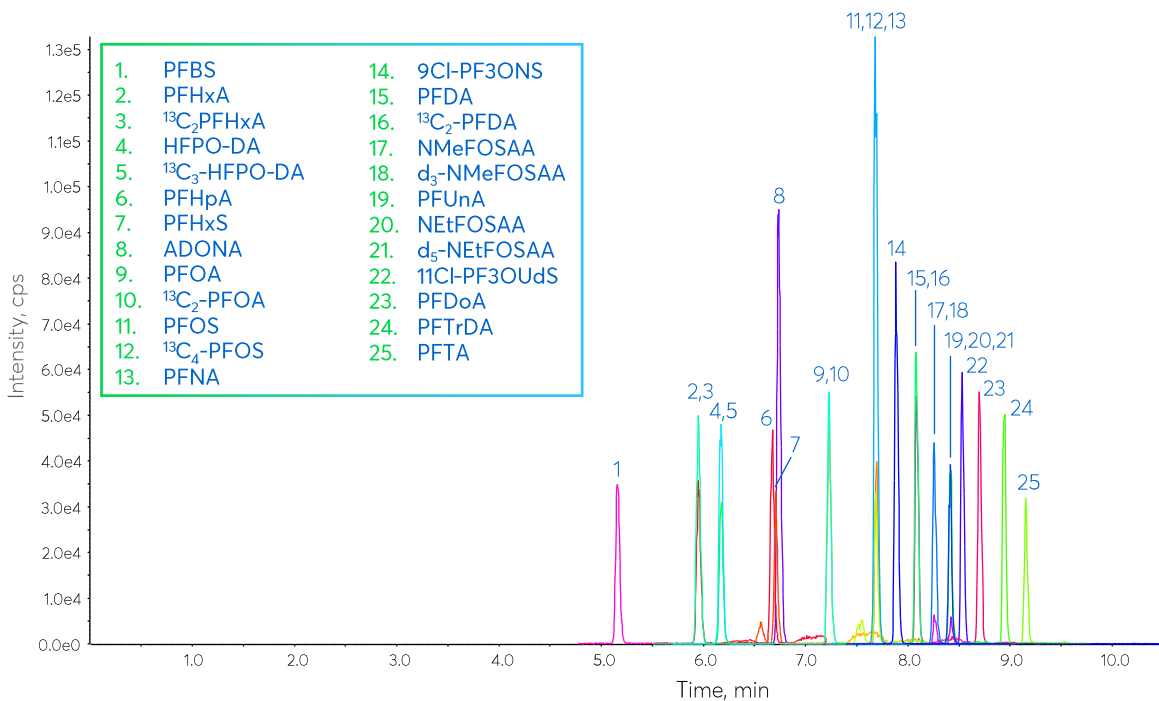


LC-MS/MS Analysis of PFAS Compounds using EPA method 537.1



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: 10 mM ammonium acetate in H₂O
 B: MeOH

Time (mins)	% B
0	5
0.1	20
8.5	95
10.5	95
10.6	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 1000 ng/L (corresponding to an in sample concentration of 4 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. PFTTrDA (Perfluorotridecanoic acid)	-662.7 → -618.7	-15	-20	-35
25. PFTA (Perfluorotetradecanoic acid)	-712.7 → -668.6	-5	-20	-41

ACCURACY AND PRECISION

Calibration curves were generated using PFAS standards and isotopically labelled internal standards between 0.005–2.5 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=6) was obtained (Table 2).

Table 1: Example accuracy data for ADONA calibration curve between 5 to 2500 ng/mL (Internal standard: ¹³C₂-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
5	0.02	110.7
10	0.04	101.7
15	0.06	110.1
50	0.2	93.0
100	0.4	106.8
200	0.8	102.4
500	2.0	91.7
1000	4.0	94.6
2000	8.0	103.8
2500	10.0	100.4

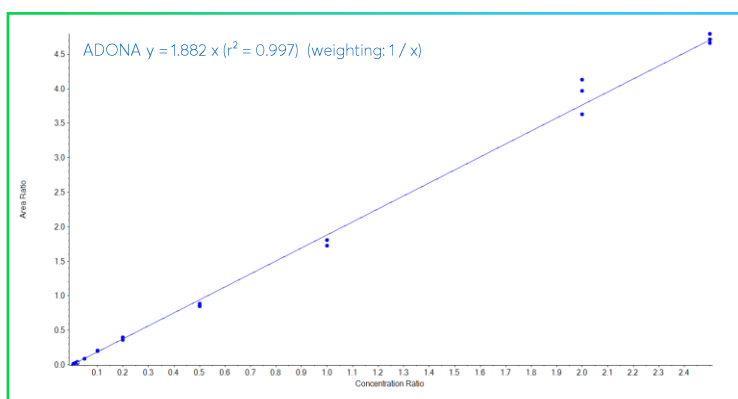


Figure 1: Example calibration curve for ADONA, fitted to linear calibration line forced through zero (weighted 1/x).

Table 2: Accuracy and Precision data for QC sample (6 replicate injections).

Analyte	Concentration (in vial) ng/L	Mean calculated ng/L	% Precision	% Accuracy
9CI-PF3ONS	100	106.0	5.3	106.0
11CI-PF3OUdS	100	104.9	1.9	104.9
ADONA	100	104.8	3.3	104.8
NEtFOSAA	100	104.9	6.2	104.9
NMeFOSAA	100	99.7	8.5	99.71
PFBS	100	105.1	3.8	105.1
PFDA	100	107.2	8.2	107.2
PFDaA	100	109.4	5.0	109.4
PFHpA	100	109.3	4.4	109.3
PFHxA	100	108.5	5.0	108.5
PFHxS	100	104.7	4.5	104.7
PFNA	100	109.9	5.3	109.9
PFOA	100	111.3	4.1	111.3
PFOS	100	113.6	3.8	113.6
PFTA	100	110.5	3.5	110.5
PFTTrDA	100	109.0	3.7	109.0
PFUnA	100	104.4	2.7	104.4
HFPO-DA	100	103.6	5.2	103.6

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
Methanol	VWR HiPerSolv CHROMANORM® for LC-MS	2.5 L	83638.320
Water	VWR HiPerSolv CHROMANORM® for LC-MS	2.5 L	83645.320
Ammonium acetate	VWR HiPerSolv CHROMANORM® for LC-MS	100 g	84885.180