

TN-0144

Method Validation of a C4-C18 PFAS Panel from Water Extracts Using Dual-Mode SPE followed by LC-MS/MS

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Introduction

Exposure to manmade perfluoroalkyl and polyfluoroalkyl substances (PFAS) is of increasing concern due to their potential toxic effects and links to ecological damage and human health. These substances can be found in drinking, surface, waste and ground waters, but are also prevalent in food, household products, and even living organisms, including fish and humans. Recently, the state of California¹ has expanded the required list of PFAS for analysis in wastewaters to 42 PFAS compounds from C4 to C18 including carboxylic and sulfonic acids, sulfonamide derivatives, telomers, and ethers. Recently, facilities with bulk fuel storage also require analysis of this expanded PFAS panel.

There are several analytical methods for PFAS, including both ISO and EPA methods, that have been validated for a specific list of PFAS compounds using SDVB or WAX SPE for extraction and preconcentration prior to analysis by LC-MS/MS. EPA 537/537.1 uses SDVB media for SPE while EPA 533 uses WAX media. The WAX approach relies on anion exchange to retain the shorter chain length PFAS compounds in method 533. Both methods have been validated and approved by EPA for drinking water only.

However, many laboratories are using these same drinking water preparation methods for other water matrices such as surface, ground and wastewaters, as well as for soils and sediments. It is not clear what sample preparation steps to apply to these more complex matrices in terms of SPE phase, PFAS compound type, chain length and matrix. In contrast, the US Department of Defense (DOD) quality assurance manual² has specified a two-step SPE approach using WAX followed by a graphitized carbon black (GCB) clean-up for all non-drinking water matrices.

We have previously demonstrated³ the utility of WAX and GCB in a stacked SPE format (known commercially as Strata PFAS) for EPA 533 and 537.1 applied to a variety of water matrices. Strata PFAS saves sample preparation time and cost by combining two sorbent phases into one cartridge. The goal of this study was to validate method performance using Strata PFAS for the expanded list of PFAS compounds recently required by the State of California Water Resources Board for wastewater discharges and bulk fuel storage facilities. Method validation elements included: initial demonstration of capability, method detection limits, limits of quantitation and matrix spike and spike duplicate recovery from several different water matrices.

Sample Preparation

The elements of the sample preparation procedure are described below.

Sample Preparation Steps	
Cartridge:	Strata PFAS-WAX/GCB; 200/50 mg (PN CS0-9207)
Sample pH:	Adjust to pH 6-7 with 1M Phosphate Buffer
Conditioning Steps:	10 mL 0.1% NH ₄ OH in MeOH 10 mL MeOH 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% NH ₄ OH in MeOH
Soak:	2 mins
Dry:	Using N ₂ evaporate to below 2 mL Adjust to 2 mL final volume using 100% MeOH

Sample Analysis Considerations

This separation uses a pH gradient approach³ to simultaneously enable the benefit of changing the eluent system and the ability to change analyte selectivity thereby allowing optimal analyte resolution. The recently introduced California regulations have significantly expanded the PFAS target analyte list to include compounds such as PFBA, PFMBA, PFHxDA and PFOcDA, which have very large differences in hydrophobicity. This presents a significant analytical challenge because PFHxDA (C16) and PFOcDA (C18) are very hydrophobic with limited solubility in water. Conversely, It has been shown that the chromatography of PFBA (C4), in an extract that is > 90% organic exhibits poor peak shape for this early eluting compound. Most methods that can successfully analyze for PFBA are either direct injection (100% water), a 1:1 water-methanol dilution or have at least 20% water in the extract (EPA 533). Some methods (ASTM D7979, D7968 and EPA 8327) add acetic acid to the extract to help improve the peak shape of PFBA. Unfortunately, this results in poorer chromatographic performance for the longer chain PFHxDA (C16) and PFOcDA (C18).

However, the use of a pH gradient using a 100% organic system (for long chain PFAS solubility) and variable mobile phase pH provides good chromatography for PFBA and other early eluting PFAS compounds. By staying within the confines of the NH₄OAc mobile phase composition but employing pH as a variable, one can realize the potential advantages mobile phase variation allowed by EPA while avoiding the primary disadvantages. This approach could be useful in overcoming the growing difficulty

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of expanding the analyte lists of the existing PFAS methods to incorporate both the hydrophilic shorter chain compounds and the extremely hydrophobic longer chain compounds.

Experimental Conditions

Outlined below are the unique method performance characteristics from Table B-15 of DOD QSM 5.3. **Tables 1** and **2** present the monitored SRM transitions for PFAS calibration standards and the isotope standards used in the validation study.

- Calibration/ICV/CCVs (standard)
- Daily instrument sensitivity checks at LOQ +/-50%
- Retention Times
 - RT vs Expected RT (+/- 0.4 min)
 - RT quant ion vs qual ion (+/- 2 sec)
 - RT vs isotope RT (eg: PFOA and 13C-PFOA +/- 0.1 min)
- Ion Ratios 50-150% expected ratios
- Use specific transitions for specific analytes unless matrix is demonstrated and documented

HPLC Conditions

HPLC: Agilent® 1100

Column: Kinetex EVO, C18 100 x 2.1 mm ([00D-4633-AN](#))

Delay Column: Kinetex EVO, C18 EVO 50 x 2.1 2mm ([00B-4633-AN](#))

Column Temp: 35°C

Injection Volume: 10 µL

Eluent A: 20mM Acetic Acid

Eluent B: 25mM Ammonium Hydroxide in Methanol

Time	% A	% B
0.00	95	5
1.20	55	45
3.60	35	65
11.00	10	90
13.00	10	90
13.01	95	5
17.00	95	5

MS Conditions

MS: Thermo TSQ Vantage

Capillary Temp: 250 C

Vapor Temp: 300 C

Sheath gas: 40 arb

Aux Gas: 15 arb

Sweep Gas: 0 arb

CID: 1.0 mTorr Argon

Q1 FWHM: 0.4 Da

Table 1. Monitored SRM Transitions for the PFAS calibration standards. Note that there are no qualifier transition ions for PFBA and PFPeA, and the transition ions for MeFOSE and EtFOSE use acetate adducts.

Analyte	ISTD #	Ion	Q1	Q3 Quantifier ion	CE	Q3 Qualifier ion	CE
PFBA	1	[M-H]-	213	169	9	—	—
PFPeA	2	[M-H]-	263	219	9	—	—
PFHxA	3	[M-H]-	313	269	9	119	25
PFHpA	4	[M-H]-	363	319	9	169	16
PFOA	5	[M-H]-	413	369	9	169	17
PFNA	6	[M-H]-	463	419	10	169	18
PFDA	7	[M-H]-	513	469	10	169	18
PFUnDA	8	[M-H]-	563	519	10	169	22
PFDoDA	9	[M-H]-	613	569	10	169	24
PFTriDA	9	[M-H]-	663	619	10	169	27
PFTeDA	10	[M-H]-	713	669	10	169	29
PFHxDA	11	[M-H]-	813	769	13	169	34
PFcODA	11	[M-H]-	913	869	15	219	30
PFBS	12	[M-H]-	299	80	36	99	28
PFPeS	12	[M-H]-	349	80	38	99	29
PFHxS	13	[M-H]-	399	80	42	99	30
PFHpS	13	[M-H]-	449	80	44	99	32
PFOS	14	[M-H]-	499	80	45	99	34
PFNS	14	[M-H]-	549	80	46	99	35
PFDS	14	[M-H]-	599	80	47	99	36
4:2-FTS	18	[M-H]-	327	307	19	81	29
6:2-FTS	19	[M-H]-	427	407	20	81	29
8:2-FTS	20	[M-H]-	527	507	26	81	33
10:2 FTS	20	[M-H]-	627	607	31	81	44
3:3 FTCA	3	[M-H]-	241	177	10	117	37
5:3 FTCA	5	[M-H]-	341	237	15	217	25
7:3 FTCA	7	[M-H]-	441	337	13	317	21
FOSA	21	[M-H]-	498	78	36	478	10
MeFOSA	22	[M-H]-	512	169	28	219	25
EtFOSA	23	[M-H]-	526	169	29	219	26
MeFOSE	24	[M+HOAc-H]-	616	59	15	—	—
EtFOSE	25	[M+HOAc-H]-	630	59	15	—	—
N-MeFOSAA	16	[M-H]-	570	419	20	483	21
N-EtFOSAA	17	[M-H]-	584	419	20	483	21
HFPO-DA	15	[M-H]-	329	285	6	169	15
ADONA	6	[M-H]-	377	251	13	85	35
9CI-PF3ONS	14	[M-H]-	530.9	351	27	—	—
			532.9	—	—	353	27
11CI-PF3OUdS	14	[M-H]-	630.9	451	30	—	—
			632.9	—	—	453	30



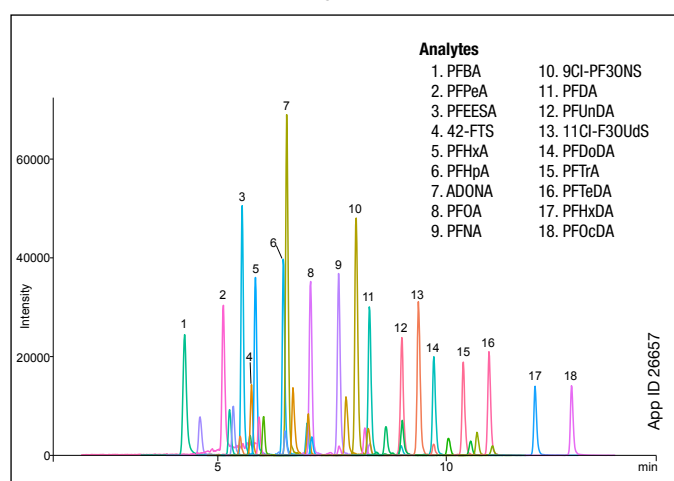
Table 2.
Monitored SRM transitions for isotope standards used in for the validation study.

EIS Analyte	ISTD #	Ion	Q1	Q3	CE
[13C4]-PFBA	1	[M-H]-	217	172	9
[13C5]-PFPeA	2	[M-H]-	268	223	9
[13C5]-PFHxA	3	[M-H]-	318	273	9
[13C4]-PFHpA	4	[M-H]-	367	322	9
[13C8]-PFOA	5	[M-H]-	421	376	9
[13C9]-PFNA	6	[M-H]-	472	427	10
[13C6]-PFDA	7	[M-H]-	519	474	10
[13C7]-PFUnDA	8	[M-H]-	570	525	10
[13C2]-PFDoDA	9	[M-H]-	615	570	10
[13C2]-PFTeDA	10	[M-H]-	715	670	10
[13C2]-PFHxDA	11	[M-H]-	815	770	13
[13C3]-PFBS	12	[M-H]-	302	80	36
[13C3]-PFHxS	13	[M-H]-	402	80	42
[13C8]-PFOS	14	[M-H]-	507	80	45
[13C3]-HFPO-DA	15	[M-H]-	332	287	6
N-MeFOSAA-D3	16	[M-H]-	573	419	20
N-EtFOSAA-D5	17	[M-H]-	589	419	20
[13C2]-4:2FTS	18	[M-H]-	329	309	19
[13C2]-6:2FTS	19	[M-H]-	429	409	20
[13C2]-8:2FTS	20	[M-H]-	529	509	26
[13C8]-FOSA	21	[M-H]-	506	78	36
N-MeFOSA-D3	22	[M-H]-	515	169	28
N-EtFOSA-D5	23	[M-H]-	531	169	29
N-MeFOSE-D7	24	[M+HOAc-H]-	623	59	15
N-EtFOSE-D9	25	[M+HOAc-H]-	639	59	15

Results and Discussion

The results of the study are presented in **Figures 1 and 2** and in **Tables 3-6**. As seen in **Figure 1**, separation of 18 diverse PFAS compounds was readily achieved using the Kinetex EVO C18 column. The chromatogram in **Figure 1** shows excellent peak shape and good chromatographic resolution for the majority of compounds, especially the early eluting PFBA.

Figure 1. Separation of spiked 40ng/L PFAS standards in MeOH.



The results for Method Detection Limits (MDL), Limits of Quantification (LOQ) and Limits of Detection (LOD) are shown in **Table 3**. Most PFAS compounds displayed MDLs in the 1-2 ng/L range, LODs in the 1-2.5 ng/L range, and LOQs at 5 or 8 ng/L. Note that RL/LOQ is the required reporting limit set forth in the California Water Resources Board order WQ 2020-0015-DWQ.

Table 3
PFAS Method Detection Limit, Limits of Detection and Quantitation for the target analytes in ng/L.

Analyte	MDL ^a	LOD ^b	RL/LOQ ^c
PFBA	0.65	1.0	5.0
PFPeA	2.1	2.5	5.0
PFHxA	0.76	1.0	5.0
PFHpA	0.76	1.0	5.0
PFOA	1.2	2.5	5.0
PFNA	0.64	1.0	5.0
PFDA	0.68	1.0	5.0
PFUnDA	0.98	1.0	5.0
PFDoDA	0.85	1.0	5.0
PFTeDA	0.94	1.0	5.0
PFHxDA	0.94	1.0	5.0
PFOS	0.88	1.0	5.0
PFBS	1.1	2.5	5.0
PFPeS	1.3	2.5	5.0
PFHxS	1.5	2.5	5.0
PFHpS	0.87	1.0	5.0
PFNS	3.2	4.5	5.0
PFDS	1.8	2.5	5.0
4:2-FTS	2.4	2.5	5.0
6:2-FTS	0.93	1.0	5.0
8:2-FTS	2.1	2.5	5.0
10:2FTS	2.5	2.5	5.0
N-MeFOSAA	1.9	2.5	8.0
N-EtFOSAA	2.3	2.5	8.0
PFOSA	2.3	2.5	8.0
HFPO-DA	2.7	2.5	8.0
ADONA	2.2	2.5	5.0
9CI-PF3ONS	0.71	1.0	5.0
11CI-PF3OUdS	1.5	2.5	5.0
3:3FTCA	1.4	2.5	5.0
5:3FTCA	4.7	4.5	5.0
7:3FTCA	1.4	2.5	8.0
EtFOSA	2.6	2.5	8.0
EtFOSE	1.8	2.5	8.0
MeFOSA	2.7	2.5	8.0
MeFOSE	2.5	2.5	8.0
NFDHA	2.5	2.5	8.0
PFEESA	2.0	2.5	5.0
PFMPA	0.90	1.0	8.0
PFMBA	0.93	1.0	8.0
PFEChS	1.5	2.5	8.0
	1.0	1.0	8.0

^a MDL was calculated according to 40 CFR Appendix B to Part 136.

^b LOQ is the lowest calibration point equivalent to the reporting limit.

^c LOD was calculated by choosing an analyte concentration between the MDL and LOQ then verified by spiking and recovering at that level. LOD must be at or below the LOQ and generally not set as low as the MDL. It is a practical level at which a known spike can be recovered.



The precision and accuracy results from spike recovery experiments using the Strata PFAS SPE column followed by LC-MS are shown in **Table 4**. The percent recoveries were well within the range required by EPA drinking water standards, with most RSDs falling below 10%.

Table 4.
Precision and Accuracy. Initial Demonstration of Capability from spike extracts into reagent water at a concentration of 20 ng/L.

Analyte	Mean (n=4)	% Rec	RSD
10:2FTS	18.8	94%	11.5%
11Cl-PF30UdS	18.6	93%	7.0%
3:3FTCA	17.2	86%	4.2%
4:2-FTS	20.6	103%	2.9%
5:3FTCA	18.7	94%	3.0%
6:2-FTS	21.9	109%	4.5%
7:3FTCA	18.1	90%	5.3%
8:2-FTS	21.0	105%	3.4%
9Cl-PF30NS	19.1	95%	5.9%
ADONA	20.0	100%	3.4%
EtFOSA	20.7	104%	11.3%
EtFOSE	18.3	92%	7.1%
HFPO-DA	20.4	102%	9.9%
MeFOSA	20.3	102%	16.7%
MeFOSE	21.8	109%	8.4%
N-EtFOSAA	20.2	101%	11.2%
NFDHA	19.9	99%	2.4%
N-MeFOSAA	19.9	99%	12.1%
PFBA	19.2	96%	0.6%
PFBS	19.6	98%	4.7%
PFDA	19.5	97%	6.1%
PFDoDA	19.5	98%	3.6%
PFDS	19.2	96%	6.9%
PFEChS	19.0	95%	5.5%
PFEESA	19.2	96%	2.8%
PFHpA	19.4	97%	3.2%
PFHpS	18.4	92%	6.1%
PFHxA	20.1	100%	5.4%
PFHxDA	19.4	97%	1.0%
PFHxS	19.0	95%	7.3%
PFMBA	19.9	100%	4.5%
PFMPA	20.8	104%	1.5%
PFNA	19.1	96%	4.8%
PFNS	18.9	95%	3.7%
PFOA	20.3	101%	3.8%
PFOcDA	17.4	87%	2.5%
PFOS	19.7	98%	5.0%
PFOSA	21.8	109%	10.3%
PFPeA	19.7	98%	4.0%
PFPeS	19.0	95%	5.7%
PFTeDA	20.1	100%	4.2%
PFTTrA	18.8	94%	2.2%
PFUnDA	19.4	97%	0.8%

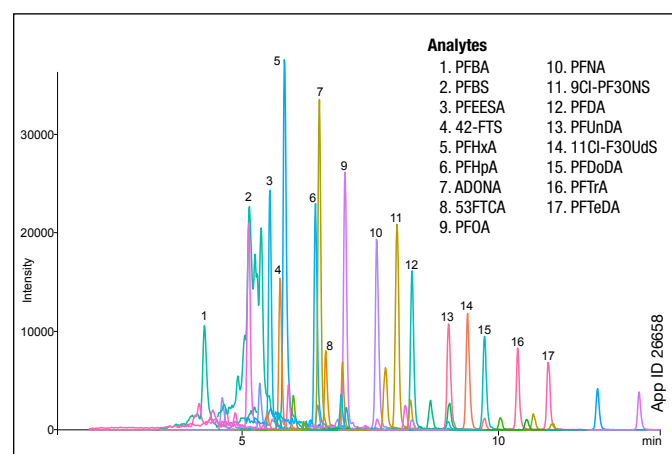
The recoveries for the C16 PFHxDA and C18 PFOcDA show that a WAX based SPE can bind long chain PFAS compounds as well as sulfonamide PFAS compounds, such as PFOSA, MeFOSA, EtFOSE, MeFOSE, EtFOSA, EtFOSSA. Notably, the use of 50mg GCB did not interfere with recoveries of any of these PFAS compounds.

Matrix Studies

Several complex matrices were evaluated to demonstrate analyte recovery. These were designated as Wastewater Effluents 1 and 2, Storm Water Runoff, and Wastewater Discharge Pond. An example of the separation of the PFAS compounds using total ion chromatograph (TIC) for the Wastewater matrix is shown in **Figure 2** below. Excellent peak shape is shown for the majority of PFAS compounds in this and other matrices. Note, however, that the matrix showed matrix interferences for PFBS.

Figure 2.

Matrix spike in Wastewater Effluent. Note the PFBS peak compared to **Figure 1**, signal enhancement resulting in poor recoveries as shown in **Table 5**.



The recovery of matrix spikes and matrix spike duplicates from various water matrices is shown in **Table 5**. The different water matrices had little effect on recoveries, being generally consistent with reagent water recoveries. One notable exception is the result for PFBS which showed a matrix interference effect for both the matrix spike (MS) and matrix spike duplicate (MSD) in all four matrices. A large peak has often been observed at the 299 → 80 in transition in wastewaters. We believe that these commonly observed interferences result from the presence of long chain fatty acids.

The DoD Quality Service Manual requires the use certain mass transitions for quantitation, in order to avoid biasing results high due to known interferences for some transitions. If these required transitions are not used, the reason must be documented and technically justified. Because PFBS showed an isobaric matrix interference at the required mass 80, we chose the alternate mass 99 for reanalysis of the WW Effluent 2 sample. As shown in **Table 5**, excellent PFBS recoveries are now seen, demonstrating that the alternate transition overcomes this common matrix interference.

Note, however, the results from the spiking of internal standards in **Table 6**. General recoverable comparability is again seen with the results of the ISD study. However, there is a notable exception in the recoveries of the fluorotelomer sulfonates, indicating matrix interference in several matrices. It might be possible that some of these interferences could be resolved by chromatography adjustments or alternative mass transition selections, as was the case with PFBS.



Table 5.

Percent Recoveries of a 2 ng/L for Matrix Spikes (MS) and Matrix Spike Duplicates (MSD) of PFAS compounds spikes in a wastewater Discharge pond, Stormwater Runoff and Two Wastewater effluents. Note the matrix effect for PFBS not seen in the IDC studies (Table 4), but subsequently resolved for WW Effluent 2 by using alternate transition mass 99.

	WW Effluent 1		WW Discharge Pond		Stormwater Runoff		WW Effluent 2	
	MS	MSD	MS	MSD	MS	MSD	MS	MSD
N-EtFOSAA	94%	92%	86%	92%	109%	114%	118%	123%
N-MeFOSAA	112%	115%	100%	117%	112%	115%	120%	136%
PFBS ^a	209%	219%	239%	218%	162%	182%	2040%	2003%
PFBS ^b	—	—	—	—	—	—	111%	106%
PFBA	104%	79%	123%	123%	103%	113%	108%	114%
PFPeA	107%	70%	133%	139%	106%	119%	114%	115%
PFHxA	125%	76%	166%	163%	102%	103%	111%	119%
PFHpA	107%	82%	106%	107%	93%	114%	122%	112%
PFHxS	103%	96%	145%	160%	117%	88%	112%	101%
PFOA	96%	71%	121%	114%	93%	113%	126%	117%
PFNS	99%	94%	104%	87%	102%	96%	105%	110%
PFNA	109%	94%	102%	108%	96%	102%	109%	114%
PFDA	113%	85%	104%	110%	99%	95%	117%	125%
PFUnDA	110%	90%	97%	95%	109%	114%	113%	114%
PFDoDA	117%	92%	83%	88%	114%	110%	119%	123%
PFOS	105%	88%	110%	114%	129%	133%	102%	106%
PFTeDA	104%	83%	71%	74%	89%	88%	95%	96%
PFPeS	93%	86%	78%	92%	82%	86%	94%	97%
PFTeDA	107%	94%	67%	67%	116%	108%	118%	127%
PFHxDA	104%	110%	54%	56%	100%	103%	116%	125%
PFoCDA	90%	101%	54%	55%	65%	63%	92%	105%
5:3-FTCA	114%	76%	126%	134%	91%	93%	107%	124%
7:3-FTCA	122%	77%	131%	144%	102%	98%	107%	106%
HFPO-DA	109%	80%	104%	99%	87%	101%	115%	86%
DONA	105%	93%	90%	106%	85%	86%	92%	95%
9CI-PF3ONS	83%	77%	92%	94%	107%	100%	103%	102%
11CI-PF3OUDS	70%	66%	55%	51%	92%	90%	95%	91%
EtFOSE	102%	100%	92%	72%	120%	102%	142%	161%
MeFOSE	86%	106%	64%	83%	124%	102%	130%	118%
EtFOSA	96%	100%	54%	61%	112%	105%	113%	124%
MeFOSA	100%	97%	67%	72%	110%	112%	137%	146%
PFOSA	127%	95%	116%	101%	120%	110%	120%	114%
4:2-FTS	117%	84%	109%	118%	98%	102%	112%	109%
PFHpS	106%	91%	68%	115%	101%	98%	102%	111%
PFDS	82%	86%	51%	69%	95%	83%	96%	96%
8:2-FTS	119%	93%	109%	111%	107%	104%	114%	111%
6:2-FTS	114%	97%	106%	134%	129%	104%	128%	107%
10:2-FTS	107%	105%	70%	69%	47%	54%	80%	87%
3:3-FTCA	101%	94%	84%	83%	83%	71%	23%	54%
PFEESA	108%	120%	97%	95%	98%	112%	111%	106%
PFMPA	121%	126%	102%	116%	132%	129%	134%	133%
PFMBA	117%	103%	107%	116%	114%	112%	125%	123%
NFDHA	111%	111%	96%	108%	104%	103%	117%	120%

^a PFBS MS 299- 80 used as quantifier ion

^b PFBS MS 299 – 99 used as quantifier ion

Table 6.

Percent Recoveries of a 2 ng/L for Matrix Spikes (MS) and Matrix Spike Duplicates (MSD) of PFAS compounds ISTD spikes in a wastewater Discharge pond, Stormwater Runoff and Two Wastewater effluents. Note the matrix effects for several fluorotelomer sulfonates seen here, but not seen in the IDC studies in Table 4.

	WW Effluent		WW Discharge Pond		Stormwater Runoff		WW Effluent	
ISTD	MS#1	MS#2	MS#3	MS#4	MS#5	MS#6	MS#7	MS#8
[13C4]-PFBA	67%	74%	84%	78%	57%	54%	65%	63%
[13C5]-PFPeA	77%	89%	92%	87%	79%	75%	86%	85%
[13C5]-PFHxA	86%	100%	96%	92%	95%	94%	101%	94%
[13C4]-PFHpA	97%	110%	114%	104%	108%	105%	107%	106%
[13C8]-PFOA	98%	112%	118%	114%	110%	111%	110%	109%
[13C9]-PFNA	99%	98%	121%	109%	120%	118%	119%	110%
[13C6]-PFDA	91%	102%	107%	99%	116%	127%	121%	116%
[13C7]-PFUnDA	91%	95%	98%	92%	109%	115%	123%	123%
[13C2]-PFDoDA	84%	86%	112%	102%	92%	99%	107%	99%
[13C2]-PFTeDA	67%	65%	119%	107%	54%	60%	62%	56%
[13C2]-PFHxDA	59%	58%	124%	113%	42%	37%	34%	29%
[13C3]-PFBS	92%	99%	116%	109%	94%	87%	93%	96%
[13C3]-PFHxS	111%	119%	148%	112%	112%	114%	107%	103%
[13C8]-PFOS	110%	105%	107%	106%	101%	115%	107%	111%
[13C3]-HFPO-DA	86%	92%	103%	88%	77%	74%	66%	8700%
N-MeFOSAA-D3	88%	96%	112%	104%	102%	115%	136%	122%
N-EtFOSAA-D5	95%	101%	135%	130%	100%	108%	128%	121%
[13C2]-4:2FTS	198%	227%	225%	205%	336%	334%	246%	255%
[13C2]-6:2FTS	136%	139%	384%	325%	325%	383%	199%	227%
[13C2]-8:2FTS	88%	95%	208%	199%	180%	191%	141%	143%
[13C8]-FOSA	79%	89%	95%	85%	84%	92%	93%	98%
N-MeFOSA-D3	75%	73%	74%	65%	84%	58%	71%	72%
N-EtFOSA-D5	75%	70%	68%	64%	43%	50%	72%	67%
N-MeFOSE-D7	88%	82%	57%	49%	59%	74%	76%	78%
N-EtFOSE-D9	75%	74%	53%	51%	54%	64%	59%	55%

Conclusions

The data shows that WAX / GCB stacked dual SPE phase can be used to extract for short and long chain PFAS compounds, for carboxylic, sulfonic, sulfonamides and derivatives, fluorotelomer sulfonates, fluorotelomer carboxylic acids, perfluoroalkyl ether carboxylic acids, chlorinated polyfluoroalkyl ether sulfonic acids from a wide variety of water matrices. The use of the Kinetex EVO C18 column (and its stability at high pH) allows the use of a pH gradient to effectively separate water and organic soluble PFAS compounds while maintaining excellent peak shape. Finally, it was demonstrated that the flexibility to use alternate transitions is crucial to overcome matrix interferences in complex water matrices.

References

1. California Water Code Sections 13267 and 13383, order for determination on the presence of per- and polyfluoroalkyl substances at publicly owned treatment works. Order WQ 2020-0015-DWQ.
2. Department of Defense (DoD) Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD Quality Systems Manual Version 5.3, 2019.
3. PFAS Analysis Based Upon a pH-Variable LC Mobile Phase Gradient, Phenomenex Technote 1280 (TN-1280).



Strata Ordering Information

PFAS (WAX/GCB)			
Format	Sorbent Mass	Part Number	Unit
Tube	200 mg/ 50 mg	CS0-9207	6 mL (30/box)

Kinetex Ordering Information

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

†SecurityGuard ULTRA cartridges require holder, Part No.: [AJ0-9000](#)

for 2.1 mm ID

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