LABORATORY DATA CONSULTANTS, INC.
2701 Loker Ave. West, Suite 220, Carlsbad, CA 92010 Bus: 760-827-1100 Fax: 760-827-1099

Tidewater
3761 Attucks Drive
Powell, OH 43065
ATTN: Mr. David Conner
David.Conner@tideh2o.net

April 6, 2023

SUBJECT: NASA JPL - Data Validation

Dear Mr. Conner,

Enclosed are the final validation reports for the fractions listed below. These SDGs were received on February 14, 2023. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project #56164:

SDG #	Fraction
22L004 22L005	Perfluoroalkyl & Polyfluoroalkyl Substances

The data validation was performed under Level III & IV guidelines. The analysis was validated using the following documents, as applicable to each method:

- USEPA Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFAS) Analyzed Using EPA Method 537 (November 2018)

Please feel free to contact us if you have any questions.

Sincerely,

Pei Geng
pgeng@lab-data.com
Project Manager/Senior Chemist

166 pages-ADV

Attachment 1

90/10 III/IV EDD

LDC# 56164 (Tidewater - Powell, OH / NASA JPL)

LDC	SDG#	DATE REC'D	(3) DATE DUE	PFAs (QSM5.3 B-15)																																	
Matrix:	Water/Soil			W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S
A	22L004	02/14/23	03/08/23	25	0																																
A	22L004	02/14/23	03/08/23	3	0																																
B	22L005	02/14/23	03/08/23	2	12																																
B	22L005	02/14/23	03/08/23	0	2																																
Total	TR/PG			30	14	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	44

Shaded cells indicate Level IV validation (all other cells are Level III validation). These sample counts do not include MS/MSD, and DUPs

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Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name: NASA JPL

LDC Report Date: April 5, 2023

Parameters: Perfluoroalkyl & Polyfluoroalkyl Substances

Validation Level: Level III & IV

Laboratory: APPL, Inc., Clovis, CA

Sample Delivery Group (SDG): 22L004

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
MW-24-S5-112922	22L0004-01	Water	11/29/22
MW-24-S4-112922	22L0004-02	Water	11/29/22
MW-24-S3-112922**	22L0004-03**	Water	11/29/22
MW-24-S2-112922	22L0004-04	Water	11/29/22
DUP-2-112922	22L0004-05	Water	11/29/22
MW-17-S5-112922	22L0004-06	Water	11/29/22
MW-17-S4-112922	22L0004-07	Water	11/29/22
MW-17-S3-112922**	22L0004-08**	Water	11/29/22
DUP-3-112922	22L0004-09	Water	11/29/22
MW-17-S2-112922	22L0004-10	Water	11/29/22
SB-2-112922	22L0004-11	Water	11/29/22
MW-4-S5-112822	22L0004-12	Water	11/28/22
DUP-1-112822	22L0004-13	Water	11/28/22
MW-4-S4-112822	22L0004-14	Water	11/28/22
MW-4-S2-112822	22L0004-15	Water	11/28/22
MW-12-S5-112822	22L0004-16	Water	11/28/22
MW-12-S4-112822**	22L0004-17**	Water	11/28/22
MW-12-S3-112822	22L0004-18	Water	11/28/22
MW-12-S2-112822	22L0004-19	Water	11/28/22
SB-1-112822	22L0004-20	Water	11/28/22
FB-1-112822	22L0004-21	Water	11/28/22
EQP-1-112822	22L0004-22	Water	11/28/22
EQP-2-112922	22L0004-23	Water	11/29/22
FB-2-112922	22L0004-24	Water	11/29/22
MW-15-113022	22L0004-25	Water	11/30/22
SB-3-113022	22L0004-26	Water	11/30/22

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
EQP-3-113022	22L0004-27	Water	11/30/22
FB-3-113022	22L0004-28	Water	11/30/22
MW-4-S2-112822MS	22L0004-15MS	Water	11/28/22
MW-4-S2-112822MSD	22L0004-15MSD	Water	11/28/22

**Indicates sample underwent Level IV validation

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with a modified outline of the USEPA Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFAS) Analyzed Using EPA Method 537 (November 2018). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

All sample results were subjected to Level III data validation, which comprises an evaluation of quality control (QC) summary results. Samples appended with a double asterisk on the cover page were subjected to Level IV data validation, which is comprised of the QC summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

J (Estimated): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.

U (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).

UJ (Non-detected estimated): The analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.

R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.

NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

II. LC/MS Instrument Performance Check

Instrument performance was checked and the requirements were met.

III. Initial Calibration and Initial Calibration Verification

Initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes.

For each calibration standard, all analytes were within 70-130% of their true value.

The signal to noise (S/N) ratio was within validation criteria for all analytes associated to samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

Retention time windows were established as required by the method for samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all analytes.

IV. Continuing Calibration and Instrument Sensitivity Check

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 30.0% for all analytes.

The signal to noise (S/N) ratio was within validation criteria for all analytes associated to samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

The percent differences (%D) of the instrument sensitivity check (ISC) were less than or equal to 30.0% for all analytes with the following exceptions:

Date	Analyte	%D	Associated Samples	Flag	A or P
12/09/22	6:2FTS NEtFOSE	31.0 40.0	MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-24-S2-112922 DUP-2-112922 MW-17-S5-112922 MW-17-S4-112922 MW-17-S3-112922** DUP-3-112922 MW-17-S2-112922 SB-2-112922 MW-4-S5-112822 DUP-1-112822 MW-4-S4-112822 MW-4-S2-112822 MW-12-S5-112822 MW-12-S4-112822** MW-12-S3-112822 MW-12-S2-112822 SB-1-112822	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A
12/15/22 (SB03858-LSV1)	PFUnA	37.2	EQP-1-112822 EQP-2-112922 MW-15-113022 EQP-3-113022	UJ (all non-detects)	A
12/15/22 (SB03860-LSV1)	PFDA HFPO-DA PFDOA	93.7 38.8 33.9	FB-1-112822 FB-2-112922 FB-3-113022	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	A

Retention times of all analytes in the calibration standards were within the established retention time windows for samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

All analyte concentrations were at the limit of quantitation (LOQ) for the ISC standard.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

Blank ID	Extraction Date	Analyte	Concentration	Associated Samples
BBL0296-BLK1	12/13/22	PFOA PFOS	0.194 ng/L 0.276 ng/L	EQP-1-112822 EQP-2-112922 MW-15-113022 EQP-3-113022

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater (>5X) than the concentrations found in the associated laboratory blanks with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
EQP-1-112822	PFOA	0.23 ng/L	0.23J ng/L
EQP-2-112922	PFOS	0.11 ng/L	0.18U ng/L
EQP-3-113022	PFOA	0.21 ng/L	0.21J ng/L

VI. Field Blanks

Samples EQP-1-112822, EQP-2-112922, and EQP-3-113022 were identified as equipment blanks. No contaminants were found with the following exceptions:

Blank ID	Collection Date	Analyte	Concentration	Associated Samples
EQP-1-112822	11/28/22	PFOA	0.23 ng/L	MW-4-S5-112822 DUP-1-112822 MW-4-S4-112822 MW-4-S2-112822 MW-12-S5-112822 MW-12-S4-112822** MW-12-S3-112822 MW-12-S2-112822
EQP-2-112922	11/29/22	PFOS	0.11 ng/L	MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-24-S2-112922 DUP-2-112922 MW-17-S5-112922 MW-17-S4-112922 MW-17-S3-112922** DUP-3-112922 MW-17-S2-112922
EQP-3-113022	11/30/22	PFOS	0.21 ng/L	MW-15-113022

Samples SB-2-112922, SB-1-112822, and SB-3-113022 were identified as source blank. No contaminants were found with the following exceptions:

Blank ID	Collection Date	Analyte	Concentration	Associated Samples
SB-2-112922	11/29/22	PFOA PFOS	0.19 ng/L 0.059 ng/L	MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-24-S2-112922 DUP-2-112922 MW-17-S5-112922 MW-17-S4-112922 MW-17-S3-112922** DUP-3-112922 MW-17-S2-112922

Blank ID	Collection Date	Analyte	Concentration	Associated Samples
SB-1-112822	11/28/22	PFOA PFOS	0.17 ng/L 0.12 ng/L	MW-4-S5-112822 DUP-1-112822 MW-4-S4-112822 MW-4-S2-112822 MW-12-S5-112822 MW-12-S4-112822** MW-12-S3-112822 MW-12-S2-112822

Samples FB-1-112822, FB-2-112922, and FB-3-113022 were identified as field blanks. No contaminants were found.

Sample concentrations were compared to concentrations detected in the field blanks. The sample concentrations were either not detected or were significantly greater (>10X) than the concentrations found in the associated field blanks with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
DUP-1-112822	PFOA	0.94 ng/L	0.94J ng/L
MW-24-S5-112922	PFOA	0.21 ng/L	0.21J ng/L
MW-24-S4-112922	PFOA	0.26 ng/L	0.26J ng/L
MW-24-S2-112922	PFOA	0.68 ng/L	0.68J ng/L
DUP-2-112922	PFOA	0.83 ng/L	0.83J ng/L
MW-17-S2-112922	PFOA	0.23 ng/L	0.23J ng/L
MW-4-S5-112822	PFOA PFOS	0.27 ng/L 0.14 ng/L	0.27J ng/L 0.14J ng/L
MW-4-S4-112822	PFOA PFOS	0.40 ng/L 0.15 ng/L	0.40J ng/L 0.15J ng/L
MW-4-S2-112822	PFOS	0.20 ng/L	0.20J ng/L

VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
MW-4-S2-112822MS/MSD (MW-4-S2-112822)	PFNS	42.2 (69-127)	66.8 (69-127)	UJ (all non-detects)	A

Relative percent differences (RPD) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
MW-4-S2-112822MS/MSD (MW-4-S2-112822)	PFNS PTRDA	44.1 (≤30) 34.1 (≤30)	NA	-

VIII. Laboratory Control Samples/Minimal Risk Levels

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Analyte	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
BBL0249-BS1/BSD1 (FB-1-112822 FB-2-112922 FB-3-113022)	PFNA PFUnA 8:2FTS NMeFOSAA	140 (69-130) 134 (69-133) 140 (67-138) 153 (65-136)	- - - -	NA	-

Relative percent differences (RPD) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
BBL0076-BS1/BSD1 (MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-24-S2-112922 DUP-2-112922 MW-17-S5-112922 MW-17-S4-112922 MW-17-S3-112922** DUP-3-112922 MW-17-S2-112922 SB-2-112922 MW-4-S5-112822 DUP-1-112822 MW-4-S4-112822 MW-4-S2-112822 MW-12-S5-112822 MW-12-S4-112822** MW-12-S3-112822 MW-12-S2-112822 SB-1-112822)	PFDOA	35.0 (≤30)	NA	-

LCS ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
BBL0296-BS1/BSD1 (EQP-1-112822 EQP-2-112922 MW-15-113022 EQP-3-113022)	PFUnA	37.3 (≤20)	NA	-
BBL0371-BS1/BSD1 (SB-3-113022)	PFTEDA	36.2 (≤20)	NA	-

Minimal risk levels (MRL) percent recoveries were within QC limits with the following exceptions:

MRL ID (Associated Samples)	Analyte	%R (Limits)	Flag	A or P
BBL0076-MRL1 (MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-24-S2-112922 DUP-2-112922 MW-17-S5-112922 MW-17-S4-112922 MW-17-S3-112922** DUP-3-112922 MW-17-S2-112922 SB-2-112922 MW-4-S5-112822 DUP-1-112822 MW-4-S4-112822 MW-4-S2-112822 MW-12-S5-112822 MW-12-S4-112822** MW-12-S3-112822 MW-12-S2-112822 SB-1-112822)	8:2FTS	64.9 (67-138)	UJ (all non-detects)	P
BBL0076-MRL1 (MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-17-S4-112922 MW-17-S3-112922**)	PFOSA	142 (67-137)	J (all detects)	P
BBL0076-MRL1 (MW-24-S2-112922 DUP-2-112922 MW-17-S5-112922 DUP-3-112922 MW-17-S2-112922 SB-2-112922 MW-4-S5-112822 DUP-1-112822 MW-4-S4-112822 MW-4-S2-112822 MW-12-S5-112822 MW-12-S4-112822** MW-12-S3-112822 MW-12-S2-112822 SB-1-112822)	PFOSA	142 (67-137)	NA	-

MRL ID (Associated Samples)	Analyte	%R (Limits)	Flag	A or P
BBL0249-MRL1 (FB-1-112822 FB-2-112922 FB-3-113022)	PFDOA PFOSA	137 (72-134) 200 (67-137)	NA	-
BBL0296-MRL1 (EQP-1-112822 EQP-2-112922 MW-15-113022 EQP-3-113022)	PFUnA PFDOA PFTEDA PFNS PFDS NMeFOSAA NMeFOSE 11CL-PF3OUDS	135 (69-133) 148 (72-134) 175 (71-132) 133 (69-127) 150 (53-142) 211 (65-136) 165 (70-130) 139 (54-138)	NA	-
BBL0371-MRL1 (SB-3-113022)	PFTEDA NMeFOSE	62.1 (71-132) 64.0 (70-130)	UJ (all non-detects) UJ (all non-detects)	P
BBL0371-MRL1 (SB-3-113022)	NEtFOSE	131 (70-130)	NA	-

IX. Field Duplicates

Samples MW-24-S2-112922 and DUP-2-112922, samples MW-17-S3-112922** and DUP-3-112922, and samples MW-4-S5-112822 and DUP-1-112822 were identified as field duplicates. No results were detected in any of the samples with the following exceptions:

Analyte	Concentration (ng/L)		RPD
	MW-24-S2-112922	DUP-2-112922	
PFBA	1.4	1.3	7
PFPEA	2.3	2.4	4
PFHXA	1.5	1.3	14
PFHPA	0.70	0.70	0
PFOA	0.68	0.83	20
PFBS	0.84	0.65	26
PFPES	0.62	0.66	6
PFHXS	2.3	2.2	4
PFOS	0.33	0.55	50

Analyte	Concentration (ng/L)		RPD
	MW-17-S3-112922**	DUP-3-112922	
PFBA	1.0	0.80	22
PFPEA	1.8	1.9	5
PFHXA	1.2	1.4	15
PFHPA	0.68	0.92	30
PFOA	2.3	2.0	14
PFNA	0.37	0.53	36
PFDA	1.4	0.91	42
PFBS	2.2	2.4	9
PFPES	0.56	0.53	6
PFHXS	1.4	1.4	0
PFOS	6.6	5.9	11
PFOSA	0.13	0.18U	32

Analyte	Concentration (ng/L)		RPD
	MW-4-S5-112822	DUP-1-112822	
PFBA	0.36	0.61	52
PFPEA	0.72	1.0	33
PFHXA	0.48	0.97	68
PFHPA	0.28	0.41	38
PFOA	0.27	0.94	111
PFDA	0.19U	0.17	11
PFBS	0.79	4.9	144

Analyte	Concentration (ng/L)		RPD
	MW-4-S5-112822	DUP-1-112822	
PFPES	0.40	0.29	32
PFHXS	0.42	0.44	5
PFOS	0.14	0.18U	25
NEtFOSAA	0.19U	0.21	10

X. Internal Standards

All extracted internal standard percent recoveries (%R) were within QC limits with the following exceptions:

Sample	Internal Standard	%R (Limits)	Affected Analyte	Flag	A or P
MW-24-S5-112922	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE	35.3 (50-150) 37.8 (50-150) 46.3 (50-150)	NMeFOSA NEtFOSA NMeFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
MW-24-S4-112922	D3-NMEFOSA D5-NETFOSA	42.4 (50-150) 43.8 (50-150)	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P
MW-24-S3-112922**	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE D9-NETFOSE	37.3 (50-150) 38.6 (50-150) 42.1 (50-150) 46.7 (50-150)	NMeFOSA NEtFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
MW-24-S2-112922	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE	41.6 (50-150) 39.3 (50-150) 47.3 (50-150)	NMeFOSA NEtFOSA NMeFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
DUP-2-112922	D3-NMEFOSA D5-NETFOSA	47.0 (50-150) 45.1 (50-150)	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P
MW-17-S5-112922	D3-NMEFOSA D5-NETFOSA	41.8 (50-150) 42.2 (50-150)	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P
MW-17-S4-112922	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE D9-NETFOSE	42.0 (50-150) 37.9 (50-150) 45.7 (50-150) 47.9 (50-150)	NMeFOSA NEtFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
MW-17-S3-112922**	D3-NMEFOSA D5-NETFOSA	44.0 (50-150) 44.0 (50-150)	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P
MW-17-S3-112922**	D3-NMEFOSAA	168 (50-150)	NMeFOSAA	NA	-

Sample	Internal Standard	%R (Limits)	Affected Analyte	Flag	A or P
DUP-3-112922	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE D9-NETFOSE	39.5 (50-150) 39.6 (50-150) 43.3 (50-150) 44.3 (50-150)	NMeFOSA NEtFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
DUP-3-112922	D3-NMEFOSAA	191 (50-150)	NMeFOSAA	NA	-
SB-2-112922	D3-NMEFOSA D5-NETFOSA	32.1 (50-150) 39.0 (50-150)	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P
MW-4-S5-112822	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE	29.4 (50-150) 40.7 (50-150) 47.1 (50-150)	NMeFOSA NEtFOSA NMeFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
DUP-1-112822	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE D9-NETFOSE	26.7 (50-150) 39.3 (50-150) 44.6 (50-150) 48.9 (50-150)	NMeFOSA NEtFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
MW-4-S4-112822	D3-NMEFOSA	41.7 (50-150)	NMeFOSAA	UJ (all non-detects)	P
MW-4-S2-112822	D3-NMEFOSA	41.4 (50-150)	NMeFOSAA	UJ (all non-detects)	P
MW-12-S5-112822	13C2-8:2FTS	180 (50-150)	8:2FTS	NA	-
MW-12-S5-112822	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE D9-NETFOSE	43.2 (50-150) 46.3 (50-150) 47.4 (50-150) 45.1 (50-150)	NMeFOSA NEtFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
MW-12-S4-112822**	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE D9-NETFOSE	39.8 (50-150) 41.0 (50-150) 42.6 (50-150) 43.9 (50-150)	NMeFOSA NEtFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
MW-12-S3-112822	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE	43.3 (50-150) 45.1 (50-150) 46.3 (50-150)	NMeFOSA NEtFOSA NMeFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
MW-12-S2-112822	D3-NMEFOSA D5-NETFOSA	47.5 (50-150) 49.8 (50-150)	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P
SB-1-112822	D3-NMEFOSA D5-NETFOSA	31.7 (50-150) 31.4 (50-150)	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P
EQP-1-112822	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE D9-NETFOSE	42.4 (50-150) 35.6 (50-150) 42.4 (50-150) 41.2 (50-150)	NMeFOSA NEtFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
MW-15-113022	D5-NETFOSA	48.3 (50-150)	NEtFOSA	UJ (all non-detects)	P

Sample	Internal Standard	%R (Limits)	Affected Analyte	Flag	A or P
FB-3-113022	D3-NMEFOSA D5-NETFOSA	43.7 (50-150) 39.7 (50-150)	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P

XI. Target Analyte Quantitation

All target analyte quantitations met validation criteria for samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

XII. Target Analyte Identification

All target analyte identifications met validation criteria for samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

All target analyte ion ratios met validation criteria with the following exceptions:

Sample	Finding	Flag	A or P
MW-24-S4-112922 MW-24-S3-112922** MW-17-S5-112922 MW-17-S4-112922 DUP-3-112922 SB-2-112922 MW-12-S5-112822 MW-12-S4-112822** EQP-2-112922	All analytes flagged "IR1" or IR2" by the laboratory due to ion ratio outside QC limits.	J (all detects)	P

XIII. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to ISC %D, MS/MSD %R, MRL %R, internal standard %R, and ion ratio, data were qualified as estimated in twenty-seven samples.

Due to laboratory blank contamination, data were qualified as not detected or estimated in three samples.

Due to equipment blank contamination, data were qualified as estimated in nine samples.

Due to source blank contamination, data were qualified as estimated in eight samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable.

NASA JPL
Perfluoroalkyl & Polyfluoroalkyl Substances - Data Qualification Summary - SDG 22L004

Sample	Analyte	Flag	A or P	Reason
MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-24-S2-112922 DUP-2-112922 MW-17-S5-112922 MW-17-S4-112922 MW-17-S3-112922** DUP-3-112922 MW-17-S2-112922 SB-2-112922 MW-4-S5-112822 DUP-1-112822 MW-4-S4-112822 MW-4-S2-112822 MW-12-S5-112822 MW-12-S4-112822** MW-12-S3-112822 MW-12-S2-112822 SB-1-112822	6:2FTS NEtFOSE	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Continuing calibration (ISC %D)
EQP-1-112822 EQP-2-112922 MW-15-113022 EQP-3-113022	PFUnA	UJ (all non-detects)	A	Continuing calibration (ISC %D)
FB-1-112822 FB-2-112922 FB-3-113022	PFDA HFPO-DA PFDOA	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	A	Continuing calibration (ISC %D)
MW-4-S2-112822	PFNS	UJ (all non-detects)	A	Matrix spike/Matrix spike duplicate (%R)
MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-24-S2-112922 DUP-2-112922 MW-17-S5-112922 MW-17-S4-112922 MW-17-S3-112922** DUP-3-112922 MW-17-S2-112922 SB-2-112922 MW-4-S5-112822 DUP-1-112822 MW-4-S4-112822 MW-4-S2-112822 MW-12-S5-112822 MW-12-S4-112822** MW-12-S3-112822 MW-12-S2-112822 SB-1-112822	8:2FTS	UJ (all non-detects)	P	Minimal risk levels (%R)

Sample	Analyte	Flag	A or P	Reason
MW-24-S5-112922 MW-24-S4-112922 MW-24-S3-112922** MW-17-S4-112922 MW-17-S3-112922**	PFOSA	J (all detects)	P	Minimal risk levels (%R)
SB-3-113022	PFTEDA NMeFOSE	UJ (all non-detects) UJ (all non-detects)	P	Minimal risk levels (%R)
MW-24-S5-112922 MW-24-S2-112922 MW-4-S5-112822 MW-12-S3-112822	NMeFOSA NEtFOSA NMeFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P	Internal standards (%R)
MW-24-S4-112922 DUP-2-112922 MW-17-S5-112922 MW-17-S3-112922** SB-2-112922 MW-12-S2-112822 SB-1-112822 FB-3-113022	NMeFOSA NEtFOSA	UJ (all non-detects) UJ (all non-detects)	P	Internal standards (%R)
MW-24-S3-112922** MW-17-S4-112922 DUP-3-112922 DUP-1-112822 MW-12-S5-112822 MW-12-S4-112822** EQP-1-112822	NMeFOSA NEtFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P	Internal standards (%R)
MW-4-S4-112822 MW-4-S2-112822	NMeFOSAA	UJ (all non-detects)	P	Internal standards (%R)
MW-15-113022	NEtFOSA	UJ (all non-detects)	P	Internal standards (%R)
MW-24-S4-112922 MW-24-S3-112922** MW-17-S5-112922 MW-17-S4-112922 DUP-3-112922 SB-2-112922 MW-12-S5-112822 MW-12-S4-112822** EQP-2-112922	All analytes flagged "IR1" or IR2" by the laboratory due to ion ratio outside QC limits.	J (all detects)	P	Target analyte identification (ion ratio)

NASA JPL
Perfluoroalkyl & Polyfluoroalkyl Substances - Laboratory Blank Data Qualification Summary - SDG 22L004

Sample	Analyte	Modified Final Concentration	A or P
EQP-1-112822	PFOA	0.23J ng/L	A

Sample	Analyte	Modified Final Concentration	A or P
EQP-2-112922	PFOS	0.18U ng/L	A
EQP-3-113022	PFOA	0.21J ng/L	A

NASA JPL
Perfluoroalkyl & Polyfluoroalkyl Substances - Field Blank Data Qualification Summary - SDG 22L004

Sample	Analyte	Modified Final Concentration	A or P
DUP-1-112822	PFOA	0.94J ng/L	A
MW-24-S5-112922	PFOA	0.21J ng/L	A
MW-24-S4-112922	PFOA	0.26J ng/L	A
MW-24-S2-112922	PFOA	0.68J ng/L	A
DUP-2-112922	PFOA	0.83J ng/L	A
MW-17-S2-112922	PFOA	0.23J ng/L	A
MW-4-S5-112822	PFOA PFOS	0.27J ng/L 0.14J ng/L	A
MW-4-S4-112822	PFOA PFOS	0.40J ng/L 0.15J ng/L	A
MW-4-S2-112822	PFOS	0.20J ng/L	A

LDC #: 56164A96 **VALIDATION COMPLETENESS WORKSHEET** Date: 4/5/23
SDG #: 22L004 Level III/IV Page: 1 of 2
Laboratory: APPL, Inc., Clovis, CA Reviewer: [illegible]
2nd Reviewer: [illegible]

METHOD: LC/MS Perfluoroalkyl & Polyfluoroalkyl Substances (QSM 5.3 Table B-15)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A / A	
II.	LC/MS Instrument performance check	A	
III.	Initial calibration/ICV	A / A	RSD≤20 %RCV≤30
IV.	Continuing calibration/ISC	A/SW	D≤30
V.	Laboratory Blanks	SW	
V.I	Field blanks	SW	SB=11, 20, 26* FB*=21, 24, 28 EB=22, 27, 29
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	LCSD, MRL check
IX.	Field duplicates	SW	D= 4+5, 8+9, 12+13
X.	Internal Standards	SW	EIS
XI.	Target analyte quantitation	A	Not reviewed for Level III validation.
XII.	Target analyte identification	SW	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	

Note: A = Acceptable; N = Not provided/applicable; SW = See worksheet; *ND = No compounds detected; R = Rinsate; FB = Field blank; D = Duplicate; TB = Trip blank; EB = Equipment blank; SB=Source blank; OTHER:

** Indicates sample underwent Level IV validation

	Client ID	Lab ID	Matrix	Date
1	MW-24-S5-112922	22L0004-01	Water	11/29/22
2	MW-24-S4-112922	22L0004-02	Water	11/29/22
3	MW-24-S3-112922**	22L0004-03**	Water	11/29/22
4	MW-24-S2-112922	22L0004-04	Water	11/29/22
5	DUP-2-112922	22L0004-05	Water	11/29/22
6	MW-17-S5-112922	22L0004-06	Water	11/29/22
7	MW-17-S4-112922	22L0004-07	Water	11/29/22
8	MW-17-S3-112922**	22L0004-08**	Water	11/29/22
9	DUP-3-112922	22L0004-09	Water	11/29/22
10	MW-17-S2-112922	22L0004-10	Water	11/29/22
11	SB-2-112922	22L0004-11	Water	11/29/22
12	MW-4-S5-112822	22L0004-12	Water	11/28/22
13	DUP-1-112822	22L0004-13	Water	11/28/22
14	MW-4-S4-112822	22L0004-14	Water	11/28/22
15	MW-4-S2-112822	22L0004-15	Water	11/28/22
16	MW-12-S5-112822	22L0004-16	Water	11/28/22

LDC #: 56164A96 **VALIDATION COMPLETENESS WORKSHEET** Date: 4/3/23
SDG #: 22L004 Level III/IV Page: 2 of 2
Laboratory: APPL, Inc., Clovis, CA Reviewer:
2nd Reviewer:

METHOD: LC/MS Perfluoroalkyl & Polyfluoroalkyl Substances (QSM 5.3 Table B-15)

	Client ID	Lab ID	Matrix	Date
17	MW-12-S4-112822**	22L0004-17**	Water	11/28/22
18	MW-12-S3-112822	22L0004-18	Water	11/28/22
19	MW-12-S2-112822	22L0004-19	Water	11/28/22
20	SB-1-112822	22L0004-20	Water	11/28/22
21 2	FB-1-112822	22L0004-21	Water	11/28/22
22 3	EQP-1-112822	22L0004-22	Water	11/28/22
23 3	EQP-2-112922	22L0004-23	Water	11/29/22
24 2	FB-2-112922	22L0004-24	Water	11/29/22
25 3	MW-15-113022	22L0004-25	Water	11/30/22
26 4	SB-3-113022	22L0004-26	Water	11/30/22
27 3	EQP-3-113022	22L0004-27	Water	11/30/22
28 2	FB-3-113022	22L0004-28	Water	11/30/22
29	MW-4-S2-112822MS	22L0004-15MS	Water	11/28/22
30	MW-4-S2-112822MSD	22L0004-15MSD	Water	11/28/22
31				
32				
33				

Notes:

1	BBL0076						
2	BBL0249						
3	BBL0296						
4	BBL0371						

LDC #: 58164196

VALIDATION FINDINGS CHECKLIST

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?	√			
Were cooler temperature criteria met?	√			
II. LC/MS Instrument performance check				
Were the instrument performance reviewed and found to be within the validation criteria?	√			
III. Initial calibration and Initial calibration verification				
Did the laboratory perform a 5-point calibration prior to sample analysis?	√			
Were all percent relative standard deviations (%RSD) ≤ 20%?	√			
Was a curve fit used for evaluation? If yes, did the initial calibration meet the coefficient of determination (r^2) criteria of ≥ 0.990?			√	
Were all analytes within 70-130% or percent differences (%D) ≤30% of their true value for each calibration standard?	√			
Was the signal to noise (S/N) ratio for all analytes within the validation criteria?	√			
Were the retention time windows properly established?	√			
Was an initial calibration verification (ICV) standard analyzed after each initial calibration for each instrument?	√			
Were all ICV percent differences (%D) of the initial calibration verification ≤ 30%?	√			
IV. Continuing calibration and Instrument sensitivity check				
Was a continuing calibration analyzed prior to sample analysis, after every 10 samples and at the end of the analytical sequence?	√			
Were all percent differences (%D) of the continuing calibration ≤ 30%?	√			
Were all the retention times within the acceptance windows?	√			
Was the signal to noise (S/N) ratio for all analytes within the validation criteria?	√			
Were all percent differences (%D) of the Instrument Sensitivity Check ≤ 30%?		√		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	√			
Was there contamination in the laboratory blanks?	√			
VI. Field blanks				
Were field blanks identified in this SDG?	√			
Were target analytes detected in the field blanks?	√			
VII. Matrix spike/Matrix spike duplicates				
Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG?	√			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		√		

Validation Area	Yes	No	NA	Findings/Comments
VIII. Laboratory control samples				
Was an LCS analyzed per extraction batch for this SDG?	√			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		√		
IX. Field duplicates				
Were field duplicate pairs identified in this SDG?	√			
Were target analytes detected in the field duplicates?	√			
X. Internal Standard				
Were extracted internal standard percent recoveries (%R) within the QC limits?		√		
Were retention times within 0.4 minutes of the associated calibration standard?	√			
XI. Target analyte quantitation				
Did the laboratory reporting limits (i.e. DL, LOD, LOQ) meet the QAPP?	√			
Did reported results include both branched and linear isomers?	√			
Were the correct ion transition, labeled compound and relative response factor (RRF) used to quantitate the analyte?	√			
Were analyte retention times within 0.1 minutes of the associated labeled compound for analytes with a labeled analog?	√			
Were analyte quantitation and reporting limits adjusted to reflect all sample dilutions and dry weight factors applicable to Stage 4 validation?	√			
XII. Target analyte identification				
Was the signal to noise (S/N) ratio for all analytes within the validation criteria?	√			
Were two transitions and the ion transition ratio per analyte monitored and documented with the exception of PFBA and PFPeA?	√			
Were ion ratios between 50-150%?		√		
Were manual integrations performed and found acceptable?	√			
Did the laboratory provide before and after printouts?		√		
XIII. Overall assessment of Data				
Overall assessment of data was found to be acceptable.	√			

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Continuing Calibration/Instrument Sensitivity Check

Reviewer: SC

Method: LC/MS PFAS (EPA Method 537M)

Continuing calibrations were analyzed prior to sample analysis, after every 10 samples and at the end of the analytical sequence. Instrument sensitivity ch were analyzed prior to sample analysis, and every 12 hours. Percent differences (%D) were ≤30 % with the following exception:

#	Date	Standard ID	Analyte	Finding %D Limit: ≤30.0%)	Associated Samples	Qualifications
	12/9/2022	SB03769-LCV1	6:2FTS	31.0	1-20 (det/ND)	J+/UJ/A
		(ISC)	NEtFOSE	40.0		
	12/15/2022	SB03858-LCV1	PFUnA	37.2	22,23,25,27 (ND)	
		(ISC)				
	12/15/2022	SB03860-LCV1	PFDA	93.7	21,24,28 (ND)	J+/UJ/A
		(ISC)	HFPO-DA	36.8		J+/UJ/A
			PFDOA	33.9		J+/UJ/A

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Blanks

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DOD QSM 5.3

Blank extraction date: 12/13/22 **Associated samples:** 22,23,25,27

Analyte	Blank ID (ng/L)	Sample Identification							
	BBL0296-BLK1	5X	22	23	27				
PFOA	0.194	0.97	0.23/J		0.21/J				
PFOS	0.276	1.38		0.11/0.18U					

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Field Blanks

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Sampling date: 11/29/22 **Associated samples:** 1-10

Analyte	Blank ID (ng/L)	Sample Identification						
	11 (SB)	5X	1	2	4	5	10	
PFOA	0.19	0.95	0.21/J	0.26/J	0.68/J	0.83/J	0.23/J	
PFOS	0.059	0.30						

Sampling date: 11/28/22 **Associated samples:** 12-19

Analyte	Blank ID (ng/L)	Sample Identification						
	20 (SB)	5X	12	14	15			
PFOA	0.17	0.85	0.27/J	0.40/J				
PFOS	0.12	0.60	0.14/J	0.15/J	0.20/J			

Sampling date: 11/29/22 **Associated samples:** 1-10

Analyte	Blank ID (ng/L)	Sample Identification						
	23 (EB)	5X	1,2,4,5,10					
PFOS	0.11	0.55	see above					

Sampling date: 11/28/22 **Associated samples:** 12-19

Analyte	Blank ID (ng/L)	Sample Identification						
	22 (EB)	5X	12,14,15	13				
PFOA	0.23	1.15	see above	0.94/J				

Sampling date: 11/30/22 **Associated samples:** 25

Analyte	Blank ID (ng/L)	Sample Identification						
	27 (EB)	5X						
PFOS	0.21	1.05						

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample.
Percent recoveries (%R) and %RPD were within QC limits with the exceptions identified below.

#	MS/MSD ID	Analyte	MS %S	MSD %R	%R Limits	RPD (≤30)	Associated Samples	Qualifications
	29/30	PFNS	42.2	66.8	69-127		15 (ND)	J/UJ/A
		PFNS				44.1		Jdets/A
		PFTRDA				34.1		Jdets/A

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

LCS and LCS Duplicate percent recoveries (%R) and %RPD were within QC limits with the exceptions identified below.

#	LCS/LCSD ID MRL ID*	Analyte	LCS %R	LCSD %R	%R Limits	RPD (≤30)	Associated Samples	Qualifications
	BBL0076-BS1/BSD1	PFDOA				35.0	1-20 (ND)	J/UJ/P
	BBL0249-BS1/BSD1	PFNA	140		69-130		21,24,28 (ND)	Jdets/P
		PFUnA	134		69-133			
		8:2FTS	140		67.138			
		NMeFOSAA	153		65-136			
	BBL0296-BS1/BSD1	PFUnA				37.3	22,23,25,27 (ND)	Jdets/P
	BBL0371-BS1/BSD1	PFTEDA				36.2	26 (ND)	Jdets/P

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

MRL percent recoveries (%R) were within QC limits with the exceptions identified below.

#	LCS/LCSD ID MRL ID*	Analyte	MRL %R	%R Limits	RPD (≤30)	Associated Samples	Qualifications
	BBL0076-MRL1	8:2FTS	64.9	67-138		1-20 (ND)	J/UJ/P
		PFOSA	142	67-137		1-20 (1-3,7,8 -det)	Jdets/P
	BBL0249-MRL1	PFDOA	137	72-134		21,24,28 (ND)	Jdets/P
		PFOSA	200	67-137			↓
	BBL0296-MRL1	PFUnA	135	69-133		22,23,25,27 (ND)	Jdets/P
		PFDOA	148	72-134			↓
		PFTEDA	175	71-132			↓
		PFNS	133	69-127			↓
		PFDS	150	53-142			↓
		NMeFOSAA	211	65-136			↓
		NMeFOSE	165	70-130			↓
		11CL-PF3OUDS	139	54-138			↓
	BBL0371-MRL1	PFTEDA	62.1	71-132		26 (ND)	J/UJ/P
		NMeFOSE	64.0	70-130			↓
		NEtFOSE	131	70-130			Jdets/P

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Analyte	Concentration (ng/L)		RPD
	4	5	
PFBA	1.4	1.3	7
PFPEA	2.3	2.4	4
PFHXA	1.5	1.3	14
PFHPA	0.70	0.70	0
PFOA	0.68	0.83	20
PFBS	0.84	0.65	26
PFPES	0.62	0.66	6
PFHXS	2.3	2.2	4
PFOS	0.33	0.55	50

Analyte	Concentration (ng/L)		RPD
	8	9	
PFBA	1.0	0.80	22
PFPEA	1.8	1.9	5
PFHXA	1.2	1.4	15
PFHPA	0.68	0.92	30
PFOA	2.3	2.0	14
PFNA	0.37	0.53	36
PFDA	1.4	0.91	42
PFBS	2.2	2.4	9
PFPES	0.56	0.53	6
PFHXS	1.4	1.4	0
PFOS	6.6	5.9	11
PFOSA	0.13	0.18U	32

Analyte	Concentration (ng/L)		RPD
	12	13	
PFBA	0.36	0.61	52
PFPEA	0.72	1.0	33
PFHXA	0.48	0.97	68
PFHPA	0.28	0.41	38
PFOA	0.27	0.94	111
PFDA	0.19U	0.17	11
PFBS	0.79	4.9	144
PFPES	0.40	0.29	32
PFHXS	0.42	0.44	5
PFOS	0.14	0.18U	25
NEtFOSAA	0.19U	0.21	10

VALIDATION FINDINGS WORKSHEET
Internal Standards

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Extracted internal standard percent recoveries (%R) were within QC limits with the exceptions identified below.

#	Sample ID	Internal Standards	%R (50-150)	Qualifications (see pg 3)
ND	MW-24-S5-112922	D3-NMEFOSA	35.3	J/UJ/P
		D5-NETFOSA	37.8	
		D7-NMEFOSE	46.3	
	MW-24-S4-112922	D3-NMEFOSA	42.4	J/UJ/P
		D5-NETFOSA	43.8	
	MW-24-S3-112922	D3-NMEFOSA	37.3	J/UJ/P
		D5-NETFOSA	38.6	
		D7-NMEFOSE	42.1	
		D9-NETFOSE	46.7	
	MW-24-S2-112922	D3-NMEFOSA	41.6	J/UJ/P
		D5-NETFOSA	39.3	
		D7-NMEFOSE	47.3	
	DUP-2-112922	D3-NMEFOSA	47.0	J/UJ/P
		D5-NETFOSA	45.1	
	MW-17-S5-112922	D3-NMEFOSA	41.8	J/UJ/P
		D5-NETFOSA	42.2	
	MW-17-S4-112922	D3-NMEFOSA	42.0	J/UJ/P
		D5-NETFOSA	37.9	
		D7-NMEFOSE	45.7	
		D9-NETFOSE	47.9	
	MW-17-S3-112922	D3-NMEFOSA	44.0	J/UJ/P
		D5-NETFOSA	44.0	
		D3-NMEFOSAA	168	Jdets/P

VALIDATION FINDINGS WORKSHEET
Internal Standards

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Extracted internal standard percent recoveries (%R) were within QC limits with the exceptions identified below.

#	Sample ID	Internal Standards	%R (50-150)	Qualifications (see pg 3)
ND	DUP-3-112922	D3-NMEFOSA	39.5	J/UJ/P
		D5-NETFOSA	39.6	
		D7-NMEFOSE	43.3	
		D9-NETFOSE	44.3	
		D3-NMEFOSAA	191	Jdets/P
	SB-2-112922	D3-NMEFOSA	32.1	J/UJ/P
		D5-NETFOSA	39.0	
	MW-4-S5-112822	D3-NMEFOSA	29.4	J/UJ/P
		D5-NETFOSA	40.7	
		D7-NMEFOSE	47.1	
	DUP-1-112822	D3-NMEFOSA	26.7	J/UJ/P
		D5-NETFOSA	39.3	
		D7-NMEFOSE	44.6	
		D9-NETFOSE	48.9	
	MW-4-S4-112822	D3-NMEFOSA	41.7	J/UJ/P
	MW-4-S2-112822	D3-NMEFOSA	41.4	J/UJ/P
	MW-12-S5-112822	13C2-8:2FTS	180	Jdets/P
		D3-NMEFOSA	43.2	J/UJ/P
		D5-NETFOSA	46.3	
		D7-NMEFOSE	47.4	
		D9-NETFOSE	45.1	
	MW-12-S4-112822	D3-NMEFOSA	39.8	J/UJ/P
		D5-NETFOSA	41.0	
		D7-NMEFOSE	42.6	
		D9-NETFOSE	43.9	

VALIDATION FINDINGS WORKSHEET
Internal Standards

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Extracted internal standard percent recoveries (%R) were within QC limits with the exceptions identified below.

#	Sample ID	Internal Standards	%R (50-150)	Qualifications
Ael ND	MW-12-S3-112822	D3-NMEFOSA	43.3	J/UJ/P
		D5-NETFOSA	45.1	
		D7-NMEFOSE	46.3	
	MW-12-S2-112822	D3-NMEFOSA	47.5	J/UJ/P
		D5-NETFOSA	49.8	
	SB-1-112822	D3-NMEFOSA	31.7	J/UJ/P
		D5-NETFOSA	31.4	
	EQP-1-112822	D3-NMEFOSA	42.4	J/UJ/P
		D5-NETFOSA	35.6	
		D7-NMEFOSE	42.4	
		D9-NETFOSE	41.2	
	MW-15-113022	D5-NETFOSA	48.3	J/UJ/P
	FB-3-113022	D3-NMEFOSA	43.7	J/UJ/P
		D5-NETFOSA	39.7	
			Association:	
			13C2-8:2FTS	8:2FTS
			D3-NMEFOSA	NMeFOSA
			D3-NMEFOSAA	NMeFOSAA
			D5-NETFOSA	NEtFOSA
			D7-NMEFOSE	NMeFOSE
			D9-NETFOSE	NEtFOSE

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Target Analyte Identification

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

#	Sample ID	Analyte	Finding	Qualifications
	2,3,6,7,9,11,16,17,23		All analytes flagged "IR1" or "IR2" by the laboratory due to	Jdets/P
			ion ratio outside QC limits.	

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

The Relative Response Factor (RRF), average RRF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following calculations:

$RRF = (A_x)(C_{is})/(A_{is})(C_x)$
average RRF = sum of the RRFs/number of standards
%RSD = 100 * (S/X)

A_x = Area of Compound
C_x = Concentration of compound
S = Standard deviation of the RRFs

A_{is} = Area of associated internal standard
C_{is} = Concentration of internal standard
X = Mean of the RRFs

#	Standard ID	Calibration Date	Compound	Reported RRF (RRF 1 std)	Recalculated RRF (RRF 1 std)	Reported Average RRF	Recalculated Average RRF	Reported %RSD	Recalculated %RSD
1	ICAL	12/7/2022	PFOA (13C8-PFOA)	Not reported	0.55665	0.51542	0.51542	6.2	6.2
			PFOS (13C8-PFOS)	↓	0.55754	0.52064	0.52064	6.2	6.2
2			PFOA (13C8-PFOA)						
			PFOS (13C8-PFOS)						
3			PFOA (13C8-PFOA)						
			PFOS (13C8-PFOS)						
4									

VALIDATION FINDINGS WORKSHEET
Continuing Calibration Calculation Verification

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
RRF = (Ax)(Cis)/(Ais)(Cx)

Where:
ave. RRF = initial calibration average RRF
RRF = continuing calibration RRF
Ax = Area of compound
Cx = Concentration of compound,
Ais = Area of associated internal standard
Cis = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (IS)	Conc	Reported Conc	Recalculated Conc	Reported %R	Recalculated %R
1	SB03769-CCV2	12/10/2022	PFOA (13C8-PFOA)	5.000	4.56	4.56	91.2	91.2
			PFOS (13C8-PFOS)	4.650	4.84	4.84	104.0	104.0
2			PFOA (13C8-PFOA)					
			PFOS (13C8-PFOS)					
3			PFOA (13C8-PFOA)					
			PFOS (13C8-PFOS)					
4								
5								

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

SSC = (Area spike) (Conc IS) / (Area IS) (average RRF spike)
%Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked concentration, SA = Spike added, SC = Sample concentration

RPD = | MS - MSD | * 2/(MS + MSD)

MS = Matrix spike recovery, MSD = Matrix spike duplicate recovery

MS/MSD ID: 29/30

Compound	SA (ng/L)		SC (ng/L)	SSC (ng/L)		MS Percent Recovery		MSD Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
PFOA	7.25	7.17	1.07	6.55	7.35	75.7	75.6	87.6	87.6	11.5	11.5
PFOS	6.74	6.67	0.197	6.71	6.07	96.6	96.6	88.0	88.1	10.1	10.0

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
LCS Results Verification

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample (LCS) and laboratory control duplicate (LCSD) were recalculated for the compounds identified below using the following calculation:

SSC = (Area spike) (Conc IS) / (Area IS) (average RRF spike)
%Recovery = 100 * SSC/SA

Where:
SSC = Spiked concentration
SA = Spike added
LCS = Laboratory control spike recovery
LCSD = Laboratory control spike duplicate recovery

RPD = | LCS - LCSD | * 2/(LCS + LCSD)

LCS/LCSD ID: BBL0076-BS1/BSD1

Compound	SA (ng/L)		SSC (ng/L)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
PFOA	8.00	8.00	7.52	7.95	94.0	94.0	99.3	99.4	5.51	5.56
PFOS	7.44	7.44	6.57	7.24	88.3	88.3	97.3	97.3	9.72	9.70

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Sample Results Verification

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Compound results for all Level IV samples reported with a positive detect were recalculated and verified using the following equation:

$$\text{Concentration} = \frac{(Ax)\,(Cis)\,(Vt)\,(DF)}{(Ais)\,(RRF)\,(Vo)}$$

Where:

Ax = Area or height of the peak for the compound to be measured
Ais = Area or height of the peak for the internal standard
Cis = Concentration of the internal standard
DF = Dilution factor
Vt = Volume of extract in milliters (mL)
RRF = Average relative response factor
Wt = Weight of sample in grams (g)
%S = Percent solids

Sample #	Compound	Ax	Ais	Cis (ng/mL)	DF	RRF	Vt (mL)	Vo (mL)	%S	Calculated Concentration (ng/L)	Reported Concentration (ng/L)	% Diff
3	PFOA	49628	280867	1	1	0.51542	2	293.05		2.3	2.3	

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name: NASA JPL

LDC Report Date: April 5, 2023

Parameters: Perfluoroalkyl & Polyfluoroalkyl Substances

Validation Level: Level III & IV

Laboratory: APPL, Inc., Clovis, CA

Sample Delivery Group (SDG): 22L005

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SB-1-0.5-113022	22L0005-01	Soil	11/30/22
SB-1-2.0-113022	22L0005-02	Soil	11/30/22
SB-2-0.5-113022	22L0005-03	Soil	11/30/22
SB-2-2.0-113022**	22L0005-04**	Soil	11/30/22
SB-3-0.5-113022	22L0005-05	Soil	11/30/22
SB-3-2.0-113022	22L0005-06	Soil	11/30/22
SB-4-0.5-113022	22L0005-07	Soil	11/30/22
SB-4-2.0-113022	22L0005-08	Soil	11/30/22
SB-5-0.5-113022	22L0005-09	Soil	11/30/22
SB-5-2.0-113022	22L0005-10	Soil	11/30/22
DUP-1-113022	22L0005-11	Soil	11/30/22
SB-6-0.5-113022**	22L0005-12**	Soil	11/30/22
DUP-2-113022	22L0005-13	Soil	11/30/22
SB-6-2.0-113022	22L0005-14	Soil	11/30/22
Field Blank	22L0005-15	Water	11/30/22
EQP-1-SOIL	22L0005-16	Water	11/30/22
SB-6-2.0-113022MS	22L0005-14MS	Soil	11/30/22
SB-6-2.0-113022MSD	22L0005-14MSD	Soil	11/30/22

**Indicates sample underwent Level IV validation

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with a modified outline of the USEPA Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFAS) Analyzed Using EPA Method 537 (November 2018). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

All sample results were subjected to Level III data validation, which comprises an evaluation of quality control (QC) summary results. Samples appended with a double asterisk on the cover page were subjected to Level IV data validation, which is comprised of the QC summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

J (Estimated): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.

U (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).

UJ (Non-detected estimated): The analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.

R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.

NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

II. LC/MS Instrument Performance Check

Instrument performance was checked and the requirements were met.

III. Initial Calibration and Initial Calibration Verification

Initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes.

For each calibration standard, all analytes were within 70-130% of their true value.

The signal to noise (S/N) ratio was within validation criteria for all analytes associated to samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

Retention time windows were established as required by the method for samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all analytes.

IV. Continuing Calibration and Instrument Sensitivity Check

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 30.0% for all analytes.

The signal to noise (S/N) ratio was within validation criteria for all analytes associated to samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

The percent differences (%D) of the instrument sensitivity check (ISC) were less than or equal to 30.0% for all analytes with the following exceptions:

Date	Analyte	%D	Associated Samples	Flag	A or P
12/09/22	PFDA PFUnA PFOS 8:2FTS	35.1 30.7 36.3 38.7	SB-1-0.5-113022 SB-1-2.0-113022 SB-2-0.5-113022 SB-2-2.0-113022** SB-3-0.5-113022 SB-3-2.0-113022 SB-4-0.5-113022 SB-4-2.0-113022 SB-5-0.5-113022 SB-5-2.0-113022 DUP-1-113022 SB-6-0.5-113022** DUP-2-113022 SB-6-2.0-113022	J (all detects) UJ (all non-detects)	A
12/15/22	PFDA HFPO-DA PFDOA	93.7 36.8 33.9	Field Blank	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	A

Retention times of all analytes in the calibration standards were within the established retention time windows for samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

All analyte concentrations were at the limit of quantitation (LOQ) for the ISC standard.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

Sample EQP-1-SOIL was identified as an equipment blank. No contaminants were found.

Sample Field Blank was identified as a field blank. No contaminants were found.

VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
SB-6-2.0-113022MS/MSD (SB-6-2.0-113022)	PFNS	168 (69-127)	-	J (all detects)	A

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
SB-6-2.0-113022MS/MSD (SB-6-2.0-113022)	9CL-PF3ONS	135 (70-130)	133 (70-130)	NA	-

For SB-6-2.0-113022MS/MSD, no data were qualified for PFOS percent recoveries outside the QC limits since the parent sample results were greater than 4X the spike concentration.

Relative percent differences (RPD) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
SB-6-2.0-113022MS/MSD (SB-6-2.0-113022)	PFNS	35.9 (≤30)	J (all detects)	A

VIII. Laboratory Control Samples/Minimal Risk Levels

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Analyte	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
BBL0249-BS1/BSD1 (Field Blank)	PFNA PFUnA 8:2FTS NMeFOSAA	140 (69-130) 134 (69-133) 140 (67-138) 153 (65-136)	- - - -	NA	-

Relative percent differences (RPD) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
BBL0371-BS1/BSD1 (EQP-1-SOIL)	PFTEDA	36.2 (≤30)	NA	-

Minimal risk levels (MRL) percent recoveries were within QC limits with the following exceptions:

MRL ID (Associated Samples)	Analyte	%R (Limits)	Flag	A or P
BBL0032-MRL1 (SB-1-0.5-113022 SB-1-2.0-113022 SB-2-0.5-113022 SB-2-2.0-113022** SB-3-0.5-113022 SB-3-2.0-113022 SB-4-0.5-113022 SB-4-2.0-113022 SB-5-0.5-113022 SB-5-2.0-113022 DUP-1-113022 SB-6-0.5-113022** DUP-2-113022 SB-6-2.0-113022)	PFHPS	68.7 (70-132)	UJ (all non-detects)	P
BBL0249-MRL1 (Field Blank)	PFDOA PFOSA	137 (72-134) 200 (67-137)	NA	-
BBL0371-MRL1 (EQP-1-SOIL)	PFTEDA NMeFOSE	62.1 (71-132) 64.0 (70-130)	UJ (all non-detects) UJ (all non-detects)	P
BBL0371-MRL1 (EQP-1-SOIL)	NEtFOSE	131 (70-130)	NA	-

IX. Field Duplicates

Samples SB-5-2.0-113022 and DUP-1-113022 and samples SB-6-0.5-113022** and DUP-2-113022 were identified as field duplicates. No results were detected in any of the samples with the following exceptions:

Analyte	Concentration (ng/L)		RPD
	SB-6-0.5-113022**	DUP-2-113022	
PFOA	0.49U	0.28	55
PFNA	0.49U	0.19	88
PFDA	0.27	0.38	34
PFUnA	0.17	0.25	38
PFDOA	0.20	0.47U	81
PFTRDA	0.12	0.47U	119
PFHXS	0.90	1.5	50
PFOS	22	48	74

Analyte	Concentration (ng/L)		RPD
	SB-6-0.5-113022**	DUP-2-113022	
PFNS	2.0	5.3	90
PFDS	1.5	2.5	50
PFOSA	0.49U	0.15	106

X. Internal Standards

All extracted internal standard percent recoveries (%R) were within QC limits with the following exceptions:

Sample	Internal Standard	%R (Limits)	Affected Analyte	Flag	A or P
SB-1-0.5-113022	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE	44.5 (50-150) 45.6 (50-150) 47.6 (50-150)	NMeFOSA NEtFOSA NMeFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
SB-1-2.0-113022	D3-NMEFOSA D7-NMEFOSE D9-NETFOSE	49.2 (50-150) 47.5 (50-150) 48.2 (50-150)	NMeFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
SB-6-0.5-113022**	D3-NMEFOSA D5-NETFOSA D7-NMEFOSE	40.7 (50-150) 41.8 (50-150) 47.8 (50-150)	NMeFOSA NEtFOSA NMeFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P
DUP-2-113022	13C8-PFOSA D3-NMEFOSA D5-NETFOSA D7-NMEFOSE D9-NETFOSE	47.8 (50-150) 30.8 (50-150) 31.7 (50-150) 42.1 (50-150) 45.3 (50-150)	PFOSA NMeFOSA NEtFOSA NMeFOSE NEtFOSE	J (all detects) UJ (all non-detects)	P
EQP-1-SOIL	D5-NETFOSA	47.5 (50-150)	NEtFOSA	UJ (all non-detects)	P

XI. Target Analyte Quantitation

All target analyte quantitations met validation criteria for samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

XII. Target Analyte Identification

All target analyte identifications met validation criteria for samples which underwent Level IV validation. Raw data were not reviewed for Level III validation.

All target analyte ion ratios met validation criteria with the following exceptions:

Sample	Analyte	Ion Ratio (Limits)	Flag	A or P
SB-2-2.0-113022**	PFDA 6:2FTS	157.3 (50-150) 184.6 (50-150)	J (all detects) J (all detects)	P
SB-6-0.5-113022**	PFUnA PFTRDA PFDS	22.5 (50-150) 48.5 (50-150) 42.2 (50-150)	J (all detects) J (all detects) J (all detects)	P

XIII. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to ISC %D, MS/MSD %R and RPD, MRL %R, internal standard %R, and ion ratio, data were qualified as estimated in seventeen samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable.

NASA JPL
Perfluoroalkyl & Polyfluoroalkyl Substances - Data Qualification Summary - SDG 22L005

Sample	Analyte	Flag	A or P	Reason
SB-1-0.5-113022 SB-1-2.0-113022 SB-2-0.5-113022 SB-2-2.0-113022** SB-3-0.5-113022 SB-3-2.0-113022 SB-4-0.5-113022 SB-4-2.0-113022 SB-5-0.5-113022 SB-5-2.0-113022 DUP-1-113022 SB-6-0.5-113022** DUP-2-113022 SB-6-2.0-113022	PFDA PFUnA PFOS 8:2FTS	J (all detects) UJ (all non-detects)	A	Continuing calibration (ISC %D)
Field Blank	PFDA HFPO-DA PFDOA	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	A	Continuing calibration (ISC %D)
SB-6-2.0-113022	PFNS	J (all detects)	A	Matrix spike/Matrix spike duplicate (%R)
SB-6-2.0-113022	PFNS	J (all detects)	A	Matrix spike/Matrix spike duplicate (RPD)
SB-1-0.5-113022 SB-1-2.0-113022 SB-2-0.5-113022 SB-2-2.0-113022** SB-3-0.5-113022 SB-3-2.0-113022 SB-4-0.5-113022 SB-4-2.0-113022 SB-5-0.5-113022 SB-5-2.0-113022 DUP-1-113022 SB-6-0.5-113022** DUP-2-113022 SB-6-2.0-113022	PFHPS	UJ (all non-detects)	P	Minimal risk levels (%R)
EQP-1-SOIL	PFTEDA NMeFOSE	UJ (all non-detects) UJ (all non-detects)	P	Minimal risk levels (%R)
SB-1-0.5-113022 SB-6-0.5-113022**	NMeFOSA NEtFOSA NMeFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P	Internal standards (%R)
SB-1-2.0-113022	NMeFOSA NMeFOSE NEtFOSE	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	P	Internal standards (%R)

Sample	Analyte	Flag	A or P	Reason
DUP-2-113022	PFOSA NMeFOSA NEtFOSA NMeFOSE NEtFOSE	J (all detects) UJ (all non-detects)	P	Internal standards (%R)
EQP-1-SOIL	NEtFOSA	UJ (all non-detects)	P	Internal standards (%R)
SB-2-2.0-113022**	PFDA 6:2FTS	J (all detects) J (all detects)	P	Target analyte identification (ion ratio)
SB-6-0.5-113022**	PFUnA PFTRDA PFDS	J (all detects) J (all detects) J (all detects)	P	Target analyte identification (ion ratio)

NASA JPL
Perfluoroalkyl & Polyfluoroalkyl Substances - Laboratory Blank Data Qualification Summary - SDG 22L005

No Sample Data Qualified in this SDG

NASA JPL
Perfluoroalkyl & Polyfluoroalkyl Substances - Field Blank Data Qualification Summary - SDG 22L005

No Sample Data Qualified in this SDG

LDC #: 56164B96 **VALIDATION COMPLETENESS WORKSHEET** Date: 4/2/23
SDG #: 22L005 Level III/IV Page: 1 of 2
Laboratory: APPL, Inc., Clovis, CA Reviewer: [illegible]
2nd Reviewer: [illegible]

METHOD: LC/MS Perfluoroalkyl & Polyfluoroalkyl Substances (QSM 5.3 Table B-15)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A / A	
II.	LC/MS Instrument performance check	A	
III.	Initial calibration/ICV	A / A	RSD≤20 TV/IV≤30
IV.	Continuing calibration/ISC	A/SW	≤30
V.	Laboratory Blanks	A	
V.I	Field blanks	ND	FB=15 EB=16
VII.	Matrix spike/Matrix spike duplicates	SW	
VIII.	Laboratory control samples	SW	LCS/D, MRL check
IX.	Field duplicates	SW	D= 10+11*, 12+13
X.	Internal Standards	SW	EIS
XI.	Target analyte quantitation	A	Not reviewed for Level III validation.
XII.	Target analyte identification	SW	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	

Note: A = Acceptable; N = Not provided/applicable; SW = See worksheet; * ND = No compounds detected; R = Rinsate; FB = Field blank; D = Duplicate; TB = Trip blank; EB = Equipment blank; SB=Source blank; OTHER:

** Indicates sample underwent Level IV validation

	Client ID	Lab ID	Matrix	Date
1	SB-1-0.5-113022	22L0005-01	Soil	11/30/22
2	SB-1-2.0-113022	22L0005-02	Soil	11/30/22
3	SB-2-0.5-113022	22L0005-03	Soil	11/30/22
4	SB-2-2.0-113022**	22L0005-04**	Soil	11/30/22
5	SB-3-0.5-113022	22L0005-05	Soil	11/30/22
6	SB-3-2.0-113022	22L0005-06	Soil	11/30/22
7	SB-4-0.5-113022	22L0005-07	Soil	11/30/22
8	SB-4-2.0-113022	22L0005-08	Soil	11/30/22
9	SB-5-0.5-113022	22L0005-09	Soil	11/30/22
10	SB-5-2.0-113022	22L0005-10	Soil	11/30/22
11	DUP-1-113022	22L0005-11	Soil	11/30/22
12	SB-6-0.5-113022**	22L0005-12**	Soil	11/30/22
13	DUP-2-113022	22L0005-13	Soil	11/30/22
14	SB-6-2.0-113022	22L0005-14	Soil	11/30/22
15 2	Field Blank	22L0005-15	Water	11/30/22
16 3	EQP-1-SOIL	22L0005-16	Water	11/30/22

LDC #: 56164B96 **VALIDATION COMPLETENESS WORKSHEET** Date: 4/3/23
SDG #: 22L005 Level III/IV Page: 2 of 2
Laboratory: APPL, Inc., Clovis, CA Reviewer: [illegible]
2nd Reviewer: [illegible]

METHOD: LC/MS Perfluoroalkyl & Polyfluoroalkyl Substances (QSM 5.3 Table B-15)

	Client ID	Lab ID	Matrix	Date
17	SB-6-2.0-113022MS	22L0005-14MS	Soil	11/30/22
18	SB-6-2.0-113022MSD	22L0005-14MSD	Soil	11/30/22
19				
20				
21				

Notes:

1	BBL0032						
2	BBLD249						
3	BBL0371						

LDC #: 56164B96

VALIDATION FINDINGS CHECKLIST

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?	√			
Were cooler temperature criteria met?	√			
II. LC/MS Instrument performance check				
Were the instrument performance reviewed and found to be within the validation criteria?	√			
III. Initial calibration and Initial calibration verification				
Did the laboratory perform a 5-point calibration prior to sample analysis?	√			
Were all percent relative standard deviations (%RSD) ≤ 20%?	√			
Was a curve fit used for evaluation? If yes, did the initial calibration meet the coefficient of determination (r^2) criteria of ≥ 0.990?			√	
Were all analytes within 70-130% or percent differences (%D) ≤30% of their true value for each calibration standard?	√			
Was the signal to noise (S/N) ratio for all analytes within the validation criteria?	√			
Were the retention time windows properly established?	√			
Was an initial calibration verification (ICV) standard analyzed after each initial calibration for each instrument?	√			
Were all ICV percent differences (%D) of the initial calibration verification ≤ 30%?	√			
IV. Continuing calibration and Instrument sensitivity check				
Was a continuing calibration analyzed prior to sample analysis, after every 10 samples and at the end of the analytical sequence?	√			
Were all percent differences (%D) of the continuing calibration ≤ 30%?	√			
Were all the retention times within the acceptance windows?	√			
Was the signal to noise (S/N) ratio for all analytes within the validation criteria?	√			
Were all percent differences (%D) of the Instrument Sensitivity Check ≤ 30%?		√		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	√			
Was there contamination in the laboratory blanks?		√		
VI. Field blanks				
Were field blanks identified in this SDG?	√			
Were target analytes detected in the field blanks?		√		
VII. Matrix spike/Matrix spike duplicates				
Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG?	√			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		√		

Validation Area	Yes	No	NA	Findings/Comments
VIII. Laboratory control samples				
Was an LCS analyzed per extraction batch for this SDG?	√			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		√		
IX. Field duplicates				
Were field duplicate pairs identified in this SDG?	√			
Were target analytes detected in the field duplicates?	√			
X. Internal Standard				
Were extracted internal standard percent recoveries (%R) within the QC limits?		√		
Were retention times within 0.4 minutes of the associated calibration standard?	√			
XI. Target analyte quantitation				
Did the laboratory reporting limits (i.e. DL, LOD, LOQ) meet the QAPP?	√			
Did reported results include both branched and linear isomers?	√			
Were the correct ion transition, labeled compound and relative response factor (RRF) used to quantitate the analyte?	√			
Were analyte retention times within 0.1 minutes of the associated labeled compound for analytes with a labeled analog?	√			
Were analyte quantitation and reporting limits adjusted to reflect all sample dilutions and dry weight factors applicable to Stage 4 validation?	√			
XII. Target analyte identification				
Was the signal to noise (S/N) ratio for all analytes within the validation criteria?	√			
Were two transitions and the ion transition ratio per analyte monitored and documented with the exception of PFBA and PFPeA?	√			
Were ion ratios between 50-150%?		√		
Were manual integrations performed and found acceptable?	√			
Did the laboratory provide before and after printouts?		√		
XIII. Overall assessment of Data				
Overall assessment of data was found to be acceptable.	√			

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
Continuing Calibration/Instrument Sensitivity Check

Reviewer: SC

Method: LC/MS PFAS (EPA Method 537M)

Continuing calibrations were analyzed prior to sample analysis, after every 10 samples and at the end of the analytical sequence. Instrument sensitivity ch were analyzed prior to sample analysis, and every 12 hours. Percent differences (%D) were ≤30 % with the following exception:

#	Date	Standard ID	Analyte	Finding %D Limit: ≤30.0%)	Associated Samples	Qualifications
	12/9/2022	SB03754-LCV1	PFDA	35.1	1-14 (det/ND)	J+/UJ/A
		(ISC)	PFUnA	30.7		
			PFOS	36.3		
			8:2FTS	38.7		
	12/15/2022	SB03860-LCV1	PFDA	93.7	15 (ND)	J+/UJ/A
		(ISC)	HFPO-DA	36.8		J+/UJ/A
			PFDOA	33.9		J+/UJ/A

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample.
Percent recoveries (%R) and %RPD were within QC limits with the exceptions identified below.

#	MS/MSD ID	Analyte	MS %S	MSD %R	%R Limits	RPD (≤30)	Associated Samples	Qualifications
	17/18	PFNS	168		69-127		14 (det)	Jdets/A
		9CL-PF3ONS	135	133	70-130		(ND)	Jdets/A
		PFOS	%Rs out					NQ. >4X
		PFNS				35.9		Jdets/A

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

LCS and LCS Duplicate percent recoveries (%R) and %RPD were within QC limits with the exceptions identified below.

#	LCS/LCSD ID MRL ID*	Analyte	LCS %R	LCSD %R	%R Limits	RPD (≤30)	Associated Samples	Qualifications
	BBL0249-BS1/BSD1	PFNA	140		69-130		15 (ND)	Jdets/P
		PFUnA	134		69-133			↑
		8:2FTS	140		67.138			↕
		NMeFOSAA	153		65-136			↓
	BBL0371-BS1/BSD1	PFTEDA				36.2	16 (ND)	Jdets/P

LDC #: 56164A96

VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

MRL percent recoveries (%R) were within QC limits with the exceptions identified below.

#	LCS/LCSD ID MRL ID*	Analyte	MRL %R	%R Limits	RPD (≤30)	Associated Samples	Qualifications
	BBL0032-MRL1	PFHPS	68.7	70-132		1-14 (ND)	Jdets/P
	BBL0249-MRL1	PFDOA	137	72-134		15 (ND)	Jdets/P
		PFOSA	200	67-137			↓
	BBL0371-MRL1	PFTEDA	62.1	71-132		16 (ND)	J/UJ/P
		NMeFOSE	64.0	70-130			↓
		NEtFOSE	131	70-130			Jdets/P

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Analyte	Concentration (ng/L)		RPD
	12	13	
PFOA	0.49U	0.28	55
PFNA	0.49U	0.19	88
PFDA	0.27	0.38	34
PFUnA	0.17	0.25	38
PFDOA	0.20	0.47U	81
PFTRDA	0.12	0.47U	119
PFHXS	0.90	1.5	50
PFOS	22	48	74
PFNS	2.0	5.3	90
PFDS	1.5	2.5	50
PFOSA	0.49U	0.15	106

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Extracted internal standard percent recoveries (%R) were within QC limits with the exceptions identified below.

#	Sample ID	Internal Standards	%R (50-150)	Qualifications (see pg 3)
ND	SB-1-0.5-113022	D3-NMEFOSA	44.5	J/UJ/P
		D5-NETFOSA	45.6	
		D7-NMEFOSE	47.6	
ND	SB-1-2.0-113022	D3-NMEFOSA	49.2	J/UJ/P
		D7-NMEFOSE	47.5	
		D9-NETFOSE	48.2	
ND	SB-6-0.5-113022	D3-NMEFOSA	40.7	J/UJ/P
		D5-NETFOSA	41.8	
		D7-NMEFOSE	48.7	
det/ND	DUP-2-113022	13C8-PFOSA	47.8	J/UJ/P
		D3-NMEFOSA	30.8	
		D5-NETFOSA	31.7	
		D7-NMEFOSE	42.1	
		D9-NETFOSE	45.3	
ND	EQP-1-SOIL	D5-NETFOSA	47.5	J/UJ/P
			Association:	
			13C8-PFOSA	PFOSA
			D3-NMEFOSA	NMeFOSA
			D5-NETFOSA	NEtFOSA
			D7-NMEFOSE	NMeFOSE
			D9-NETFOSE	NEtFOSE

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
Target Analyte Identification

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

#	Sample ID	Analyte	Ion ratio (50-150%)	Qualifications
	4	PFDA	157.3	Jdets/P
		6:2FTS	184.6	
	12	PFUnA	22.5	Jdets/P
		PFTRDA	48.5	
		PFDS	42.2	
		For other samples with IRs out, the lab did not flag the reports, they indicated that it is possible this report predates inclusion of IR flags.		

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

The Relative Response Factor (RRF), average RRF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following calculations:

$RRF = (A_x)(C_{is})/(A_{is})(C_x)$
average RRF = sum of the RRFs/number of standards
%RSD = 100 * (S/X)

A_x = Area of Compound
C_x = Concentration of compound
S = Standard deviation of the RRFs

A_{is} = Area of associated internal standard
C_{is} = Concentration of internal standard
X = Mean of the RRFs

#	Standard ID	Calibration Date	Compound	Reported RRF (RRF 1 std)	Recalculated RRF (RRF 1 std)	Reported Average RRF	Recalculated Average RRF	Reported %RSD	Recalculated %RSD
1	ICAL	12/7/2022	PFOA (13C8-PFOA)	Not reported	0.55665	0.51542	0.51542	6.2	6.2
			PFOS (13C8-PFOS)		0.55754	0.52064	0.52064	6.2	6.2
2			PFOA (13C8-PFOA)						
			PFOS (13C8-PFOS)						
3			PFOA (13C8-PFOA)						
			PFOS (13C8-PFOS)						
4									

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
Continuing Calibration Calculation Verification

Reviewer: SC

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
RRF = (Ax)(Cis)/(Ais)(Cx)

Where:
ave. RRF = initial calibration average RRF
RRF = continuing calibration RRF
Ax = Area of compound
Cx = Concentration of compound,
Ais = Area of associated internal standard
Cis = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (IS)	Conc	Reported Conc	Recalculated Conc	Reported %R	Recalculated %R
1	SB03754-CCV2	12/9/2022	PFOA (13C8-PFOA)	5.000	4.81	4.81	96.2	96.2
			PFOS (13C8-PFOS)	4.650	4.49	4.49	96.5	96.5
2			PFOA (13C8-PFOA)					
			PFOS (13C8-PFOS)					
3			PFOA (13C8-PFOA)					
			PFOS (13C8-PFOS)					
4								
5								

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

SSC = (Area spike) (Conc IS) / (Area IS) (average RRF spike)
%Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked concentration
SA = Spike added
SC = Sample concentration
MS = Matrix spike recovery
MSD = Matrix spike duplicate recovery

RPD = | MS - MSD | * 2/(MS + MSD)

MS/MSD ID: 17/18

Compound	SA (ng/g)		SC (ng/g)	SSC (ng/g)		MS Percent Recovery		MSD Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
PFOA	2.18	2.14	0.165	2.40	2.24	103.0	102.5	96.9	97.0	7.25	6.90
PFOS	2.03	1.99	125	155	118	1510	1505	-367	-366	27.7	27.7

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
LCS Results Verification

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample (LCS) and laboratory control duplicate (LCSD) were recalculated for the compounds identified below using the following calculation:

SSC = (Area spike) (Conc IS) / (Area IS) (average RRF spike)
%Recovery = 100 * SSC/SA

Where:
SSC = Spiked concentration
SA = Spike added
LCS = Laboratory control spike recovery
LCSD = Laboratory control spike duplicate recovery

RPD = | LCS - LCSD | * 2/(LCS + LCSD)

LCS/LCSD ID: BBL0032-BS1/BSD1

Compound	SA (ng/g)		SSC (ng/g)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
PFOA	1.89	1.96	2.00	1.93	106	106	98.6	98.5	3.62	3.56
PFOS	1.75	1.82	1.95	1.83	111	111	100	101	6.29	6.35

LDC #: 56164B96

VALIDATION FINDINGS WORKSHEET
Sample Results Verification

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3

Compound results for all Level IV samples reported with a positive detect were recalculated and verified using the following equation:

$$\text{Concentration} = \frac{(Ax)\,(Cis)\,(Vt)\,(DF)}{(Ais)\,(RRF)\,(Wt)\,(\%S)}$$

Where:

Ax = Area or height of the peak for the compound to be measured
Ais = Area or height of the peak for the internal standard
Cis = Concentration of the internal standard
DF = Dilution factor
Vt = Volume of extract in milliters (mL)
RRF = Average relative response factor
Wt = Weight of sample in grams (g)
%S = Percent solids

Sample #	Compound	Ax	Ais	Cis (ng/mL)	DF	RRF	Vt (mL)	Wt (g)	%S	Calculated Concentration (ng/g)	Reported Concentration (ng/g)	% Diff
12	PFOS	4318596	877924	0.9275	1	0.52064	2	1.02	0.7939	21.6	22.0	