

# DATA VALIDATION REPORT

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

> SDG: FC3825 SGS Orlando, FL

Prepared by ENVIRONMENTAL DATA SERVICES, LTD.

Prepared for **AECOM Environmental** 

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# **EXECUTIVE NARRATIVE**

Sample Delivery Group: FC3825 Laboratory: SGS North America Inc - Orlando Site: Red Hill Bulk Storage Facility, CV 23F0104 Sampling dates: 3/29/2023 Number of Samples: 1 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<br>Sample<br>Identification | Matrix      | Validation<br>Stage |
|------------------------------|--|-------------|---------------------|
| AF-RHMW225401-WGN01B-2303W4  | FC3825-1                               | groundwater | 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 recovery for 7:3FTCA was outside of acceptance limits for the ISC associated with the sample in this SDG. The result reported for the impacted analyte in the associated sample has been qualified "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:

No sample was submitted as a field blank in association with the samples in this sample delivery group (SDG).

## 6. EXTRACTED INTERNAL STANDARDS

All samples are spiked with labeled standard compounds prior to sample preparation and analyses to evaluate overall laboratory performance and efficiency of the analytical technique. The reported project samples had observed surrogate recoveries within the established limits in all cases with the following exceptions. No problems were found for this criterion.

#### 7. NON-EXTRACTED INTERNAL STANDARDS

Non-extracted internal standard peak areas are used to quantify extracted internal standard recoveries. The reported project samples had non-extracted internal standard area counts within the established limits in all cases with the following exceptions.

No problems were found for this criterion.

#### 8. COMPOUND IDENTIFICATION

The project target analyte compounds are identified on the LC/MS/MS by using the analytes retention time (RT). The retention time of each target analyte should be within  $\pm$  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.

#### 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-RHMW225401-WGN01B-2303W4 was submitted for MS evaluations in association with this SDG. Upon evaluation all accuracy indicators were acceptable.

#### 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 be used when original samples and its duplicate.

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.

#### 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? |       | criteria |
|---|-------------------------------|-------|----------|
|   | Yes                           | No    |          |
| Per-fluorinated Compounds                                   |                               | Major | Minor    |
| Holding Time/Sample Handling                                | X                             |       |          |
| Method Blanks   | X                             |       |          |
| Instrument Blanks   | х                             |       |          |
| Field Blanks  | NA                            |       |          |
| Calibration Percent Relative Standard Deviation and Percent |                               |       |          |
| Difference  | x                             |       |          |
| Instrument Sensitivity Check                                |                               |       | Х        |
| Extracted Internal Standards                                | х                             |       |          |
| Non-Extracted Internal Standards                            | х                             |       |          |
| Compound Identification                                     | х                             |       |          |
| Matrix Spike/Matrix Spike Duplicate                         | х                             |       |          |
| 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

| 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 2Data Validation Qualifiers

| Table 3                 |       |
|-------------------------|-------|
| <b>PFAS Definitions</b> | Table |

| NO | CAS #       | Target Name   | Target Abbreviation |
|----|-------------|---|---------------------|
| 1  | 763051-92-9 | 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11CI-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    | 9CI-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               |

| Data Qualificat | ion Reason Codes  |  |  |
|-----------------|---|--|--|
|                 |   |  |  |
| Reason Code     | Reason Code Description   |  |  |
| Α               | Serial dilution   |  |  |
| A1              | Ambient Blank   |  |  |
| В               | The analyte was found in an associated blank as well as in the sample.  |  |  |
| B2              | ССВ   |  |  |
| B3              | CCB - Neg   |  |  |
| B4              | Grinding Blank  |  |  |
| С               | 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/r^2/r   |  |  |
| G3              | ICV RRF   |  |  |
| H1              | Test Hold Time  |  |  |
| H2              | Prep Hold Time  |  |  |
| I               | Surrogate recovery outside project limits.  |  |  |
| J               | CRA/CRI Recovery  |  |  |
| к               | 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   |  |  |
| М               | MS Recovery   |  |  |
| M2              | Post Spike  |  |  |
| N               | Blank - No Action   |  |  |
| 0               | ICS   |  |  |
| Р               | 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 LinearCalibration Range  |
| S  | Internal standard  |
| Т  | 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)   |
| Х  | Raised reporting limit   |
| Y  | Cooler temperature greater than 10 degreec 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 Re Date Validated: 4/11/2023 Re Project: Red Hill SDG: FC3825 LAB: SGS North America Inc. - Orlando Samples Collected: 3/29/2023 1 GW

Reviewer: DM

Reviewed: 4/14/2023

# 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 3/30 at 2.1C

# HOLDING TIMES

- ✓ Recommended storage temp is ≤ -20°C
- ✓ Per method 1633: aqueous samples may be held in the lab for up to 90 days when stored at recommended temp and protected from light; when stored at 0-6 °C and protected from light samples can be held for up to 28 days (see method for additional details)
- ✓ Per method 1633: solid samples may be held in the lab for up to 90 days when stored at recommended temp or 0-6 °C (see method for additional details)
- ✓ Per method 1633: biosolid samples may be held in the lab for up to 90 days when stored at recommended temp or 0-6 °C; however, freezing is recommended (see method for additional details)
- ✓ Samples extracts should be stored at 0-4°C protected from light and analyzed within 90 days

- ✓ If hold time is exceeded qualify J/UJ
- ✓ If hold time is grossly exceeded (2X hold time) J/X

| Matrix<br>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<br>degradation<br>occurs after 7<br>days   | 90 days                                 | None   |
| Solid and<br>Tissue | 90 days                                 | Should be<br>prepared as<br>soon as<br>possible if<br>NFDHA is a<br>target analyte               | 90 days                                 | Should be prepared<br>as soon as possible<br>if NFDHA is a target<br>analyte |
| Biosolid            | 90 days                                 | Not<br>recommended<br>due to the<br>production of<br>gases due to<br>microbiological<br>activity | 90 days                                 | None   |

# 244 Table II. Sample Storage and Holding Time Requirements

Samples collected 3/29/23 Extracted 3/31 Analyzed 4/1

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-. Noddetects 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)

# All ok

# Non-Extracted Internal STANDARDS

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

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.

OP96191-BS all ok

OP96191-LLBS 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:

AF-RHMW225401-WGN01B-2303W4 MS all ok

# 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

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

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

313 LOD = Limit of Detection

none

# OP96191-MB all ND

All instrument blanks 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</p>
- ✓ 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</p>
- ✓ 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</p>
- ✓ 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

## 4/4/2023

all %RSD <20%

S6Q239-ICV239 6Q16015.D 04/04/23 16:21 03:11 Initial cal verification 4 S6Q239-ICV239 6Q16016.D 04/04/23 16:35 03:25 Initial cal verification 20

S6Q240-CC239 6Q16134.D 04/05/23 20:47 07:32 Continuing cal 1.0LL 7:3FTCA >130% S6Q240-CC239 6Q16144.D 04/05/23 23:07 09:52 Continuing cal 4 sample S6Q240-CC239 6Q16144.D 04/05/23 23:07 09:52 Continuing cal 4

## COMPOUND INDENTIFICATION

- $\checkmark$  RT within <u>+</u>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

SEE FIELD DUPLICATE WORKSHEET

# **Data Validation Report for FC3825**

| Facility:                 | RH Fire Suppression System             |
|---------------------------|--|
| Event:                    | AFFF Assessment Sampling GW 2023 March |
| SDG:                      | FC3825                                 |
| 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-RHMW225401-WGN01B-<br>2303W4 | FC3825-1      | Water  | Field Sample/N | Х       |

## **Data Validation Report for FC3825**

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) FC3825. 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
- 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 1 results (2.50%) out of the 40 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. 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.

No Outliers were associated with this sample delivery group.

#### **Table of All Qualified Results**

| Test Method: E1633DR                        |      | Extraction Method: METHOD                     |     |            |                  |      |       |        |
|---|------|---|-----|------------|------------------|------|-------|--------|
| FieldSample ID /<br>LabSample ID            | Туре | Analyte                                       | LOQ | Lab Result | Qualified Result | Bias | Units | Reason |
| AF-RHMW225401-<br>WGN01B-2303W4<br>FC3825-1 | Ν    | 3-Perfluoroheptyl<br>propanoic acid (7:3FTCA) | 120 | 19.0 U     | 19.0 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.

#### Table of Results with Modified Qualifiers

| Modified Qualifiers for test method E1633DR |      |   |     |            |            |                 |        |
|---|------|---|-----|------------|------------|-----------------|--------|
| FieldSample ID /<br>LabSample ID            | Туре | Analyte                                       | LOQ | Lab Result | ADR Result | Modified Result | Reason |
| AF-RHMW225401-<br>WGN01B-2303W4<br>FC3825-1 | Ν    | 3-Perfluoroheptyl<br>propanoic acid (7:3FTCA) | 120 | 19.0 U     | 19.0 U     | 19.0 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 |   |
|------|------------|---|
|      |            | _ |

#### **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<br>and the associated numerical value was the estimated concentration in<br>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.               |
| Х    | Result may require rejection; PDT attention required  |

#### Bias

| Dius |   |  |  |  |
|------|---|--|--|--|
| -    | The result may be biased low  |  |  |  |
| +    | The result may be biased high   |  |  |  |
| Note | Note - The bias field is a separate field; however, it is an integral part of the final flag (qualifier) on the sample result |  |  |  |

**Review Questions**