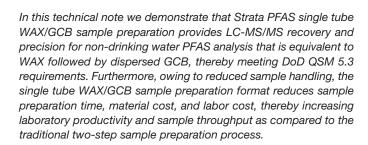
# **P**phenomenex

# TN-0145

# Comparison of PFAS Recoveries Between Cartridge Format WAX/GCB vs. Dispersive GCB for DoD Compliance

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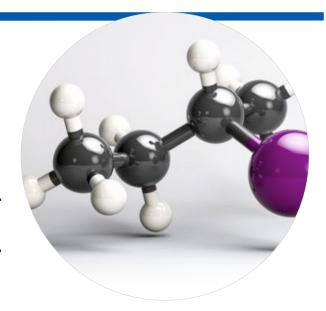
### Introduction

Extensive LC-MS/MS analysis of drinking water samples has firmly established the efficacy of solid phase extraction with a weak anion exchange (WAX) sorbent to extract, pre-concentrate and purify a wide variety of PFAS compounds prior to analysis. However, the analysis of non-drinking water samples, such as treated wastewater, untreated wastewater, and stormwater runoff, is much more challenging. These more complex matrices can introduce additional chromatographic and spectral interferences which can unacceptably depress or inflate analyte recoveries. For such water samples (and also for sediments and soils), additional sample clean-up is required.

# **DoD Development History**

The US Department of Defense (DoD) has undertaken an extensive investigation of PFAS contamination at military installations arising from the use of PFAS containing Aqueous Film Forming Foam (AFFF) to fight jet fuel fires. Fire crews at DoD sites are continually training with AFFF, and this material can then leach into the soil around these sites and potentially enter into the water supply systems for nearby residential areas, both military and civilian. This creates the requirement to analyze both the soil and the water (ground-, surface-, and wastewater) for PFAS contamination at or near these installations.

DoD has established Quality Service Manual (QSM) 5.3 to guide the laboratories that analyze samples from DoD facilities. Table B-15 in QSM 5.3 has specified a two-step sample preparation step utilizing WAX followed by graphitized carbon black (GCB) for the analysis of all non-drinking water matrices. GCB is believed to remove additional matrix interferences (such as humic and cholic acids), which can cause spectral interference and/or suppress ionization. However, long chain PFAS analytes can be strongly adsorbed on GCB, requiring thorough elution of the sorbent prior to LC-MS/MS analysis.



QSM 5.3 does not specify the GCB format to be applied. Initially, laboratories applied the WAX + GCB procedures sequentially by adding finely powdered GCB to the WAX treated eluent, thereby resulting in a dispersion (dGCB). The dGCB must then be removed by centrifugation and filtration and separately eluted to recover the adsorbed PFAS analytes. This time consuming and imprecise procedure was later improved by placing the GCB in a separate SPE tube. This results in a sequential, two-tube procedure (WAX followed by GCB) that is less error prone than WAX followed by dGCB. However, this two-step preparation process is still time consuming and increases laboratory cost through the addition of a second cartridge. A final improvement came when the WAX and GGB were combined in a single tube (Strata PFAS) to offer equivalent analytical performance, but with additional improvements in lower cost and higher laboratory productivity.

# **Experimental Objective**

In this technical note, we present data to demonstrate that PFAS accuracy and precision results obtained using the Strata PFAS single tube sample preparation procedure are equivalent to those obtained using the two-step WAX + dGCB procedure, thereby meeting DoD QSM 5.3 requirements. In **Exhibits 1 - 4** that follow, data will be presented from three commercial laboratories with demonstrated proficiency in generating DoD QSM 5.3 compliant PFAS data. These data are a compilation of excerpts from previously published technical notes and supporting unpublished data which have been curated and assembled to focus on the issue of equivalency and compliance. Readers may refer to the full technical notes for more complete experimental description and discussion, or to consult with the listed authors who are willing to share their experience.

# TN-0145

# Exhibit 1.

Recovery of QSM 5.3 Target Analytes from a Laboratory Control Sample Using Strata® PFAS SPE (WAX/GCB)

(Reference 1. Data provided by Eurofins Lancaster Laboratories)

### **SPE Conditions**

Cartridge: Strata PFAS (WAX/GCB) Dimensions: 200 mg/50 mg/6 mL

Part No.: CSO-9207

Sample pH: Adjust to pH 6-7 with 1M Phosphate Buffer Conditioning: 1:10 mL 0.1% Ammonium Hydroxide in Methanol 2:10 mL Methanol

3:  $10 \, mL$  Phosphate Buffer, pH = 7

Load: 250 mL of sample
Wash: 5 mL 0.1 % Formic Acid in 50:50 Water/Methanol Dry: 2 mins

Elute: 4 mL 0.1 % Ammonium Hydroxide in Methanol Soak: 2 mins

Evaporate: Using Nitrogen, evaporate to below 2 mL

Adjust to 2 mL final volume using 100 % Methanol

# **Discussion**

As shown in Table 1, analyte recoveries in this typical laboratory control sample are well within the recommended method recovery limits for this 31 analyte PFAS panel with a mean recovery of 99.0%, thereby demonstrating compliance with QSM 5.3. Notably, the longer chain PFAS analytes (such as PFDoS and PFOcDA) are well recovered with respect to their lower allowed recovery limits, demonstrating efficient GCB elution in the single tube SPE format.

# Results

Table 1. Recovery of QSM 5.3 Target Analytes from a Typical Laboratory Control Sample

		Using Strata PFAS SPE (WAX/GCB)			
Analyte	Actual Conc.	Sample Result	% Recovery	Method Limits	Pass/Fail
PFBA	25.600	22.640	88	84-135	Pass
PFPeA	25.600	22.157	87	75-138	Pass
PFBS	22.640	22.300	99	81-133	Pass
4:2-FTS	23.920	22.078	92	64-134	Pass
PFHxA	25.600	24.644	96	80-137	Pass
PFPeS	24.000	21.699	90	82-132	Pass
HFPODA	25.600	26.336	103	70-130	Pass
PFHpA	25.600	27.018	106	80-140	Pass
PFHxS	24.200	24.713	102	71-131	Pass
DONA	24.120	26.083	108	70-130	Pass
6:2-FTS	24.280	24.217	100	51-155	Pass
PFHpS	24.360	23.015	94	80-129	Pass
PFOA	25.600	25.043	98	83-138	Pass
PFOS	24.480	22.492	92	54-139	Pass
PFNA	25.600	25.872	101	73-140	Pass
9CI-PF3ONS	23.840	21.863	92	70-130	Pass
PFNS	24.560	21.993	90	71-121	Pass
8:2-FTS	24.520	22.231	91	62-133	Pass
PFOSA	25.600	25.714	100	73-121	Pass
PFDS	24.640	22.873	93	69-124	Pass
PFUnDA	25.600	26.353	103	70-134	Pass
11CI-Pf3OUdS	24.120	22.625	94	70-130	Pass
PFDoDA	25.600	27.710	108	75-139	Pass
10:2-FTS	24.680	26.626	108	50-124	Pass
NMeFOSSA	25.600	29.745	121	38-153	Pass
PFDoS	24.800	21.509	87	39-121	Pass
NEtFOSAA	25.600	25.846	112	36-156	Pass
PFTrDA	25.600	25.814	101	67-144	Pass
PFTeDA	25.600	25.446	99	79-134	Pass
PFHxDA	25.600	29.662	116	36-136	Pass
PFOcDA	25.600	27.373	107	10-124	Pass

Recovery Range: 87.0 % - 116.0 % Average Recovery: 98.8 % Mean Recovery: 99.0 %

# Exhibit 2.

Comparison of Strata® PFAS and Sequential WAX and dGCB Sample Preparation for Laboratory Control Samples

(Reference 2. Data provided by Babcock Laboratories)

### Strata SPE PFAS Conditions

Cartridge: Strata PFAS (WAX/GCB) Dimensions: 200 mg/50 mg/6 mL

Part No.:

Conditioning: 1:10 mL 0.1 % Ammonium Hydroxide in Methanol

2: 10 mL Methanol

3: 10 mL Phosphate Buffer, pH = 7

Load: 250 mL of sample

Wash: 5 mL 0.1 % Formic Acid in 50:50 Water/Methanol

Drv: 2 mins

Elute: 4 mL 0.1 % Ammonium Hydroxide in Methanol

Soak: 2 mins

Evaporate: Using Nitrogen, evaporate to below 2 mL

Adjust to 2 mL final volume using 100 % Methanol

### Sequential WAX and dGCB Conditions

# **WAX Conditions**

Cartridge: Strata X-AW Dimensions: 200 mg/6 mL

Part No.: 8B-S038-FCH
Conditioning: 1:10 mL 0.1% Ammonium Hydroxide in Methanol

2: 10 mL Methanol

3: 10 mL Phosphate Buffer, pH = 7

Load: 250 mL of sample

Wash: 5 mL 0.1 % Formic Acid in 50:50 Water/Methanol Dry: 2 mins

Elute: 4 mL 0.1 % Ammonium Hydroxide in Methanol

Soak: 2 mins

Evaporate: Using Nitrogen, evaporate to below 2 mL

Adjust to 2 mL final volume using 100 % Methanol

# dGCB Conditions

Sample: 2 mL WAX eluent Sorbent: 50 mg ENVI-Carb™ Vortex: 30 sec. Centrifuge: 3000 rpm for 1 min

Syringe Filter: CLARIFY™-PP (polypropylene) 0.45 µm,13 mm (<u>AF8-7101-12</u>)

# **Discussion**

The Strata PFAS recovery and precision results for this 36 analyte PFAS panel presented in Table 2 are within the recommended ranges and are comparable to (but on average slightly better than) those of sequential WAX and dGCB, thereby demonstrating both compliance and equivalency. Note, however, that the dGCB LCS samples were spiked at 50 ng/L whereas the Strata PFAS LSCs were spiked at 20 ng/L, which provided a greater challenge to the equivalency demonstration.

Table 2. Recovery Comparison of WAX + dGCB SPE and Strata **PFAS** 

	Sequentia	l WAX and d	GCB	Strata P	FAS WAX-GO	СВ
Analyte	Spike Conc. (ng/L)	% Recovery	% RSD	Spike Conc. (ng/L)	% Recovery	% RSD
10:2 FTS	50.00	80	6.2	20.00	94	11.5
11CI- PF3OUdS	50.00	85	23.6	20.00	93	7.0
3:3 FTCA	50.00	89	11.6	20.00	86	4.2
4:2 FTS	50.00	100	2.9	20.00	103	2.9
5:3 FTCA	50.00	86	0.6	20.00	94	3.0
6:2 FTS	50.00	98	5.0	20.00	109	4.5
7:3 FTCA	50.00	79	12.6	20.00	90	5.3
8:2 FTS	50.00	97	5.8	20.00	105	3.4
9CI-PF3ONS	50.00	94	18.6	20.00	95	5.9
ADONA	50.00	99	3.2	20.00	100	3.4
EtFOSA	50.00	109	9.6	20.00	104	11.3
EtFOSE	50.00	92	11.8	20.00	92	7.1
HFPO-DA	50.00	110	6.9	20.00	102	9.9
MeFOSA	50.00	108	6.6	20.00	102	16.7
MeFOSE	50.00	93	11.2	20.00	109	8.4
N-MeFOSAA	50.00	103	9.7	20.00	99	12.1
PFBA	50.00	96	1.4	20.00	96	0.6
N-EtFOSAA	50.00	96	6.1	20.00	101	11.2
PFBS	50.00	97	3.2	20.00	98	4.7
PFDA	50.00	101	4.3	20.00	97	6.1
PFDoDA	50.00	100	1.7	20.00	98	3.6
PFDS	50.00	85	21.5	20.00	96	6.9
PFHpA	50.00	99	2.8	20.00	97	3.2
PFHpS	50.00	102	1.9	20.00	92	6.1
PFHxA	50.00	96	2.3	20.00	100	5.4
PFHxDA	50.00	73	15.6	20.00	97	1.0
PFHxS	50.00	97	0.8	20.00	95	7.3
PFNS	50.00	97	10.5	20.00	95	3.7
PFOA	50.00	106	8.0	20.00	101	3.8
PFOcDA	50.00	32	23.8	20.00	87	2.5
PFOS	50.00	96	12.5	20.00	98	5.0
PFPeA	50.00	96	3.6	20.00	98	4.0
PFPeS	50.00	95	4.2	20.00	95	5.7
PFTeDA	50.00	100	2.6	20.00	100	4.2
PFTrDA	50.00	96	12.5	20.00	94	2.2
PFUndA	50.00	104	5.9	20.00	97	0.8
Average (n=4)		94 %	8%		98%	6%

# Exhibit 3.

Comparison of Recoveries using Sequential WAX and dGCB Compared to Strata® PFAS Single Tube WAX-GCB

(Reference 1. Data provided by Eurofins Lancaster Laboratories; Reference 2. Data Provided by Babcock Laboratories; Reference 3. Data Provided by Babcock Laboratories)

### Strata PFAS Conditions

Cartridge: Strata PFAS-WAX/GCB Dimensions: 200 mg/50 mg/6 mL Part No.:

Conditioning: 1:10 mL 0.1 % Ammonium Hydroxide in Methanol

2: 10 mL Methanol

3: 10 mL Phosphate Buffer, pH = 7

Load: 250 mL of sample

Wash: 5 mL 0.1 % Formic Acid in 50:50 Water/Methanol

Dry: 2 mins

Elute: 4 mL 0.1 % Ammonium Hydroxide in Methanol

Soak: 2 mins

Evaporate: Using Nitrogen, evaporate to below 2 mL

Adjust to 2 mL final volume using 100 % Methanol

## Sequential WAX and dGCB Conditions

### **WAX Conditions**

Cartridge: Strata X-AW Dimensions: 200 mg/6 mL Part No.: 8B-S038-FCH

Conditioning: 1: 10 mL 0.1 % Ammonium Hydroxide in Methanol 2: 10 mL Methanol

3:  $10 \, \text{mL}$  Phosphate Buffer, pH = 7

Load: 250 mL of sample

Wash: 5 mL 0.1 % Formic Acid in 50:50 Water/Methanol

Dry: 2 mins Elute: 4 mL 0.1 % Ammonium Hydroxide in Methanol

Soak: 2 mins

Evaporate: Using Nitrogen, evaporate to below 2 mL

Adjust to 2 mL final volume using 100 % Methanol

# dGCB Conditions

Sample: 2 mL WAX eluent Sorbent: 50 mg ENVI-Carb" Vortex: 30 sec Centrifuge: 3000 rpm for 1 min

Syringe Filter: CLARIFY™-PP (polypropylene) 0.45 µm,13 mm (AF8-7101-12)

# Results

Figure 1. Separation of 18 Diverse PFAS Compounds on Kinetex® Kinetex® 5 µm EVO C18 100 x 2.1 mm (Part No.:00D-4633-AN) spiked at 40 ng/L. For future reference in Discussion, note analytes 14, 16, 17 and 18. (From Reference 3)

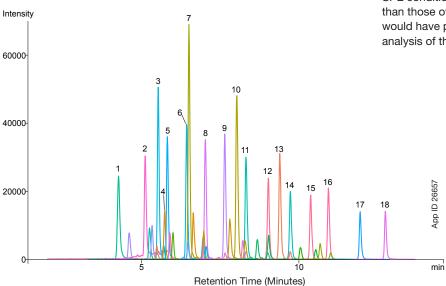


Table 3. Recovery Comparisons of Sequential WAX and dGCB and Strata Single Tube WAX-GCB

	Data from	Data from	Data from
	Reference 1	Reference 2	Reference 3
Analyte	WAX SPE + dGCB Reagent Water	Strata PFAS Reagent Water	Strata PFAS Non-Drinking Water <sup>1</sup>
13C2-PFDoDA	77 %	84 %	106 %2
13C2-PFTeDA	63 %	84 %	104 %²
PFOcDA	38 %	78 %	78 %
PFHxDA	63 %	89 %	96 %

<sup>&</sup>lt;sup>1</sup> The average of the MS and MSD results for four different non-drinking water matrices

# **Discussion**

Figure 1 presents a typical chromatogram for this 16 analyte panel taken from Reference 3. All analytes are well separated by the Kinetex 5 µm EVO C18 column. Note the placement of peaks 14 (PFDoDA), 16 (PFTeDA), 17 (PFOcDA) and 18 (PFHxDA). Recovery data for these long-chain analytes are presented in Table 3.

**Table 3** compares the recovery of these four PFAS analytes by WAX and dGCB (column 1) with their recoveries from Strata PFAS (columns 2 and 3).

The data taken from Reference 1 compares the recoveries achieved for the four long chain PFAS analytes spiked into reagent water. The recoveries of these analytes are higher for Strata PFAS which is consistent with the difficulties that some laboratories have observed in trying to fully elute these analytes from dispersed GCB. The third column presents data taken from Reference 3, which adds a comparison of the same four compounds analyzed in non-drinking water samples (ignoring the differences between native and C13-labled PFOcDA and PFTeDA). These data are the average recoveries of the MS and MSDs for four different non-drinking water matrices for each of the four compounds. The Strata PFAS recoveries from these complex water matrices are equivalent to or slightly higher than the Strata PFAS reagent water results, but are all significantly higher than the sequential WAX and dGCB reagent water results.

These results further demonstrate Strata PFAS equivalency with sequential WAX SPE and dGCB. Although these results were generated in different laboratories and employed slightly different SPE conditions, the Strata PFAS recoveries are sufficiently higher than those of dispersive GCB to suggest that dispersive GCB would have performed more poorly than Strata PFAS for the analysis of the four non-drinking water matrices.

<sup>&</sup>lt;sup>2</sup> Data provided here is for the native versions of these PFAS analytes

# Exhibit 4.

Comparison of Strata® PFAS and Sequential WAX and dGCB Sample Preparation for Laboratory Control Samples

(Reference 4. Data provided by Eurofins TestAmerica)

# **Strata SPE Conditions**

Cartridge: Strata PFAS-WAX/GCB Dimensions: 200 mg/50 mg/6 mL Part No.:

Conditioning: 1:10 mL 0.1 % Ammonium Hydroxide in Methanol

2: 10 mL Methanol

3: 10 mL Phosphate Buffer, pH = 7

Load: 250 mL of sample

Wash: 5 mL 0.1 % Formic Acid in 50:50 Water/Methanol

Drv: 2 mins

Elute: 4 mL 0.1 % Ammonium Hydroxide in Methanol

Soak: 2 mins

Evaporate: Using Nitrogen, evaporate to below 2 mL

Adjust to 2 mL final volume using 100 % Methanol

# Sequential WAX and dGCB Conditions

### **WAX Conditions**

Cartridge: Strata X-AW Dimensions: 200 mg/6 mL Part No.: 8B-S038-FCH

Conditioning: 1: 10 mL 0.1 % Ammonium Hydroxide in Methanol

2: 10 mL Methanol

3:  $10 \, mL$  Phosphate Buffer, pH = 7

Load: 250 mL of sample

Wash: 5 mL 0.1 % Formic Acid in 50:50 Water/Methanol

Dry: 2 mins Elute: 4 mL 0.1 % Ammonium Hydroxide in Methanol

Soak: 2 mins

Evaporate: Using Nitrogen, evaporate to below 2 mL

Adjust to 2 mL final volume using 100 % Methanol

# dGCB Conditions

Sample: 2 mL WAX eluent Sorbent: 50 mg ENVI-Carb Vortex: 30 sec Centrifuge: 3000 rpm for 1 min

Syringe Filter: CLARIFY™-PP (polypropylene) 0.45 µm,13 mm (<u>AF8-7101-12</u>)

# **Results**

The data in Table 4 were taken from a MDL study for 35 PFAS analytes spiked into 7 reagent water samples at the 2 ng/L level. These data directly compare the analyte recoveries for Strata PFAS with those of WAX followed by dGCB. Likewise, Table 5 presents recovery comparison data for the 25 associated isotope dilution analytes spiked at a level of 50 ng/L.

# **Discussion**

The data in this exhibit are consistent with DoD QSM 5.3 acceptance criteria and further demonstrate Strata PFAS equivalency with sequential WAX SPE + dGCB.

Table 4. Recovery Comparison of Native PFAS Analytes Spiked at 2 ng/L (N = 7)

	Strata PFAS		WAX + d	WAX + dGCB	
Analyte	% Recovery	% RSD	% Recovery	% RSD	
Perfluoro(2-propoxypropanoic) acid	107.1	4.11	106.7	4.58	
Perfluorobutanesulfonic acid	108.9	2.07	109.0	3.76	
Perfluorobutanoic acid	127.6	2.68	124.6	2.14	
Perfluorodecanesulfonic acid	103.4	4.74	105.6	10.1	
Perfluorodecanoic acid	103.6	6.18	102.4	5.96	
Perfluorododecanesulfonic acid (PFDoS)	91.90	14.8	96.76	8.60	
Perfluorododecanoic acid	110.7	5.65	105.9	5.55	
Perfluoroheptanesulfonic acid	109.6	4.82	111.7	4.20	
Perfluoroheptanoic acid	112.2	5.12	109.1	5.47	
Perfluorohexadecanoic acid	106.9	6.37	106.8	6.58	
Perfluorohexanesulfonic acid	117.1	7.21	113.5	3.28	
Perfluorohexanoic acid	115.1	2.38	114.9	3.51	
Perfluorononanesulfonic acid	105.1	4.13	105.9	6.51	
Perfluorononanoic acid	119.9	4.71	113.4	4.37	
Perfluorooctadecanoic acid	101.2	8.33	93.00	12.7	
Perfluorooctanesulfonamide	117.6	4.30	115.1	3.41	
Perfluorooctanesulfonic acid	111.7	4.90	110.0	3.78	
Perfluorooctanoic acid	121.2	4.79	113.5	5.05	
Perfluoropentanesulfonic acid	107.7	4.82	107.0	3.92	
Perfluoropentanoic acid	114.9	3.68	112.4	6.02	
Perfluorotetradecanoic acid	110.4	6.05	114.6	8.56	
Perfluorotridecanoic acid	113.1	10.0	109.0	6.87	
Perfluoroundecanoic acid	101.8	6.66	100.8	7.86	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2)	113.9	2.81	113.6	5.32	
1H,1H,2H,2H-perfluorododecanesulfonic acid (10:2)	98.37	5.29	104.6	8.33	
1H,1H,2H,2H-perfluorooctanesulfonic acis (6:2)	118.5	5.22	119.6	6.81	
2(N-ethylperfluoro-1-octanesulfonamido) ethanol	121.9	6.52	126.5	10.9	
2(N-methylperflouro-1-octanesulfonamido) ethanol	116.6	8.99	115.1	8.15	
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	105.5	4.17	111.5	5.33	
11-Chloroeicosafluoro-3-oxaundecane-1-su- fonic acid	98.56	7.12	100.5	5.41	
N-ethylperfluoro-1-octanesulfonamide	104.9	7.59	95.36	8.61	
N-ethylperfluorooctanesulfonamidoacetic acid	111.9	6.77	108.9	7.55	
N-methylperfluoro-1-octanesulfonamide (NMeFOSA)	99.50	8.51	99.57	4.14	
(NH4+) 4,8-dioxa-3H-perfluorononanoate (ADONA)	121.8	3.59	119.3	5.27	
4,8-dioxa-3H-perfluorononanoate (DONA)	121.7	3.64	119.3	5.34	
Average	111 %	5.80 %	110%	6.12 %	

**Table 5.** Recovery Comparison of PFAS Isotope Dilution Analytes Spiked at 50 ng/L (N = 7)

	Strata	Strata PFAS		dGCB
Analyte	Mean Rec. %	% RSD	% Recovery	% RSD
13C2 PFDA	94.97	4.55	100.1	5.52
13C2 PFDoA	84.66	4.96	92.43	5.79
13C2 PFHxA	82.20	3.49	85.11	7.85
13C2 PFHxDA	74.94	10.6	79.29	8.02
13C2 PFTeDA	80.51	7.34	90.89	6.45
13C2 PFUnA	90.09	5.10	97.17	3.57
13C3 HFPO-DA	82.29	3.57	84.83	6.34
13C3 PFBS	72.26	7.20	78.31	8.00
13C4 PFBA	78.89	7.21	80.71	8.26
13C4 PFHpA	86.74	1.94	90.23	7.72
13C4 PFOA	87.06	2.67	92.49	5.46
13C4 PFOS	75.31	6.18	78.00	9.06
13C5 PFNA	85.46	5.12	91.89	4.15
13C5 PFPeA	81.00	4.08	84.43	8.71
13C8 FOSA	66.09	10.5	71.40	8.83
1802 PFHxS	75.48	6.02	79.58	11.3
d3-NMeFOSSA	74.14	10.6	75.77	7.24
d5-NEtFOSAA	74.49	11.0	76.69	5.96
d7-N-MeFOSE-M	48.34	4.43	35.83	10.1
d9-N-EtFOSE-M	39.03	11.9	32.31	12.2
d-N-EtFOSA-M	58.20	12.0	55.37	11.4
d-N-MeFOSA-M	61.11	8.35	64.54	7.95
M2-4:2 FTS	89.32	12.0	84.28	7.89
M2 -6:2 FTS	92.51	12.2	85.83	9.26
M2-8:2 FTS	83.39	9.35	83.72	7.88
Average	76.8 %	7.29 %	78.9 %	7.80 %

# **Overall Technical Note Conclusions**

The data presented in this technical note demonstrate that the Strata® PFAS single tube WAX/GCB sample preparation procedure is in all cases equivalent to — and in some cases better than — the two step procedure employing SPE-WAX and dispersive GCB. In **Table 6** above we have summarized relevant, essential requirements and acceptance criteria from QSM 5.3 Table B-15 (pg 213-224) that pertain to establishing equivalency. The data presented in this technical note — provided by the contributing laboratory authors — were developed under these requirements and fulfilled these acceptance criteria. Therefore, we conclude that the use of Strata PFAS is compliant with DoD QSM 5.3 and can be effectively applied to the analysis of PFAS in non-drinking water samples from DoD facilities.

**Table 6.**Strata PFAS Satisfaction of DoD QSM 5.3 Acceptance Criteria

DoD QSM 5.3 Requirement Category	DoD QSM 5.3 Acceptance Criteria	Phenomenex Equivalency Demonstration
Aqueous Sample Preparation	Solid Phase Extraction (SPE) must be used unless samples are known to contain high PFAS concentrations (e.g., Aqueous Film Forming Foam (AFFF) formulations).	All data presented herein meet these criteria. Therefore, Strata PFAS meets this requirement.
Sample Cleanup Procedure	ENVI-Carb™ or equivalent must be used on each sample and batch QC sample.	All data presented herein meet these criteria. Therefore, Strata PFAS meets this requirement.
Sample PFAS Identification	lon ratios must not exceed 50-150 %. S/N must be ≥ 10 (quantitation) and ≥3 (confirmation). Quant ion and confirmation ion must be present.	All data presented herein meet these criteria. Therefore, Strata PFAS meets this requirement.
ICV/CCV Criteria	Must be within ±30 of true value.	All data presented herein meet these criteria. Therefore, Strata PFAS meets this requirement.
Extracted Internal Standard (EIS) Recovery	Must be within 50 % to 150 % of ICAL midpoint standard area.	All data presented herein meet these criteria. Therefore, Strata PFAS meets this requirement.
Laboratory Control Sample (LCS) Criteria	Blank spiked (one per preparatory batch) with all analytes at concentration >LOQ and < the midlevel concentration. Use Appendix C Table C-44 limits for batch control.	All data presented herein meet these criteria. Therefore, Strata PFAS meets this requirement.
Matrix Spike Duplicate (MS/ MSD) Criteria	Sample spiked (one per preparatory batch) with all analytes at concentration >LOQ and < the midlevel concentration. Use Appendix C Table C-44 limits for batch control. RPD < 30% between MS and MSD	All data presented herein meet these criteria. Therefore, Strata PFAS meets this requirement.

# **Epilog**

While not discussed in this technical note, but demonstrated through the analysis of many thousands of PFAS samples in commercial laboratories — Strata PFAS also offers significant operational and economic benefits over the traditional sample preparation approach. These benefits include:

- Lower reagent consumption and disposal cost
- Lower sample preparation labor cost
- Lower materials cost
- Higher sample throughput
- High process robustness
- Greater laboratory productivity

This combination of high quality data and favorable analytical economics has the potential to favorably advance public and private assessment and remediation of PFAS contamination.

# References

- Data taken from Application Note: Per- and Polyfluoroalkyl Substances (PFAS) Extraction by LC-MS/MS Using Strata PFAS for a Stacked Solid Phase Extraction (SPE) Solution. This application note was developed in collaboration with Eurofins, Lancaster Laboratory, Lancaster, PA. (https://phenomenex.blob.core.windows.net/documents/ba582f2f-4927-4408-aace-4ecfff1a51c2.pdf)
- Previously unpublished data generated in the development of Reference 3 below.
- 3. Data taken from Technical Note TN-1295: Method Validation of a C4-C18 PFAS Panel from Water Extracts Using Dual Mode SPE followed by LC-MS/MS. This technical note was developed in collaboration with Babcock Laboratories, Riverside, CA. (https://phenomenex.blob.core.windows.net/documents/9535e62b-b646-492c-89a7-712b3f1e010b.pdf)
- Previously unpublished data provided by Eurofins TestAmerica, West Sacramento, CA.

Acknowledgement: Special thanks to Thep Phomsopha, Eurofins TestAmerica, for his insights and feedback.

# **Ordering Information**

# Strata® PFAS(WAX/GCB)

Format	Sorbent Mass	Part Number	Unit	
Tube				
strata MAS	200 mg/50 mg	<u>CS0-9207</u>	6 mL (30/box)	

### Kinetex®

5 µm Minibore Columns (mm)					SecurityGuard™ ULTRA Cartridges‡
Phases	30 x 2.1	50 x 2.1	100 x 2.1	150 x 2.1	3/pk
EV0 C18	00A-4633-AN	00B-4633-AN	00D-4633-AN	00F-4633-AN	AJ0-9298
					for 2.1 mm ID

<sup>‡</sup>SecurityGuard ULTRA Cartridges require holder, Part No.: <u>AJO-9000</u>

# **PFAS CRM Native Standards**

All analytes at the same concentration in acid form for easy calculation and dilution.

Product	PN	Qty.	Conc.
EPA 533 mix	AL0-101838	1 mL	2 μg / mL in methanol
EPA 537.1 mix	AL0-101839	1 mL	$2\mu g$ / mL in methanol
EPA 533 + 537.1 mix	AL0-101840	1 mL	2 µg / mL in methanol

# **More PFAS Products for Your PFAS Methods**

Description	Part No.
Luna™ Omega Column 3 µm PS C18 50 x 3 mm	00B-4758-Y0
Kinetex™ EVO Column 5 µm C18 100 x 2.1 mm	00D-4633- AN
Strata™ PFAS (WAX/GCB) SPE 200 mg, /50 mg, /6 mL tubes, 30/pk	CS0-9207
Strata SDB-L SPE 500 mg/6 mL tubes, 30/pk	8B-S014-HCH
Verex™ Vial, 9 mm Screw, PP, 1.7 mL, 1000/pk	AR0-39P0-13
Verex Vial, 9 mm Screw, PP, 300 µL, 1000/pk	AR0-39P2-13
Verex Vial, 9 mm Screw, PP, 700 μL, 1000/pk	AR0-39P1-13
Vial Cap Verex™ Cert+ Cap (one-piece), 9 mm, PE w/ Starburst pre-Slit, 2 mL, 1000/pk	AR0-89P6-13-C

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