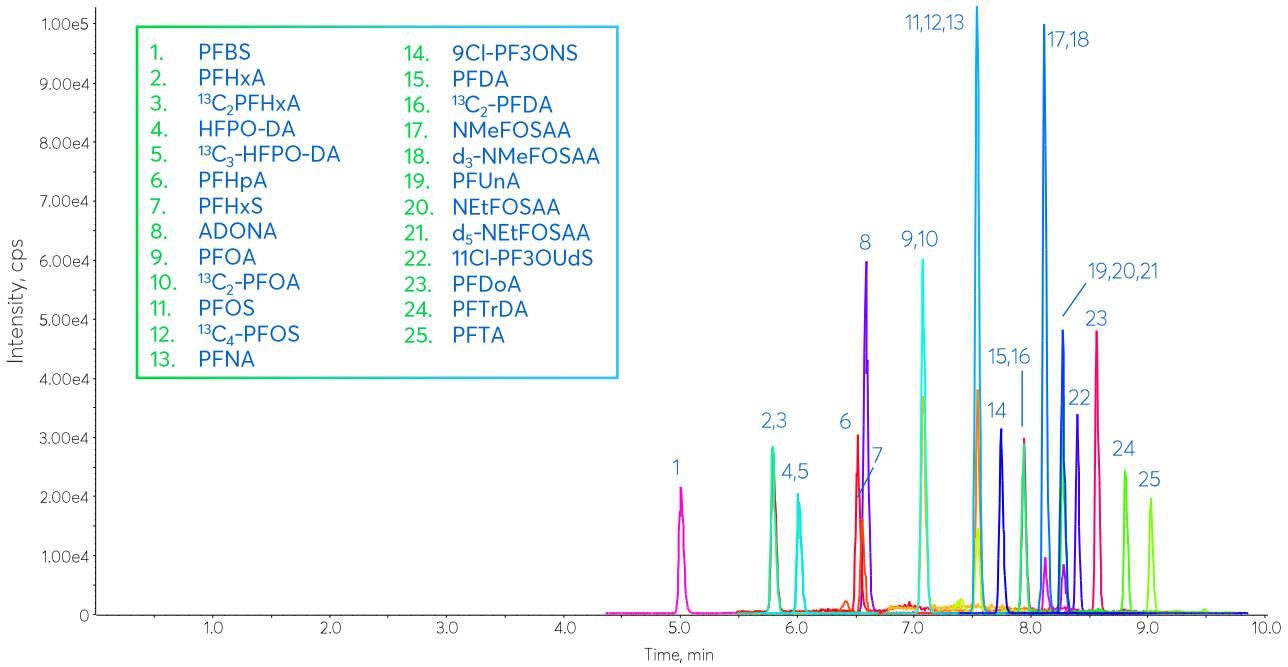
avantor™

Chromatography Solutions

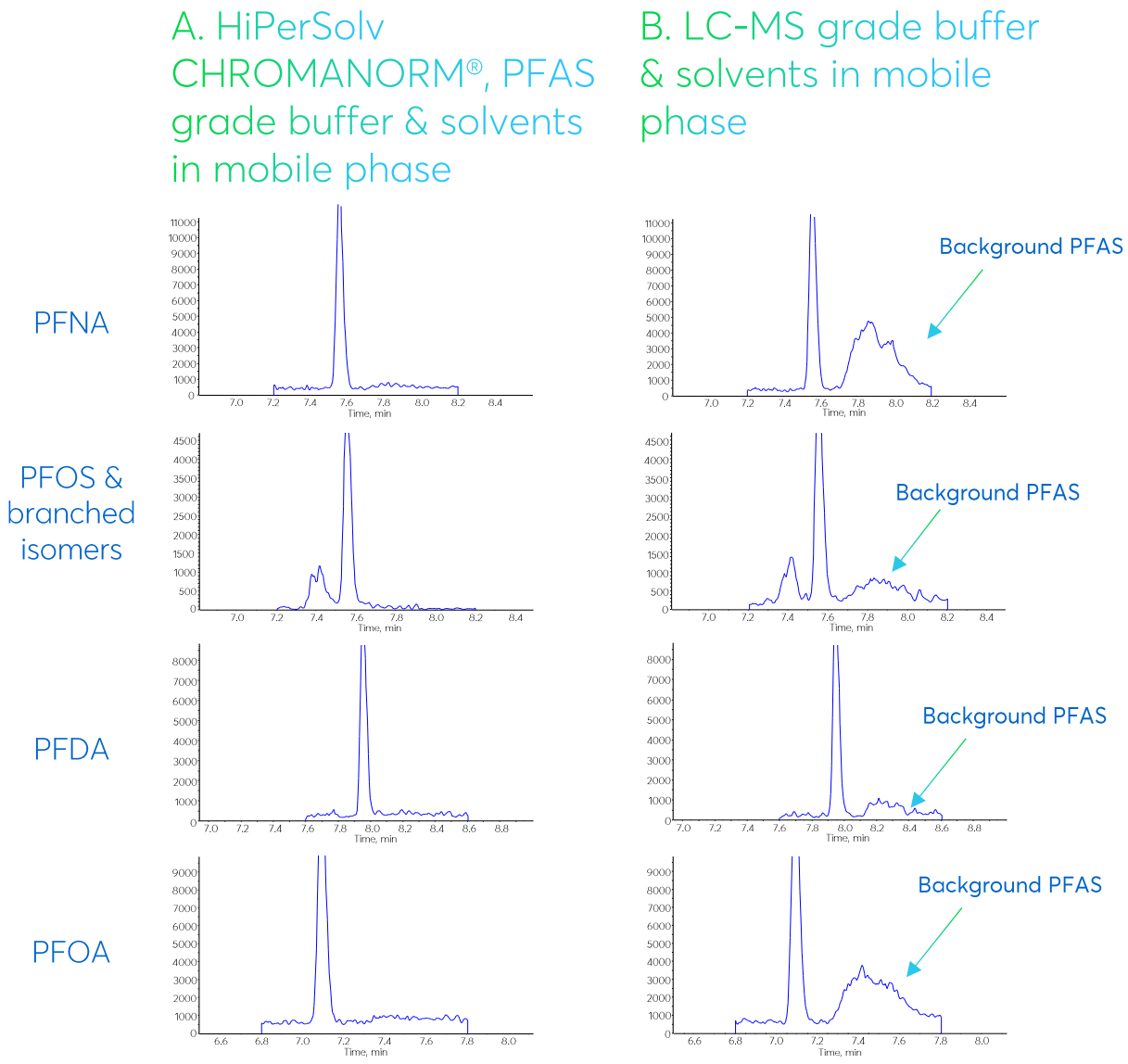
Application note #7820

PFAS Analysis by EPA method 537.1 using VWR® HiPerSolv CHROMANORM® PFAS grade solvents



Avantor® ACE®

Comparison of CHROMANORM® PFAS grade 5 mM ammonium acetate to LC-MS grade



Background PFAS present in the mobile phase are trapped by the delay column and elute after the sample PFAS peak. Lower levels of background PFAS were observed when using HiPerSolv CHROMANORM®, PFAS grade pre-mixed 5 mM ammonium acetate in the mobile phase compared to LC-MS grade solvents and buffers. Mobile phases: A) HiPerSolv CHROMANORM®, PFAS grade 5 mM ammonium acetate (premixed) in Line A and HiPerSolv CHROMANORM®, PFAS grade MeOH in Line B, B) LC-MS grade ammonium acetate (5 mM) in LC-MS grade H_2O in Line A, LC-MS grade MeOH in Line B.

Method Details

CONDITIONS

Column: Avantor® ACE® Excel® C18

Particle Size: 3 µm

Dimensions: 100 x 2.1 mm

Delay Column: Avantor® ACE® PFAS Delay Column

Dimensions: 50 x 2.1 mm

Mobile Phases: A: 5 mM ammonium acetate in H_2O (HiPerSolv CHROMANORM®, PFAS grade)

B: MeOH (HiPerSolv CHROMANORM®, PFAS grade)

Time (mins)	% B
0	5
0.1	20
8.5	95
10.5	95
10.6	5
13.2	5

Flow Rate: 0.4 mL/min

Temperature: 40 °C

Injection volume: 1 µL

Detection: Sciex QTRAP® 6500+ LC-MS/MS system.

Ionisation mode: ESI, negative mode; Source temperature: 450 °C; Curtain gas: 30 psig; Ionspray™ source voltage: -4500 V; Ion source gas: 60 psig

Sample: Calibration standard with PFAS standards, internal standards and surrogate standards at 500 ng/L (corresponding to an in sample concentration of 2 ng/L, taking into account 250x sample pre-concentration during sample preparation specified in EPA method 537.1).

MRM TRANSITIONS

Analyte	MRM	Optimised MS Parameters		
		Declustering potential (V)	Collision energy (V)	Cell exit potential (V)
1. PFBS (Perfluorobutanesulfonic acid)	-298.8 → -79.9	-60	-66	-13
2. PFHxA (Perfluorohexanoic acid)	-312.8 → -268.8	-5	-12	-29
3. $^{13}C_2$PFHxA (Surrogate standard)	-314.8 → -269.9	-5	-12	-17
4. HFPO-DA (Hexafluoropropylene oxide dimer acid)	-285.0 → -169.0	-5	-10	-35
5. $^{13}C_3$-HFPO-DA (Surrogate standard)	-286.8 → -168.9	-10	-10	-15
6. PFHpA (Perfluoroheptanoic acid)	-362.8 → -318.8	-5	-14	-25
7. PFHxS (Perfluorohexanesulfonic acid)	-398.8 → -79.9	-5	-86	-9
8. ADONA (4,8-Dioxa-3H-perfluorononanoic acid)	-376.8 → -250.8	-15	-16	-23
9. PFOA (Perfluorooctanoic acid)	-412.8 → -368.9	-10	-14	-23
10. $^{13}C_2$-PFOA (Internal standard)	-414.8 → -369.8	-10	-14	-19
11. PFOS (Perfluorooctanesulfonic acid)	-498.8 → -79.9	-5	-102	-9
12. $^{13}C_4$-PFOS (Internal standard)	-502.8 → -79.9	-5	-104	-9
13. PFNA (Perfluorononanoic acid)	-462.8 → -418.8	-5	-16	-23
14. 9Cl-PF3ONS (9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid)	-530.7 → -350.8	-20	-36	-29
15. PFDA (Perfluorodecanoic acid)	-512.8 → -468.8	-25	-16	-45
16. $^{13}C_2$-PFDA (Surrogate standard)	-514.8 → -469.8	-5	-16	-25
17. NMeFOSAA (N-methyl perfluorooctanesulfonamidoacetic acid)	-569.8 → -418.8	-5	-30	-29
18. d_3-NMeFOSAA (Internal standard)	-572.8 → -418.9	-5	-30	-29
19. PFUnA (Perfluoroundecanoic acid)	-562.8 → -518.7	-5	-18	-33
20. NEtFOSAA (N-ethyl perfluorooctanesulfonamidoacetic acid)	-583.8 → -418.9	-30	-28	-19
21. d_5-NEtFOSAA (Surrogate standard)	-588.8 → -418.9	-25	-28	-21
22. 11Cl-PF3OUdS (11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid)	-630.7 → -450.8	-5	-42	-23
23. PFDoA (Perfluorododecanoic acid)	-612.7 → -568.8	-10	-18	-35
24. PFTrDA (Perfluorotridecanoic acid)	-662.7 → -618.7	-15	-20	-35
25. PFTA (Perfluorotetradecanoic acid)	-712.7 → -668.6	-5	-20	-41

ACCURACY AND PRECISON

Calibration curves were generated using PFAS standards and isotopically labelled internal standards between 0.01–2.0 ng/mL (Figure 1). Table 1 shows accuracy values calculated for triplicate injections of the calibration standards. Good precision from multiple mid-level QC samples (n=7) was obtained (Table 2).

Table 1: Example accuracy data for ADONA calibration curve (Internal standard: $^{13}C_2$-PFOA). *The corresponding in sample concentration takes into account the 250x concentration step during sample preparation specified in EPA method 537.1.

Concentration (in vial) ng/L	Concentration (in sample) ng/L*	% Accuracy
10	0.04	110.3
15	0.06	103.5
20	0.08	110.0
50	0.2	96.8
100	0.4	107.3
200	0.8	104.9
500	2.0	106.2
1000	4.0	106.7
2000	8.0	94.1

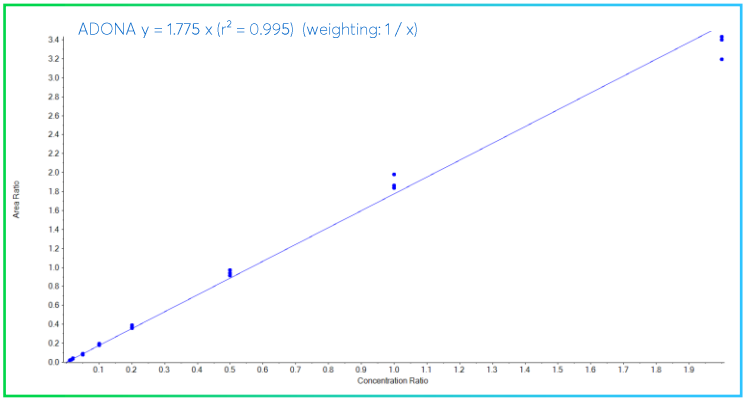


Figure 1: Example calibration curve for ADONA, fitted to linear calibration line forced through zero (weighted 1/x).

Table 2: Accuracy and Precision data for QC sample (7 replicate injections).

Analyte	Concentration (in vial) ng/L	Mean calculated ng/L	% Precision	% Accuracy
9Cl-PF3ONS	200	198.4	5.3	99.2
11Cl-PF3OUdS	200	189.0	5.4	94.5
ADONA	200	186.4	1.5	93.2
NEtFOSAA	200	200.9	5.5	100.4
NMeFOSAA	200	193.9	6.4	96.9
PFBS	200	184.1	3.5	92.0
PFDA	200	191.6	5.3	95.8
PFDoA	200	193.1	4.6	96.6
PFHpA	200	189.3	4.8	94.6
PFHxA	200	195.4	3.2	97.7
PFHxS	200	189.0	2.9	94.5
PFNA	200	193.9	3.6	96.9
PFOA	200	187.6	3.6	93.8
PFOS	200	213.3	2.8	106.6
PFTA	200	186.3	4.4	93.1
PFTrDA	200	193.0	4.8	96.5
PFUnA	200	185.9	4.4	93.0
HFPO-DA	200	188.7	3.1	94.3
$^{13}C_2$-PFDA (Surrogate)	200	203.5	2.2	101.7
$^{13}C_2$PFHxA (Surrogate)	200	199.8	3.1	99.9
$^{13}C_3$-HFPO-DA(Surrogate)	200	205.0	3.7	102.5
d_5-NEtFOSAA (Surrogate)	800	825.9	4.8	103.2

ORDERING TABLE

Product	Details	Size	Part Number
Avantor® ACE® Excel® C18	HPLC Column	100 x 2.1 mm	EXL-111-1002U
Avantor® ACE® PFAS Delay Column	Pre-column trap	50 x 2.1 mm	ACE-PFASD-0502
Ammonium acetate 5 mM in water	VWR® HiPerSolv CHROMANORM® PFAS grade eluent for LC-MS	1 L	92500.290
Methanol ≥99.9%	VWR® HiPerSolv CHROMANORM® PFAS grade for LC-MS	2.5 L	92498.320

RELATED PRODUCTS

Product	Details	Size	Part Number
Acetonitrile ≥99.9%	VWR® HiPerSolv CHROMANORM® PFAS grade for LC-MS	2.5 L	92497.320
Water ≥99.9%	VWR® HiPerSolv CHROMANORM® PFAS grade for LC-MS	2.5 L	92499.320