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Perfluorinated Alkyl Substance (PFAS) Analyte Testing and Additional Analytical Evaluation of Relevant Firefighting Foam Formulations and Samples

DR. KATHERINE M. HINNANT

Navy Technology Center for Safety and Survivability Section
Chemistry Division

JANICE L. WILLEY

Naval Sea Systems Command Oversight and Assessment Division
Goose Creek, S.C.

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Dr. Katherine M. Hinnant and Janice L. Willey*

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In an effort to better understand the potential toxicity of new fluorine-free firefighting foams, a series of commercial laboratory analysis was performed to quantify potentially harmful chemical components. Analysis was conducted on 7 foam concentrates: 4 commercial-off-the-shelf (COTS) self-identified fluorine-free concentrates, 2 experimental fluorine-free foam concentrates made in a laboratory setting by a manufacturer and the U.S. Naval Research Laboratory, and one COTS fluorinated aqueous film forming foam (AFFF) for comparison to the other materials. Laboratory analysis included PFAS by DoD/DOE QSM Table B-15 Compliant Method, Extractable Organic Fluorine (EOF), EPA Method 8270D, EPA Method 300/SW-846 Method 9056A, EPA Method 6010C, EPA Method 7470, GC/MS, and UPLC-QTOF-MS.

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Perfluorinated Alkyl Substance (PFAS) Analyte Testing and Additional Analytical Evaluation of Relevant Firefighting Foam Formulations and Samples

1.0 INTRODUCTION

This report summarizes the collection, sampling, and analysis of fluorine-free firefighting foam samples provided through the Strategic Environmental Research and Development Plan (SERDP) Program ER21-3503. Under 2020 National Defense Authorization Act (NDAA), fluorinated aqueous film-forming foam may not be used at any military installation on or after October 1, 2024. Alternatives were assessed for individual PFAS components, extractable organic fluorine (EOF), various ions and metals, mercury, through GC/MS and UPLC-QTOF-MS. This comprehensive test series was conducted to assess the potential environmental impact of alternative fluorine-free firefighting foams (F3 foams).

2.0 APPROACH AND METHOLODY

2.1 Materials

2.1.1 Commercial F3 Concentrates. Four commercial fluorine-free concentrates were purchased and evaluated. These four products were National Foam AVIO Green KHC 3%, Fomtec ENVIRO 2-3% FFF, Solberg Re-healing Foam RF3 3%, and Bio-Ex ECOPOL A 3% FFF. Product names have been excluded from the tabulated data and each foam assigned a random number. Each product was purchased as a 5 gallon drum of concentrate, these containers were shipped to the Naval Research Laboratory (NRL) detachment at Chesapeake Beach, Maryland (CBD). National Foam, Fomtec, and Bio-Ex drums all had manufacturing dates of 2020. The Solberg drum had a manufacturing date of 2018. At CBD, drums were opened and samples extracted using 1cm diameter glass drum thieves. One liter samples were extracted into fresh HDPE plastic bottles. The bottles were then sent to NRL DC for further sampling. Sampling at NRL DC was conducted June 2021.

2.1.2 Experimental F3 Concentrates. Two concentrates analyzed were from experimental formulations developed by a manufacturer and NRL. A 1 L sample bottle was shipped from the manufacturer to NRL DC, it listed a manufacture date of 3/22/2021 and was opened 06/01/2021. Commercial components were mixed into a 1 L quantity of the NRL 502W Formulation the day of sampling, 06/01/2021. Components were mixed in a fresh HDPE plastic bottle. These two formulations have also been assigned a random number in the following data.

2.1.3 Commercial AFFF Concentrate. An AFFF on the qualified products list was also evaluated as a comparison. A 5 gallon drum of this product was also purchased and sampled into a 1 L fresh HDPE bottle at NRL CBD. The bottle was then sent to NRL DC for further sampling. This product represents a C6 fluorinated product and had a manufacture date of 2019. As this sample intentionally contains a fluorinated surfactant, it has been assigned the number 7 in the tabulated results.

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2.2 Laboratory Testing through SGS AXYS

Six replacement formulations underwent various tests to determine their chemical composition using various standardized and non-standardized procedures, detailed in Table 1. Each sample was prepared and analyzed in triplicate for each test. Analysis was completed on a sample, a second collected sample (a duplicate), and a retest of the duplicate. In addition to these six formulations, the fluorinated concentrate, sample 7, was also prepared and analyzed three times for the determination of 40 PFAS by a method compliant with the requirements of the DoD/DOE QSM Table B-15 and extractable organic fluorine (EOF). Samples were sent to various laboratories and the laboratories that performed each test are noted in Table 1.

Table 1. Formulations Report Key		
Method	**Analytes**	**Laboratory**
PFAS by DoD/DOE QSM Table B-15 Compliant Method	40 PFAS	SGS AXYS Canada
Extractable Organic Fluorine (EOF)	Extractable Organic Fluorine	SGS AXYS Antwerp
EPA Method 8270D	Semi-volatiles (targeted and non-targeted)	SGS Orlando
EPA Method 300/SW-846 Method 9056A	chloride, fluoride, nitrate, sulfate	SGS Orlando
EPA Method 6010C	aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium, selenium, silver, sodium, thallium, vanadium, zinc	SGS Orlando
EPA Method 7470A	mercury	SGS Orlando
GC/MS	Non-volatile and semi-volatile compounds	SGS AXYS Antwerp
UPLC-QTOF-MS	Non-targeted PFAS	SGS AXYS Antwerp

Samples were also submitted to SGS Orlando for analysis by EPA SW-846 Method 8260D, however, due to the excessive dilution required to prevent foaming, results of this test would render no useful information and the test was cancelled.

SGS AXYS provided appropriate, clean sample bottles for each test series along with a blank sent with the bottles for analysis. Due to the viscosity of the concentrates, sample was poured directly from the 1 L HDPE bottle into the sampling containers. 40 mL VOA vials were filled for volatiles analysis. The same samples were used for multiple test methods. 60 mL HDPE bottles were supplied for ion, metals, and mercury testing. The same samples were used for multiple test methods. 60 mL HDPE bottles were supplied for the PFAS analysis. 500 mL HDPE bottles were supplied for testing at SGS AXYS Antwerp. These samples were used for multiple test methods.

3.0 RESULTS AND DISCUSSION

A summary of the compounds identified in each formulation is provided in Table 3. Details of each test are provided in subsequent sections of this report.

Table 2. Positively Identified Compounds		
Analyte	**Concentration**	**Units**
Sample 1		
Hexylene Glycol[1]	1000 JN, 16,667 JN	µg/L, mg/kg
Cyclododecane	51 JN	µg/L
15-Crown-5	29 JN	µg/L
3,6,9,12,15-Pentaoxanonadecan-1-ol	35 JN	µg/L
1-Decene	63 JN	µg/L
1,3,3-Trimethoxybutane	43 JN	µg/L
3,6,9,12-Tetraoxahexadecan-1-ol	54 JN	µg/L
Propanoic acid, 3-methoxy-, methyl ester	29 JN	µg/L
Chloride	3.0	mg/L
Copper	2.6 J	µg/L
Potassium	225 J	µg/L
Selenium	2.9 J	µg/L
Sodium	3933 J	µg/L
1-Dodecanol	3,267 J	mg/kg
Methylamine, N-(1-methylheptylidene)-	617 J	mg/kg
1-Tetradecanol	1,967 J	mg/kg
Ethanol, 2-(dodecyloxy)-	957 J	mg/kg
Ethanol, 2-(tetradecyloxy)-	503 J	mg/kg
Diethylene glycol monododecyl ether	847 J	mg/kg
Triethylene glycol monododecyl ether	587 J	mg/kg
Tetraethylene glycol monododecyl ether	337 J	mg/kg
Sample 2		
15-Crown-5	230 JN	µg/L
Ethanol, 2-(2-butoxyethoxy)-	200 JN	µg/L
Ethanol, 2-[2-(2-butoxyethoxy)ethoxy]-	97 JN	µg/L
Octane, 1,1'-oxybis-	27 JN	µg/L
1-o-Octyl-.beta.-D-glucopynosid	55 JN	µg/L
Pyrazolo[1,5-a]pyridine, 3,7-dimethyl-2-	36 JN	µg/L
Hexanoic acid	770 JN	µg/L
1,4,7,10,13,16-Hexaoxacyclooctadecane	220 JN	µg/L
Bacchotricuneatin	27 JN	µg/L
3,6,9,12,15,18,21-Heptaoxabicyclo[21.3.1]	30 JN	µg/L
1,4,7,10,13,16-Hexaoxanonadecane, 18-(2-	190 JN	µg/L
Propanoic acid, 2-(methoxymethoxy)-	37 JN	µg/L
Fluoride	0.073 J	mg/L
Copper	10.3 J	µg/L

Table 2. Positively Identified Compounds		
Analyte	**Concentration**	**Units**
Zinc	7.9 J	µg/L
Ethanol, 1-(2-butoxyethoxy)-	74,667 J	mg/kg
Ethanol, 2-butoxy-	290 J	mg/kg
Butane, 1,1'-[oxybis(2,1-ethanediyloxy)]bis-	310 J	mg/kg
Octyl-.beta.-D-glucopyranoside	2,333 J	mg/kg
Sample 3		
Benzyl Alcohol	21.1 J	µg/L
N-Nitrosodi-n-propylamine	164	µg/L
Cyclododecane	1900 JN	µg/L
1,4,7,10,13,16-Hexaoxacyclooctadecane	190 JN	µg/L
Silane, (dodecyloxy)trimethyl-	320 JN	µg/L
Cyclotetradecane	2600 JN	µg/L
1-Dodecanamine, N,N-dimethyl-	1700 JN	µg/L
Tridecanoic acid, tert-butyldimethylsily	1300 JN	µg/L
1,1,3-Trimethyl-1-silacyclobutane	170 JN	µg/L
Cyclohexadecane	490 JN	µg/L
1-Pentadecanamine, N,N-dimethyl-	260 JN	µg/L
Silane, (hexadecyloxy)trimethyl-	640 JN	µg/L
1-Tridecanamine, N,N-dimethyl-	1400 JN	µg/L
12-Methylaminolauric acid	100 JN	µg/L
6,6-Dimethyl-9-methylene-undecane-2,5,10	180 JN	µg/L
N,N-Dimethyloctylamine	190 JN	µg/L
N,N-Dimethyltetradecanamine	1500 JN	µg/L
10-Methyl-E-11-tridece-1-ol acetate	130 JN	µg/L
1R,3-cis-Diethoxy-5-cis-methylcyclohexan	140 JN	µg/L
Tetradecyltrimethylammonium bromide	240 JN	µg/L
Estra-1,3,5,7,9-pentaen-17-one, 3-methox	90 JN	µg/L
p-Butoxybenzylidene p-propylaniline	78 JN	µg/L
Nitrate	0.049 J	mg/L
Sulfate	0.91 J	mg/L
Calcium	189 J	µg/L
Copper	5.8 J	µg/L
Nickel	0.40 J	µg/L
Selenium	3.95 J	µg/L
Sodium	10,190	µg/L
Zinc	22.9	µg/L
Ethanol, 1-(2-butoxyethoxy)-	57,667 J	mg/kg
1-Tetradecene	5,067 J	mg/kg
1-Dodecanamine, N,N-dimethyl-	7,833 J	mg/kg
1-Tetradecanol	16,000 J	mg/kg
1-Tetradecanamine, N,N-dimethyl-	4,900 J	mg/kg
1-Hexadecanamine, N,N-dimethyl-	600 J	mg/kg
1-Decene	287 J	mg/kg

Table 2. Positively Identified Compounds		
Analyte	**Concentration**	**Units**
Diisopropyl ether	247 J	mg/kg
7-Tetradecenal	9,600 J	mg/kg
Didodecyldimethylammonium bromide	79 J	mg/kg
Sample 4		
Cyclododecane	370 JN	µg/L
1-Decene	160 JN	µg/L
Ethanol, 2-(2-butoxyethoxy)-	710 JN	µg/L
1-Dodecanamine, N,N-dimethyl-	210 JN	µg/L
Pentadecane	130 JN	µg/L
2-Dodecanol	71 JN	µg/L
Pentaethylene glycol	67 JN	µg/L
1,4,7,10,13,16-Hexaoxanonadecane,	47 JN	µg/L
Perfluoronaphthoquinone	48 JN	µg/L
12-Crown-4	53 JN	µg/L
Cyclopropane, nonyl-	310 JN	µg/L
1-Dodecanamine, N,N-dimethyl-	270 JN	µg/L
Decane, 3,8-dimethyl-	120 JN	µg/L
2-Hexadecanol	57 JN	µg/L
Dodecane, 1-(methoxymethoxy)-	65 JN	µg/L
Pentaethylene glycol	57 JN	µg/L
1,4-Anthracenedione, 2-hydroxy-5-m	29 JN	µg/L
Perfluoronaphthoquinone	55 JN	µg/L
1,4,7,10,13,16-Hexaoxacyclooctadecane	70 JN	µg/L
7-Hexadecene, (Z)-	320 JN	µg/L
l-Guanidinosuccinimide	120 JN	µg/L
1,4,7,10,13,16-Hexaoxanonadecane, 18-pro	69 JN	µg/L
Pentaethylene glycol	74 JN	µg/L
Perfluoronaphthoquinone	54 JN	µg/L
Nitrate	0.051 J	mg/L
Sulfate	0.81 J	mg/L
Calcium	79 J	µg/L
Potassium	489 J	µg/L
Selenium	3.7 J	µg/L
Mercury	0.11 J	µg/L
Ethanol, 1-(2-butoxyethoxy)-	24,000 J	mg/kg
1-Dodecanol	10,300 J	mg/kg
Furfural or isomer	237 J	mg/kg
2,8,9-Trioxa-5-aza-1-silabicyclo[3.3.3]undecane, 1-methyl-	103 J	mg/kg
Dodecanal	297 J	mg/kg
1-Tetradecanol	5,433 J	mg/kg
Ethanol, 2-(dodecyloxy)-	1,073 J	mg/kg
Diethylene glycol monododecyl ether	713 J	mg/kg
Sample 5		

Table 2. Positively Identified Compounds		
Analyte	**Concentration**	**Units**
Cyclododecane	1600 JN	µg/L
Ethanol, 2-(2-butoxyethoxy)-	200 JN	µg/L
Cyclotetradecane	1000 JN	µg/L
1-Dodecanamine, N,N-dimethyl-	690 JN	µg/L
Ethanol, 2-butoxy-[1]	2200 JN, 1,967 JN	µg/L, mg/kg
Cyclooctane, 1,2-dimethyl-	490 JN	µg/L
2-Isobutylthiazole	930 JN	µg/L
Thiazole, 5-methyl-	660 JN	µg/L
3,5-Diamino-1,2,4-triazole	390 JN	µg/L
Acetone, 1-[4-(dimethylaminoethoxy)pheny	200 JN	µg/L
Triethyl phosphate	450 JN	µg/L
Glycocyanidine	150 JN	µg/L
Hexanoic acid, anhydride	120 JN	µg/L
1,4-Dioxaspiro[4.5]decan-8-ol	140 JN	µg/L
Chloride	3.8	mg/L
Fluoride	0.28	mg/L
Nitrate	0.053 J	mg/L
Calcium	86 J	µg/L
Copper	1.3 J	µg/L
Iron	57.5 J	µg/L
Selenium	3.3 J	µg/L
Sodium	5473 J	µg/L
Ethanol, 1-(2-butoxyethoxy)-	27,667 J	µg/L
Ethanol, 2-butoxy-	1,967 J	mg/kg
1-Dodecanol	9,467 J	mg/kg
1-Tetradecanol	3,633 J	mg/kg
1-Octanol	167 J	mg/kg
1-Decanol	367 J	mg/kg
Octane, 1,1'-oxybis-	130 J	mg/kg
2,8,9-Trioxa-5-aza-1-silabicyclo(3.3.3)undecane, 1-methoxy-	600 J	mg/kg
2,8,9-Trioxa-5-aza-1-silabicyclo[3.3.3]undecane, 1-methyl-	320 J	mg/kg
Dodecanal	473 J	mg/kg
1-Dodecanamine, N,N-dimethyl-	1,287 J	mg/kg
1-Tetradecanamine, N,N-dimethyl-	1,000 J	mg/kg
1-Hexadecanol	623 J	mg/kg
Sample 6		
PFOS	10.25 J	µg/L
Cyclododecane	86 JN	µg/L
1-Decene	88 JN	µg/L
Ethanol, 2-(2-butoxyethoxy)-	200 JN	µg/L
Ethanol, 2-butoxy-	34 JN	µg/L
Cyclooctane, 1,2-dimethyl-	55 JN	µg/L

Table 2. Positively Identified Compounds		
Analyte	**Concentration**	**Units**
2-Isobutylthiazole	110 JN	µg/L
Triethyl phosphate	100 JN	µg/L
Hexanoic acid, anhydride	31 JN	µg/L
1-Tetradecene	53 JN	µg/L
Trifluoroacetic acid, n-tetradecyl	42 JN	µg/L
2-Butanone, 4-(dimethylamino)-3-me	30 JN	µg/L
Chloride	2.8	mg/L
Fluoride	0.078 J	mg/L
Nitrate	0.052 J	mg/L
Calcium	184 J	µg/L
Copper	2.6 J	µg/L
Iron	19.2 J	µg/L
Magnesium	54.3 J	µg/L
Manganese	17.7	µg/L
Nickel	0.40 J	µg/L
Potassium	402 J	µg/L
Sodium	3396 J	µg/L
Ethanol, 1-(2-butoxyethoxy)-	31,667 J	mg/kg
1-Dodecanol	3,600 J	mg/kg
Furfural or isomer	413 J	mg/kg
5-Hydroxymethylfurfural	2,133 J	mg/kg
1-Decanol	317 J	mg/kg
1-Undecanol	270 J	mg/kg
Ethanol, 2-(dodecyloxy)- or isomer	160 J	mg/kg
1-Tetradecanol	1,567 J	mg/kg
Diethylene glycol monododecyl ether or isomer	163 J	mg/kg
1-Hexadecanol	377 J	mg/kg
Triethylene glycol monododecyl ether or isomer	140 J	mg/kg
Tetraethylene glycol monododecyl ether or isomer	74 J	mg/kg

1 = Compound was tentatively identified via both EPA SW-846 Method 8270D and a GC/MS screening method. In these cases, the concentration estimated using EPA SW-846 Method 8270D is listed first.

J = indicates concentration is an estimation (qualitative)

N = Tentatively identified compound; Estimated concentration

3.1 Summarized Results for PFAS Analysis by SGS AXYS Canada

3.1.1 PFAS Compounds Identified in Analysis. In accordance with their DoD ELAP-accredited PFAS procedures, SGS AXYS Method MLA-110, SGS AXYS Canada prepared and analyzed

samples of 6 fluorine-free firefighting foam concentrates and one fluorinated concentrate in triplicate for the determination of 40 PFAS (Table 3).

Table 3. Names, Abbreviations, and CAS Registry Numbers for the Method Analytes		
Target Analyte Name	**Abbreviation**	**CAS Number**
Perfluoroalkyl carboxylic acids		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluoroalkyl sulfonic acids		
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentansulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
Fluorotelomer sulfonic acids		
1*H*,1*H*, 2*H*, 2*H*-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4
1*H*,1*H*, 2*H*, 2*H*-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2
1*H*,1*H*, 2*H*, 2*H*-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4
Perfluorooctane sulfonamides		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2
Perfluorooctane sulfonamidoacetic acids		
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
Perfluorooctane sulfonamide ethanols		
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2

Table 3. Names, Abbreviations, and CAS Registry Numbers for the Method Analytes		
Target Analyte Name	**Abbreviation**	**CAS Number**
Per- and Polyfluoroether carboxylic acids		
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
4,8-Dioxa-3*H*-perfluorononanoic acid	ADONA	919005-14-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Ether sulfonic acids		
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7
Fluorotelomer carboxylic acids		
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5
2*H*,2*H*,3*H*,3*H*-Perfluorooctanoic acid	5:3FTCA	914637-49-3
3-Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4

3.1.2 QC Sample Results. The preparation batch included a two method blanks (MB), two laboratory control samples (LCS, or OPR), a matrix spike (MS), and a matrix spike duplicate (MSD). Preparation batch QC samples were evaluated against the criteria in SGS AXYS Method MLA-110. The method blank (MB) was evaluated against the acceptance criteria of no PFAS at concentrations greater than ½ the limit of quantitation (LOQ). No PFAS were detected in either MB and all extracted internal standard (EIS) compound recoveries met their acceptance criteria. The LCSs met the spike recovery acceptance criteria for all PFAS and EIS compound spike recovery acceptance criteria with the exception of the LCS associated with HFPO-DA. That LCS had an HFPO-DA spike recovery of 56%, which failed to meet the 70-130% acceptance criteria. Because HFPO-DA was not detected in any of the associated samples and the recovery of the isotopically labeled analog of HFPO-DA was within the acceptance criteria, HFPO-DA is considered a non-detect without any additional uncertainty applied to it. These results are listed as "U^3" in Table 4. The matrix spike (MS) and matrix spike duplicate (MSD) that were prepared by spiking an aliquot of Sample 1, met all the spike recovery and MS/MSD relative percent difference acceptance criteria for all PFAS and EIS compounds with the exception of NFDHA which had a recovery of 161%, which failed to meet the 65-140% acceptance criteria. Since NFDHA was not detected in Sample 1, this exceedance had no impact on the sample result for Sample 1. This result is listed as "U^4" in Table 4.

3.1.3 Concentrate Results. The results of the 7 concentrates are presented in Table 4. If an analyte was not detected in a sample, the result was listed as a non-detect with a "U". In instances where the sample results for an analyte were at a concentration between the detection limit and the limit of quantitation (LOQ), the average of the three sample results was reported

with the standard deviation, a "J" flag (indicating this is an estimated value), and highlighted green. In instances where the analyte concentration was above the LOQ, the average of three sample results was reported along with the standard deviation and highlighted blue.

The recovery of the EIS compounds spiked into each sample was evaluated to assess the bias associated with the sample preparation procedure. In instances where the recovery of an associated EIS of an analyte exceeded the high end of its recovery acceptance criteria range and the analyte was not detected, the exceedance had no impact on the analyte's quantitation. These failures are identified with as "U[1]" in Table 4. In instances where the recovery of an associated EIS of an analyte exceeded the high end of its recovery acceptance criteria range and the analyte was detected in the sample, the concentration reported is negatively biased. These results are highlighted in pink and have a "1" superscript associated with the average of the triplicate sample results.

In instances where the recovery of an associated EIS of an analyte fell below the low end of its recovery acceptance criteria range, but above 20%, and the analyte was not detected, the exceedance causes the non-detect status of the analyte to be interpreted as an estimate (qualitative determination). These instances are identified as "U[2]". In one instance, the associated EIS of an 8:2FTS did not recover at all and 8:2FTS was not detected in the sample, this data point cannot be used either qualitatively or quantitatively. This was identified with a "U[3]" and is highlighted in yellow.

Table 4. PFAS Results*

Analyte	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7
PFBA	U	U	U	U	U	U	1100 (50)
PFPeA	U	U	U	U	U	U	291 (21)
PFHxA	U	U	U	U	U	U	2670 (382)
PFHpA	U	U	U	U	U	U	51.3[1] (4.5)
PFOA	U	U	U	U	U	U	U
PFNA	U	U	U	U	U	U	U
PFDA	U	U	U	U	U	U	U
PFUnA	U	U	U	U	U	U	U
PFDoA	U	U	U	U[2]	U	U	U
PFTrDA	U	U	U	U	U	U	U
PFTeDA	U	U	U	U[2]	U	U	U
PFBS	U	U	U	U	U	U	U[1]
PFPeS	U	U	U	U	U	U	U
PFHxS	U	U	U	U	U	U	U
PFHpS	U	U	U	U	U	U	U
PFOS	U	U	U	U	U	10.25 J (0.73)	U
PFNS	U	U	U	U	U	U	U
PFDS	U	U	U	U	U	U	U
PFDoS	U	U	U	U	U	U	U

4:2 FTS	U	U	U	U	U	U[1]	347[1] (32)
6:2 FTS	U	U	U	U	U	U	91,900[1] (5450)
8:2 FTS	U	U[1]	U[2]	U[3]	U	U	U[1]
PFOSA	U	U	U	U	U	U	U
N-MeFOSA	U	U	U	U	U	U	U
N-EtFOSA	U	U	U	U	U	U	U
MeFOSAA	U	U	U	U	U	U[1]	U[1]
EtFOSAA	U	U	U	U	U	U[1]	U[1]
N-MeFOSE	U	U	U	U	U	U	U
N-EtFOSE	U	U	U	U	U	U	U
HFPO-DA	U	U	U	U	U	U	U
ADONA	U[4]	U[4]	U[4]	U	U[4]	U[4]	U[4]
9Cl-PF3ONS	U	U	U	U	U	U	U
11Cl-PF3OUdS	U	U	U	U	U	U	U
3:3 FTCA	U	U	U	U	U	U	U
5:3 FTCA	U	U	U	U	U	U	U
7:3 FTCA	U	U	U	U	U	U	U
PFEESA	U	U	U	U	U	U	U
PFMPA	U	U	U	U	U	U	U
PFMBA	U	U	U	U	U	U	U
NFDHA	U[5]	U	U	U	U	U	U

*Sample results are reported as averages in ng/g (ppb) followed by the standard deviation provided in parentheses

[1] Associated EIS compound recovery exceeded the high limit of the acceptance range

[2] Associated EIS compound recovery fell below the lower limit of the acceptance range

[3] Associated EIS compound did not recover (0% recovery)

[4] Analyte recovery fell below the low end of the acceptance range in associated OPR

[5]Analyte recovery exceeded the high end of the acceptance range in associated MSD

3.2 Summarized Results for EOF Analyzed by SGS AXYS Antwerp

In accordance with their in-house standard operating procedure (SOP), SGS AXYS Antwerp prepared and analyzed the seven concentrates for total extractable organic fluorine (EOF) content using combustion ion chromatography (CIC)/ion chromatography(IC). Each formulation was prepared and analyzed in triplicate using a 1 gram aliquot of each formulation.

3.2.1 QC Sample Results. No information was provided on QC samples associated with these samples.

3.2.2 Concentrate Results. The reporting limit of detection for EOF associated with each sample is 10 mg/kg. Fluorine was not detected in the six F3 concentrates. Sample 7 reported an average of 4500 mg/kg EOF, with a standard deviation of the triplicates of 11.5.

3.3 Summarized Results for EPA SW-846 Method 8270D Analyzed by AXYS Orlando

3.3.1 Compounds Identified in Analysis. SGS AXYS Orlando prepared and analyzed six F3 concentrates using their DoD ELAP accredited procedure for EPA SW-846 Method 8270D for the semi-volatiles compounds listed in Table 5. Each sample received was prepared and analyzed in triplicate using a 1:1000 dilution of the sample received.

Table 5. SW-846 EPA Method 8270D Analyte List	
Benzoic Acid	1,2-Dichlorobenzene
4-Chloro-3-methyl Phenol	1,3-Dichlorobenzene
2-Chlorophenol	1,4-Dichlorobenzene
2,4-Dichlorophenol	3,3'-Dichlorobenzidine
2,4-Dimethylphenol	Diethyl Phthalate
2,4-Dinitrophenol	Dimethyl Phthalate
4,6-Dinitro-o-cresol	Di-n-butyl Phthalate
2-Methylphenol	Dibenzo(a,h)anthracene
3&4-Methylphenol	Dibenzofuran
2-Nitrophenol	Di-n-octyl Phthalate
4-Nitrophenol	2,4-Dinitrotoluene
Pentachlorophenol	2,6-Dinitrotoluene
Phenol	1,2-Diphenylhydrazine
2,4,5-Trichlorophenol	bis(2-Ethylhexyl)phthalate
2,4,6-Trichlorophenol	Fluoranthene
Acenaphthene	Fluorene
Acenaphthylene	Hexachlorobenzene
Aniline	Hexachlorobutadiene
Anthracene	Hexachlorocyclopentadiene
Benzidine	Hexachloroethane
Benzo(a)anthracene	Indeno(1,2,3-cd)pyrene
Benzo(a)pyrene	Isophorone
Benzo(b)fluoranthene	1-Methylnaphthalene
Benzo(g,h,i)perylene	2-Methylnaphthalene
Benzo(k)fluoranthene	Naphthalene
Benzyl Alcohol	2-Nitroaniline
4-Bromophenyl Phenyl Ether	3-Nitroaniline
Butyl Benzyl Phthalate	3-Nitroaniline
Carbazole	Nitrobenzene
4-Chloroaniline	N-Nitrosodimethylamine
4-Chloroaniline	N-Nitrosodi-n-propylamine
bis(2-Chloroethyl)ether	N-Nitrosodiphenylamine
2,2'-Oxybis(1-chloropropane)	Phenanthrene
2-Chloronaphthalene	Pyrene
4-Chlorophenyl Phenyl Ether	Pyridine
Chrysene	1,2,4-Trichlorobenzene
Dibenzo(a,h)anthracene	Di-n-octyl Phthalate

Table 5. SW-846 EPA Method 8270D Analyte List	
Dibenzofuran	2,4-Dinitrotoluene

3.3.2 QC Sample Results. The analysis included preparation batch quality control samples required by the DoD/DOE Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.3. For this analysis, the preparation batch included a method blank (MB), laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD). The MB and LCS met the DoD QSM spike acceptance criteria. The MS and MSD were prepared using an aliquot of Sample 2. All DoD QSM spike acceptance criteria were not met. Exceedances are detailed in Table 6.

Table 6. SW-846 EPA Method 8270D MS/MSD Recovery and Relative Percent Difference Exceedances

QC Sample	Analyte	% Recovery	QSM Recovery Acceptance Limits (%)	QSM % Relative Percent Difference (RPD)	QSM RPD Acceptance Limits (%)
Matrix Spike (MS)	4-Chloro-3-methyl Phenol	134	52-119	NA	NA
	1-methylnaphthalene	155	41-119	NA	NA
	2-methylnaphthalene	166	40-121	NA	NA
	3-nitroaniline	23	41-128	NA	NA
	N-nitrosodi-n-propylamine	171	49-119	NA	NA
	1,2,4-trichlorobenzene	119	29-116	NA	NA
	nitrobenzene-d5 (surrogate)	111	44-120	NA	
Matrix Spike Duplicate (MSD)	2-nitrophenol	137	47-123	Met criteria	
	bis(2-chloroethoxy)methane	143	48-120	Met criteria	
	3,3'-dichlorobenzidine	Met criteria		39	20
	hexachloroethane	Met criteria		22	20
	indeno(1,2,3-cd)pyrene	151	52-134	Met criteria	
	isophorone	142	42-124	Met criteria	
	1-methylnaphthalene	178	41-119	Met criteria	
	2-methylnaphthalene	190	40-121	Met criteria	
	3-nitroaniline	37	41-128	47	20
	nitrobenzene	140	45-121	Met criteria	
	N-nitrosodi-n-propylamine	176	49-119	Met criteria	
	1,2,4-trichlorobenzene	138	29-116	Met criteria	
	Nitrobenzene-d5 (surrogate)	130	44-120	NA	

The spike recovery of 3-nitroaniline in the MS and MSD fell below the lower end of the acceptance criteria range, which would indicate results for that analyte in the associated sample (Sample 2) may be low bias, if detected; however, 3-nitroaniline was not detected in the sample (Sample 2). In this case, the limit of detection (DL) and limit of quantitation (LOQ) of the analytes they are associated with are potentially biased low and should therefore be considered estimated values. As none of the analytes that exceeded the upper end of the acceptance criteria were detected in Sample 2, detection and quantification of these analytes was not affected by these exceedances. The surrogate with a recovery exceeding the upper end of the acceptance criteria range also had no impact on the detection and quantification of the analytes it is associated with as none were detected in the sample; however, it is an indication of the presence of matrix interferences present in the sample.

3.3.3 Concentrate Results. Concentrate results are presented in Table 7. Sample extracts required additional dilutions due to matrix interferences, therefore, the LOQ associated with each analyte in each sample greatly varied. The only method analytes detected in any of the samples were benzyl alcohol and N-nitrosodi-n-propylamine, which were detected in Sample 3. Benzyl alcohol was detected and quantified in all three replicates at an average concentration of 21.1 µg/L. Since this concentration is below the LOQ, but greater than the DL, it is considered an estimated concentration, signified by a "J" qualifier and the standard deviation of the three replicates is provided in parentheses in Table 7 below. N-Nitrosodi-n-propylamine was detected and quantified in only one of the replicates.

Table 7. SW-846 EPA Method 8270D Targeted Analysis Sample Results

Analytes	Units	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Target Analytes							
Benzyl Alcohol	µg/L	U	U	21.1 J (3)	U	U	U
N-Nitrosodi-n-propylamine	µg/L	U	U	164	U	U	U

Surrogate recoveries in all samples indicated varying degrees of matrix interferences were present in each sample. Surrogates with recoveries exceeding the upper end of the acceptance criteria range had no impact on the detection and quantification of the analytes they are associated with as none were detected in the sample. Surrogates with recoveries falling below the lower end of the acceptance criteria range indicate the limit of detection (DL) and limit of quantitation (LOQ) of the analytes they are associated with are potentially biased low, therefore should be considered estimated values. A list of the surrogate exceedances associated with each sample, where one or more replicates exceeded the acceptance criteria, is provided in Table 8 below. In the case of Sample 3, all surrogates had a 0% recovery associated with them. As a result, the data associated with this sample is rejected.

Table 8. EPA SW-846 Method 6270D Sample Surrogate Recovery Exceedances		
Sample ID	**Surrogate**	**% Recovery**
Sample 1	2-fluorophenol	One or more replicates had recoveries below acceptance criteria range
	2,4,6-tribromophenol	One or more replicates had recoveries below acceptance criteria range
	nitrobenzene-d5	One or more replicates had recoveries below acceptance criteria range
	2-fluorobiphenyl	One or more replicates had recoveries below acceptance criteria range
	terphenyl-d14	One or more replicates had recoveries below acceptance criteria range
Sample 2	terphenyl-d14	One or more replicates had recoveries below acceptance criteria range
Sample 3	2-fluorophenol	Surrogate was diluted out (0% recovery)
	2,4,6-tribromophenol	Surrogate was diluted out (0% recovery)
	nitrobenzene-d5	Surrogate was diluted out (0% recovery)
	2-fluorobiphenyl	Surrogate was diluted out (0% recovery)
	terphenyl-d14	Surrogate was diluted out (0% recovery)
Sample 4	2-fluorophenol	One or more replicates had recoveries below acceptance criteria range
	2,4,6-tribromophenol	One or more replicates had recoveries below acceptance criteria range
	terphenyl-d14	One or more replicates had recoveries below acceptance criteria range
Sample 5	nitrobenzene-d5	One or more replicates had recoveries greater that acceptance criteria range
Sample 6	2-fluorophenol	One or more replicates had recoveries below acceptance criteria range
	2,4,6-tribromophenol	One or more replicates had recoveries below acceptance criteria range
	nitrobenzene-d5	One or more replicates had recoveries below acceptance criteria range
	2-fluorobiphenyl	One or more replicates had recoveries below acceptance criteria range
	terphenyl-d14	One or more replicates had recoveries below acceptance criteria range

In addition to the targeted analysis performed, compounds were tentatively identified by comparison to a NIST-traceable mass spectra library of compounds. Based on a comparison of the ion masses in this library, compounds are tentatively identified in samples. Concentrations are qualitatively estimated. A list of the tentatively identified compounds reported in each formulation can be found in Table 9.

Table 9. Tentatively Identified Compounds							
Analytes	**Units**	**Sample 1**	**Sample 2**	**Sample 3**	**Sample 4**	**Sample 5**	**Sample 6**
Tentatively Identified Compounds							
Hexylene Glycol	µg/L	1000 JN	NR	NR	NR	NR	
Cyclododecane	µg/L	51 JN	NR	1900 JN	370 JN	1600 JN	86 JN
15-Crown-5	µg/L	29 JN	230 JN	NR	NR	NR	
3,6,9,12,15-Pentaoxanonadecan-1-ol	µg/L	35 JN	NR	NR	NR	NR	
1-Decene	µg/L	63 JN	NR	NR	160 JN	NR	88 JN
1,3,3-Trimethoxybutane	µg/L	43 JN	NR	NR	NR	NR	
3,6,9,12-Tetraoxahexadecan-1-ol	µg/L	54 JN	NR	NR	NR	NR	
Propanoic acid, 3-methoxy-, methyl ester	µg/L	29 JN	NR	NR	NR	NR	
Ethanol, 2-(2-butoxyethoxy)-	µg/L	NR	200 JN	NR	710 JN	200 JN	200 JN
Ethanol, 2-[2-(2-butoxyethoxy)ethoxy]-	µg/L	NR	97 JN	NR	NR	NR	NR
Octane, 1,1'-oxybis-	µg/L	NR	27 JN	NR	NR	NR	NR
1-o-Octyl-.beta.-D-glucopynosid	µg/L	NR	55 JN	NR	NR	NR	NR
Pyrazolo[1,5-a]pyridine, 3,7-dimethyl-2-	µg/L	NR	36 JN	NR	NR	NR	NR
Hexanoic acid	µg/L	NR	770 JN	NR	NR	NR	NR
1,4,7,10,13,16-Hexaoxacyclooctadecane	µg/L	NR	220 JN	190 JN	NR	NR	NR
Bacchotricuneatin	µg/L	NR	27 JN	NR	NR	NR	NR
3,6,9,12,15,18,21-Heptaoxabicyclo[21.3.1]	µg/L	NR	30 JN	NR	NR	NR	NR
1,4,7,10,13,16-Hexaoxanonadecane, 18-(2-	µg/L	NR	190 JN	NR	NR	NR	NR
Propanoic acid, 2-(methoxymethoxy)-	µg/L	NR	37 JN	NR	NR	NR	NR
Silane, (dodecyloxy)trimethyl-	µg/L	NR	NR	320 JN	NR	NR	NR

Table 9. Tentatively Identified Compounds							
Analytes	**Units**	**Sample 1**	**Sample 2**	**Sample 3**	**Sample 4**	**Sample 5**	**Sample 6**
Cyclotetradecane	µg/L	NR	NR	2600 JN	NR	1000 JN	NR
1-Dodecanamine, N,N-dimethyl-	µg/L	NR	NR	1700 JN	270 JN	690 JN	NR
Tridecanoic acid, tert-butyldimethylsily	µg/L	NR	NR	1300 JN	NR	NR	NR
1,1,3-Trimethyl-1-silacyclobutane	µg/L	NR	NR	170 JN	NR	NR	NR
Cyclohexadecane	µg/L	NR	NR	490 JN	NR	NR	NR
1-Pentadecanamine, N,N-dimethyl-	µg/L	NR	NR	260 JN	NR	NR	NR
Silane, (hexadecyloxy)trimethyl-	µg/L	NR	NR	640 JN	NR	NR	NR
1-Tridecanamine, N,N-dimethyl-	µg/L	NR	NR	1400 JN	NR	NR	NR
12-Methylaminolauric acid	µg/L	NR	NR	100 JN	NR	NR	NR
6,6-Dimethyl-9-methylene-undecane-2,5,10	µg/L	NR	NR	180 JN	NR	NR	NR
N,N-Dimethyloctylamine	µg/L	NR	NR	190 JN	NR	NR	NR
N,N-Dimethyltetradecanamine	µg/L	NR	NR	1500 JN	NR	NR	NR
10-Methyl-E-11-tridece-1-ol acetate	µg/L	NR	NR	130 JN	NR	NR	NR
1R,3-cis-Diethoxy-5-cis-methylcyclohexan	µg/L	NR	NR	140 JN	NR	NR	NR
Tetradecyltrimethylammonium bromide	µg/L	NR	NR	240 JN	NR	NR	NR
Estra-1,3,5,7,9-pentaen-17-one, 3-methox	µg/L	NR	NR	90 JN	NR	NR	NR
p-Butoxybenzylidene p-propylaniline	µg/L	NR	NR	78 JN	NR	NR	NR
Pentadecane	µg/L	NR	NR	NR	130 JN	NR	NR
2-Dodecanol	µg/L	NR	NR	NR	71 JN	NR	NR
Pentaethylene glycol	µg/L	NR	NR	NR	67 JN	NR	NR
1,4,7,10,13,16-Hexaoxanonadecane,	µg/L	NR	NR	NR	47 JN	NR	NR
Perfluoronaphthoquinone	µg/L	NR	NR	NR	48 JN	NR	NR
12-Crown-4	µg/L	NR	NR	NR	53 JN	NR	NR

Table 9. Tentatively Identified Compounds							
Analytes	**Units**	**Sample 1**	**Sample 2**	**Sample 3**	**Sample 4**	**Sample 5**	**Sample 6**
Cyclopropane, nonyl-	µg/L	NR	NR	NR	310 JN	NR	NR
Decane, 3,8-dimethyl-	µg/L	NR	NR	NR	120 JN	NR	NR
2-Hexadecanol	µg/L	NR	NR	NR	57 JN	NR	NR
Dodecane, 1-(methoxymethoxy)-	µg/L	NR	NR	NR	65 JN	NR	NR
Pentaethylene glycol	µg/L	NR	NR	NR	57 JN	NR	NR
1,4-Anthracenedione, 2-hydroxy-5-m	µg/L	NR	NR	NR	29 JN	NR	NR
Perfluoronaphthoquinone	µg/L	NR	NR	NR	55 JN	NR	NR
1,4,7,10,13,16-Hexaoxacyclooctadecane	µg/L	NR	NR	NR	70 JN	NR	NR
7-Hexadecene, (Z)-	µg/L	NR	NR	NR	320 JN	NR	NR
l-Guanidinosuccinimide	µg/L	NR	NR	NR	120 JN	NR	NR
1,4,7,10,13,16-Hexaoxanonadecane, 18-pro	µg/L	NR	NR	NR	69 JN	NR	NR
Pentaethylene glycol	µg/L	NR	NR	NR	74 JN	NR	NR
Perfluoronaphthoquinone	µg/L	NR	NR	NR	54 JN	NR	NR
Ethanol, 2-butoxy-	µg/L	NR	NR	NR	NR	2200 JN	34 JN
Cyclooctane, 1,2-dimethyl-	µg/L	NR	NR	NR	NR	490 JN	55 JN
2-Isobutylthiazole	µg/L	NR	NR	NR	NR	930 JN	110 JN
Thiazole, 5-methyl-	µg/L	NR	NR	NR	NR	660 JN	NR
3,5-Diamino-1,2,4-triazole	µg/L	NR	NR	NR	NR	390 JN	NR
Acetone, 1-[4-(dimethylaminoethoxy)pheny	µg/L	NR	NR	NR	NR	200 JN	NR
Triethyl phosphate	µg/L	NR	NR	NR	NR	450 JN	100 JN
Glycocyanidine	µg/L	NR	NR	NR	NR	150 JN	
Hexanoic acid, anhydride	µg/L	NR	NR	NR	NR	120 JN	31 JN
1,4-Dioxaspiro[4.5]decan-8-ol	µg/L	NR	NR	NR	NR	140 JN	
1-Tetradecene	µg/L	NR	NR	NR	NR	NR	53 JN
Trifluoroacetic acid, n-tetradecyl	µg/L	NR	NR	NR	NR	NR	42 JN

Table 9. Tentatively Identified Compounds							
Analytes	**Units**	**Sample 1**	**Sample 2**	**Sample 3**	**Sample 4**	**Sample 5**	**Sample 6**
2-Butanone, 4-(dimethylamino)-3-me	µg/L	NR	NR	NR	NR	NR	30 JN

NR = indicates compound not found in sample

J = indicates concentration is an estimation (qualitative)

N = Tentatively identified compound; Estimated concentration

3.4 EPA Method 300/SW-846 Method 9056A Analyzed by AXYS Orlando

SGS AXYS Orlando prepared and analyzed the six F3 concentrates using their DoD ELAP accredited procedure for EPA Method 300/SW-846 Method 9056A for the 5 anions listed in Table 10. Each sample received was prepared and analyzed in triplicate using a 1:1000 dilution of the sample received.

3.4.1 QC Sample Results. The analysis included preparation batch quality control samples required by the DoD/DOE Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.3. A method blank (MB), laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD) were prepared with the samples. The sample associated with the MS/MSD pair was the NRL 502W sample. All QC samples met DoD QSM acceptance criteria.

3.4.2 Concentrate Results. Concentrate results are presented in Table 10. In instances where a concentration was determined in each triplicate, the average concentration is reported along with the standard deviation of the concentrations in parentheses. In instances where the anion was positively identified, but the concentration fell between the DL and LOQ, a "J" flag is applied to the concentration to indicate it is an estimated value. In instances where the anion was detected in only one of the replicates, that value is reported; when an anion was detected in only two of the three replicates, the average is reported.

Table 10. EPA Method 300/SW-846 Method 9056A Sample Results

	Units	LOQ	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Chloride	mg/L	2.0	3.0 (0)	U	U	U	3.8 (0)	2.8 (0)
Fluoride	mg/L	0.2	U	0.073 J	U	U	0.28 (0.01)	0.078 J (0.001)
Nitrate	mg/L	0.10	U	U	0.049 J (0)	0.051 J (0.01)	0.053 J (0.001)	0.052 J (0.001)
Nitrite	mg/L	0.10	U	U	U	U	U	U
Sulfate	mg/L	2.0	U	U	0.91 J (0.01)	0.81 J (0.01)	U	U

U = indicates anion was not detected above the DL
J = indicates concentration is an estimation (qualitative), less than the LOQ, but greater than the DL

3.5 EPA Method SW-846 Method 6010D Analyzed by AXYS Orlando

SGS AXYS Orlando prepared and analyzed six F3 concentrates using their DoD ELAP accredited procedure for EPA SW-846 Method 6010D for the metals listed in Table 11. Each sample received was prepared and analyzed in triplicate using a 1:1000 dilution of the sample received.

3.5.1 QC Sample Results. The analysis included preparation batch quality control samples required by the DoD/DOE Consolidated Quality Systems Manual for Environmental

Laboratories, Version 5.3. A method blank (MB), laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD) were prepared with the samples. The sample associated with the MS/MSD pair was Sample 2. All QC samples met DoD QSM acceptance criteria.

3.5.2 Concentrate Results. Concentrate results are presented in Table 11. In instances where a concentration was determined in each triplicate, the average concentration is reported along with the standard deviation of the concentrations in parentheses. In instances where the metal was positively identified, but the concentration fell between the DL and LOQ, a "J" flag is applied the concentration to indicate it is an estimated value. In instances where the metal was detected in only one of the replicates, that value is reported; when a metal was detected in only two of the three replicates, the average is reported.

Table 11. EPA Method SW-846 Method 6010D Sample Results

Analytes	Units	LOQ	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Aluminum	µg/L	200	U	U	U	U	U	U
Antimony	µg/L	6.0	U	U	U	U	U	U
Arsenic	µg/L	10	U	U	U	U	U	U
Barium	µg/L	200	U	U	U	U	U	U
Beryllium	µg/L	4.0	U	U	U	U	U	U
Cadmium	µg/L	5.0	U	U	U	U	U	U
Calcium	µg/L	1,000	U	U	189 J	79 J (6)	86 J (2)	184 J (49)
Chromium	µg/L	10	U	U	U	U	U	U
Cobalt	µg/L	50	U	U	U	U	U	U
Copper	µg/L	25	2.6 J	10.3 J	5.8 J	U	1.3 J (0.1)	2.6 J (0.6)
Iron	µg/L	300	U	U	U	U	57.5 J	19.2 J
Lead	µg/L	5.0	U	U	U	U	U	U
Magnesium	µg/L	5,000	U	U	U	U	U	54.3 J
Manganese	µg/L	15	U	U	U	U	U	17.7
Nickel	µg/L	40	U	U	0.40 J	U	U	0.40 J
Potassium	µg/L	10,000	225 J	U	U	489 J (32)	U	402 J (123)
Selenium	µg/L	10	2.9 J	U	3.95 J	3.7 J	3.3 J	U
Silver	µg/L	10	U	U	U	U	U	U
Sodium	µg/L	10,000	3933 J (472)	U	10,190		5473 J (59)	3396 J (960)
Thallium	µg/L	10	U	U	U	U	U	U
Vanadium	µg/L	50	U	U	U	U	U	U
Zinc	µg/L	20	U	7.9 J	22.9	U	U	U

3.6 EPA Method SW-846 Method 7470D Analyzed by AXYS Orlando

SGS AXYS Orlando prepared and analyzed six F3 concentrates using their DoD ELAP accredited procedure for EPA SW-846 Method 7470D for the determination of mercury. Each sample received was prepared and analyzed in triplicate using a 1:1000 dilution of the sample received.

3.6.1 QC Sample Results. The analysis included preparation batch quality control samples required by the DoD/DOE Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.3. A method blank (MB), laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD) were prepared with the samples. The sample associated with the MS/MSD pair and duplicate was Sample 2. All QC samples met DoD QSM acceptance criteria with the exception of the MS and MSD. Both the MS and MSD had a spike recovery of 60.7% while the DoD QSM acceptance criteria is 82-119%.

3.6.2 Concentrate Results. No mercury was detected above the DL in any sample, except for Sample 4. One of the triplicates of this sample had a detectable amount of mercury. The concentration, 0.11µg/L, is barely above the DL, which is 0.10 µg/L. Confirmation of this detection, using a sample prepared using a dilution lower than 1,000, is recommended.

3.7 GC/MS Analyzed by AXYS Antwerp

SGS AXYS Antwerp prepared and analyzed six F3 concentrates using an in-house standard operating procedure which screens for semi-volatile and nonvolatile compounds using GC/MS. Since this is a screening method, it does not fall under DoD ELAP accreditation, therefore the laboratory is not DoD ELAP accredited for this method. Each sample received was prepared in triplicate, by diluting an aliquot of the sample in tetrahydrofuran (THF). Samples were further diluted based on the level of matrix interference and foaming present with each sample. This qualitative test tentatively identified compounds through comparison with a NIST-traceable mass spectra library of compounds. Based on a comparison of the compound masses in this library, compounds are tentatively identified in samples. Concentrations are qualitatively determined.

3.7.1 QC Sample Results. A quality control sample was formed containing a mixture of components at known concentrations. The recoveries of this sample were calculated as a quality check and are shown in Table 12. No standards for acceptable recovery criteria are noted.

Table 12. Recovery of Quality Control Sample

Component	Recovery (%)
Cumene	92
Phenol	92
Nonanal	83
3-Chloroaniline	90
Dodecanoic acid	84
Ethyldecanoate	97
Tetradecane (C14)	97
Diphenylether	93

1-Dodecanol	79
Tributylphosphate	94
Dibutylphthalate	95
Heneicosane	96
Pyrene	94

3.8.2 Concentrate Results. A list of the tentatively identified compounds reported in each concentrate can be found in Table 13. In instances where a concentration was estimated in each triplicate, the average concentration is reported along with the standard deviation of the concentrations in parentheses. All determinations are based on presumptive evidence, as indicated by the "N" qualifier and are an estimated concentrations, as indicated by the "J" qualifier.

Table 13. GC/MS Sample Results

Analyte	Units	Average Estimated Concentration
Sample 1		
Hexylene glycol	mg/kg	16,667 JN (577)
1-Dodecanol	mg/kg	3,267 JN (115)
Methylamine, N-(1-methylheptylidene)-	mg/kg	617 JN (35)
1-Tetradecanol	mg/kg	1,967 JN (58)
Ethanol, 2-(dodecyloxy)-	mg/kg	957 JN (45)
Ethanol, 2-(tetradecyloxy)-	mg/kg	503 JN (25)
Diethylene glycol monododecyl ether	mg/kg	847 JN (42)
Triethylene glycol monododecyl ether	mg/kg	587 JN (31)
Tetraethylene glycol monododecyl ether	mg/kg	337 JN (15)
Sample 2		
Ethanol, 1-(2-butoxyethoxy)-	mg/kg	74,667 JN (5033)
Ethanol, 2-butoxy-	mg/kg	290 JN (20)
Butane, 1,1'-[oxybis(2,1-ethanediyloxy)]bis-	mg/kg	310 JN (70)
Octyl-.beta.-D-glucopyranoside	mg/kg	2,333 JN (462)
Sample 3		
Ethanol, 1-(2-butoxyethoxy)-	mg/kg	57,667 JN (10263)
1-Tetradecene	mg/kg	5,067 JN (929)
1-Dodecanamine, N,N-dimethyl-	mg/kg	7,833 JN (833)
1-Tetradecanol	mg/kg	16,000 JN (1732)
1-Tetradecanamine, N,N-dimethyl-	mg/kg	4,900 JN (173)
1-Hexadecanamine, N,N-dimethyl-	mg/kg	600 JN (80)
1-Decene	mg/kg	287 JN (12)
Diisopropyl ether	mg/kg	247 JN (75)
7-Tetradecenal	mg/kg	9,600 JN (693)
Didodecyldimethylammonium bromide	mg/kg	79 JN (20)
Sample 4		

Table 13. GC/MS Sample Results		
Analyte	**Units**	**Average Estimated Concentration**
Ethanol, 1-(2-butoxyethoxy)-	mg/kg	24,000 JN (1000)
1-Dodecanol	mg/kg	10,300 JN (1572)
Furfural or isomer	mg/kg	237 JN (38)
2,8,9-Trioxa-5-aza-1-silabicyclo[3.3.3]undecane, 1-methyl-	mg/kg	103 JN (6)
Dodecanal	mg/kg	297 JN (47)
1-Tetradecanol	mg/kg	5,433 JN (1332)
Ethanol, 2-(dodecyloxy)-	mg/kg	1,073 JN (467)
Diethylene glycol monododecyl ether	mg/kg	713 JN (153)
Sample 5		
Ethanol, 1-(2-butoxyethoxy)-	mg/kg	27,667 JN (2517)
Ethanol, 2-butoxy-	mg/kg	1,967 JN (115)
1-Dodecanol	mg/kg	9,467 JN (611)
1-Tetradecanol	mg/kg	3,633 JN (1518)
1-Octanol	mg/kg	167 JN (35)
1-Decanol	mg/kg	367 JN (12)
Octane, 1,1'-oxybis-	mg/kg	130 JN (10)
2,8,9-Trioxa-5-aza-1-silabicyclo(3.3.3)undecane, 1-methoxy-	mg/kg	600 JN (118)
2,8,9-Trioxa-5-aza-1-silabicyclo[3.3.3]undecane, 1-methyl-	mg/kg	320 JN (26)
Dodecanal	mg/kg	473 JN (6)
1-Dodecanamine, N,N-dimethyl-	mg/kg	1,287 JN (287)
1-Tetradecanamine, N,N-dimethyl-	mg/kg	1,000 JN (100)
1-Hexadecanol	mg/kg	623 JN (98)
Sample 6		
Ethanol, 1-(2-butoxyethoxy)-	mg/kg	31,667 JN (6028)
1-Dodecanol	mg/kg	3,600 JN (1136)
Furfural or isomer	mg/kg	413 JN (42)
5-Hydroxymethylfurfural	mg/kg	2,133 JN (321)
1-Decanol	mg/kg	317 JN (57)
1-Undecanol	mg/kg	270 JN (56)
Ethanol, 2-(dodecyloxy)- or isomer	mg/kg	160 JN (36)
1-Tetradecanol	mg/kg	1,567 JN (306)
Diethylene glycol monododecyl ether or isomer	mg/kg	163 JN (32)
1-Hexadecanol	mg/kg	377 JN (60)
Diethylene glycol monododecyl ether or isomer	mg/kg	143 JN (25)
Triethylene glycol monododecyl ether or isomer	mg/kg	140JN (17)
Tetraethylene glycol monododecyl ether or isomer	mg/kg	74 JN (16)

3.8 UPLC-QTOF-MS Analyzed by AXYS Antwerp

SGS AXYS Antwerp prepared and analyzed six F3 concentrates using an in-house standard operating procedure which screens for semi-volatile and nonvolatile compounds using UPLC/QTOF in negative ion mode. Since this is a screening method, it does not fall under DoD ELAP accreditation, therefore the laboratory is not DoD ELAP accredited for this method. An MB was prepared and analyzed with the samples for comparison. Each sample received was prepared in triplicate, by making a 1:100,000 dilution of each sample with a 50:50 solution of acetonitrile:reagent water. This qualitative test attempts to identify the components of each sample by comparison with a mass spectra library of compounds. Results are provided in Table 14. All determinations are based on presumptive evidence. Since the samples had to undergo significant dilution prior to analysis, the value of this data is limited.

Table 14. UPLC/QTOF Sample Results		
Sample 1 Results		
Elemental Composition	**m/z**	**Possible Compounds**
C11H21NO	184.1600	Ion masses differed by 28 mass units, indicating the presence of alkane chains such as fatty acids.
C13H25NO	212.1996	
C15H29NO	240.2313	
C17H33NO	268.2620	
C19H37NO	296.2937	
C21H41NO	324.3256	
Sample 2 Results		
Multiple clusters of ions at different retention times with a mass difference of 44 mass units were detected and identified as polyethylene glycols.		
Sample 3 Results		
Elemental Composition	**m/z**	**Possible Compounds**
44 mass unit difference		Polyethylene glycol
C13H27N	230.2478	Decylamine
C15H31N	258.2791	N-Isononycyclohexylamine
C16H33N	240.2686	Hexadecenylamine
Sample 4 Results		
Elemental Composition	**m/z**	**Possible Compounds**
44 mass unit differences		Polyethylene glycols
C13H27N	230.2478	Decylamine
C18H37N	268.2990	Oleylamine
Sample 5 Results		
Elemental Composition	**m/z**	**Possible Compounds**
C11H21NO	184.1600	Ion masses differed by 28 mass units, indicating the presence of alkane chains such as fatty acids.
C13H25NO	212.1996	
C15H29NO	240.2313	
C17H33NO	268.2620	
C19H37NO	296.2937	
C21H41NO	324.3256	
Sample 6 Results		

Table 14. UPLC/QTOF Sample Results		
Elemental Composition	**m/z**	**Possible Compounds**
C11H21NO	184.1600	Ion masses differed by 28 mass units, indicating the presence of alkane chains such as fatty acids.
C13H25NO	212.1996	
C15H29NO	240.2313	
C17H33NO	268.2620	
C19H37NO	296.2937	
C21H41NO	324.3256	

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