-ethoxyethane]sulfonic acid |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV131  | 25C0006-02 | Perfluoro-3,6-dioxahexanoic acid       |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV131  | 25C0006-02 | 7:3 Fluorotelomer carboxylate          |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV132  | 25C0006-03 | 3:3 Fluorotelomer carboxylic acid      |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV132  | 25C0006-03 | Perfluoro-3-methoxypropanoic acid      |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV132  | 25C0006-03 | 5:3 Fluorotelomer carboxylate          | 5.0    |                | J+                   | J+                     | NG_L        | V           |
| JV132  | 25C0006-03 | Perfluoro-4-methoxybutanoic Acid       |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV132  | 25C0006-03 | Perfluoro[2-ethoxyethane]sulfonic acid |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV132  | 25C0006-03 | Perfluoro-3,6-dioxahexanoic acid       |        | U              | UJ                   | UJ                     | NG_L        | V           |
| JV132  | 25C0006-03 | 7:3 Fluorotelomer carboxylate          |        | U              | UJ                   | UJ                     | NG_L        | V           |

**Table II: Qualification Code Reference Table**

| Qualifier | Organics                                                                                                                                | Inorganics                                                                                                                              |
|-----------|-----------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------|
| H         | Holding times were exceeded.                                                                                                            | Holding times were exceeded.                                                                                                            |
| S         | Surrogate recovery was outside QC limits.                                                                                               | The sequence or number of standards used for the calibration was incorrect.                                                             |
| C         | Calibration %RSD, $r_1$ , $r_2$ or %D were noncompliant                                                                                 | Correlation coefficient is <0.995.                                                                                                      |
| R         | Calibration RRF was <0.05.                                                                                                              | %R for calibration is not within control limits                                                                                         |
| B         | Presumed contamination from preparation (method blank)                                                                                  | Presumed contamination from preparation (method) blank or calibration blank                                                             |
| L         | Laboratory Control Sample/Laboratory Control Sample Duplicate %R or RPD was not within control limits                                   | Laboratory Control Sample/Laboratory Control Sample Duplicate %R or RPD was not within control limits                                   |
| Q         | MS/MSD recovery was poor                                                                                                                | MS/MSD recovery was poor.                                                                                                               |
| E         | MS/MSD or Duplicate RPD was high.                                                                                                       | MS/MSD or Duplicate RPD or difference was high.                                                                                         |
| I         | Internal standard performance was unsatisfactory                                                                                        | ICP ICS results were unsatisfactory.                                                                                                    |
| A         | Not applicable.                                                                                                                         | ICP Serial Dilution %D were not within control limits                                                                                   |
| M         | Instrument Performance Check (BFB or DFTPP) was noncompliant                                                                            | Not applicable.                                                                                                                         |
| T         | Presumed contamination from trip blank.                                                                                                 | Not applicable.                                                                                                                         |
| F         | Presumed contamination from FB or ER.                                                                                                   | Presumed contamination from FB or ER.                                                                                                   |
| D         | The analysis with this flag should not be used because another more technically sound analysis is available.                            | The analysis with this flag should not be used because another more technically sound analysis is available.                            |
| P         | Instrument performance for pesticides was poor                                                                                          | Post Digestion Spike recovery was not within control limits                                                                             |
| V         | Unusual problems found with the data that have been described in the validation report where a description of the problem can be found. | Unusual problems found with the data that have been described in the validation report where a description of the problem can be found. |

**Data Validation Worksheet**

# DATA VALIDATION PFAS

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

Validator: GAP

Reviewer: DLW

Date Validated: 04/02/2025

Reviewed: 4/4/25

Project: Red Hill

SDG: 25C0006

LAB: APPL

Samples Collected: 02/27/2025

4 WATER

### 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 3/1 at 2.7°C

### 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                                                              |
| <b>Aqueous</b>          | 28 days                                 | Precursor degradation occurs after 7 days                                      | 90 days                                 | None                                                                |
| <b>Solid and Tissue</b> | 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 |
| <b>Biosolid</b>         | 90 days                                 | Not recommended due to the production of gases due to microbiological activity | 90 days                                 | None                                                                |

Samples collected 02/27

Extracted 03/05

Analyzed 03/06 and 03/11

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

Per QAPP:

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

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

All ok except

JV131 (25C0006-02 ) initial

|             |   |                                                                                                              |
|-------------|---|--------------------------------------------------------------------------------------------------------------|
| 13C5-PFPEA  | ↓ | PFPeA reported from 100X; no action<br>3:3FTCA flag UJ<br>PFMPA flag UJ<br>PFMBA flag UJ                     |
| 13C5-PFHXA  | ↓ | PFHxA reported from 100X; no action<br>5:3FTCA FLAG J+<br>7:3FTCA flag UJ<br>PFEESA flag UJ<br>NFDHA flag UJ |
| 13C2-6:2FTS | ↑ | 6:2 FTS REPORTED FROM 1000x; NO ACTION                                                                       |

JV132 (25C0006-03 ) initial

|             |   |                                                                                                              |
|-------------|---|--------------------------------------------------------------------------------------------------------------|
| 13C5-PFPEA  | ↓ | PFPeA reported from 100X; no action<br>3:3FTCA flag UJ<br>PFMPA flag UJ<br>PFMBA flag UJ                     |
| 13C5-PFHXA  | ↓ | PFHxA reported from 100X; no action<br>5:3FTCA FLAG J+<br>7:3FTCA flag UJ<br>PFEESA flag UJ<br>NFDHA flag UJ |
| 13C2-6:2FTS | ↑ | 6:2 FTS REPORTED FROM 1000x; NO ACTION                                                                       |

## Non-Extracted Internal STANDARDS

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

All ok

## Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (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 lab limits to evaluate  
All 40 compounds included.

MRL Check (BEC0032-MRL1) all ok  
LCS (BEC0032-BS1) all ok

## MS/MSD and Matrix Duplicate

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

Use lab limits to evaluate  
Sample: none

## BLANKS

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

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

Table A: Sample Qualification in the Presence of Blank Contamination

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

*LOD = Limit of Detection*

FB/EBs  
none

**Blank (BEC0032-BLK1)**  
ND

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](#) Fafnir

03/01/25

all %RSE <20%

Initial Cal Blank SE00753-ICB1 F2025-02-28B (9) 03/01/25 02:42

NMeFOSAA 0.03 J ng/ml ND; no action

NEtFOSAA 0.05 J ng/ml ND; no action

Secondary Cal Check SE00753-SCV1 F2025-02-28B (10) 03/01/25 03:03

Low Cal Check SE00794-LCV1 F2025-03-05A (2) 03/05/25 16:03  
Calibration Check SE00794-CCV2 F2025-03-05A (21) 03/05/25 22:46  
Calibration Blank SE00794-CCB3 F2025-03-05A (22) 03/05/25 23:07

Samples 1-3

Calibration Check SE00794-CCV3 F2025-03-05A (30) 03/06/25 01:56  
Calibration Blank SE00794-CCB4 F2025-03-05A (31) 03/06/25 02:18

Instrument Elliott

03/05/25 all %RSE <20%

Initial Cal Blank SE00796-ICB1 E2025-03-05A (9) 03/05/25 18:40  
Secondary Cal Check SE00796-SCV1 E2025-03-05A (10) 03/05/25 19:01  
Low Cal Check SE00817-LCV1 E2025-03-06A (2) 03/06/25 17:26  
Calibration Check SE00817-CCV1 E2025-03-06A (3) 03/06/25 17:47  
Calibration Blank SE00817-CCB2 E2025-03-06A (5) 03/06/25 18:28

Samples 02re1,re2,re3; sample 3re1,re2,re3

Calibration Check SE00817-CCV2 E2025-03-06A (12) 03/06/25 20:52  
Calibration Blank SE00817-CCB3 E2025-03-06A (13) 03/06/25 21:12  
5:3FTCA 0.11 J ng/ml not reported in associated samples

Instrument Fafnir

03/10/25 all %RSE <20%

Initial Cal Blank SE00857-ICB1 F2025-03-10A (9) 03/10/25 19:44  
NMeFOSAA 0.05 J ng/ml ND; no action  
NEtFOSAA 0.04 J ng/ml ND; no action  
Secondary Cal Check SE00857-SCV1 F2025-03-10A (10) 03/10/25 20:06  
Low Cal Check SE00877-LCV1 F2025-03-11A (2) 03/11/25 15:21  
Calibration Check SE00877-CCV1 F2025-03-11A (3) 03/11/25 15:42  
Calibration Blank SE00877-CCB2 F2025-03-11A (5) 03/11/25 16:24

Sample 4

Calibration Check SE00877-CCV2 F2025-03-11A (16) 03/11/25 20:18  
Calibration Blank SE00877-CCB3 F2025-03-11A (17) 03/11/25 20:39

## 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

AECOM DVA SOP Reason Code: V

All ok

## FIELD DUPLICATES

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

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

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RPD  $\leq 50\%$  water <sup>c</sup>  
 RPD  $\leq 100\%$  soil/sediment (judgmental) <sup>c</sup>

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

JV131 and JV132      all ok