

Sample pretreatment

MN Appl. No. 306950

Solid phase extraction according to EPA Draft Method 1633

Sample preparation:

This method is applicable to aqueous samples containing up to 50 mg of suspended solids per sample. The procedure requires the preparation of the entire sample. Subsampling should be avoided whenever possible. Typical sample size is 500 mL.

1. Homogenize the sample by inverting the sample 3 – 4 times and allowing the sample to settle. Do not filter the sample.
2. Check that the pH is 6.5 ± 0.5 . If necessary, adjust pH with 50 % formic acid or ammonium hydroxide. The extract is now ready for solid-phase extraction (SPE).
3. Add the spiking solution containing the internal standard substances to the water sample (500 mL) in the sample bottle (adding 2.5 ng of each) and mix thoroughly by shaking.

Column: CHROMABOND® WAX, 6 mL 150 mg (REF 7300011)

Conditioning:

With 15 mL of 1 % methanolic ammonium hydroxide, followed by 5 mL of 0.3M formic acid. Do not allow the SPE to run dry. Discard the wash solvents.

Sample application:

Add 500 mL water sample with a flow rate of 5 mL/min to the cartridge. (Do not let the sorbent material in the cartridge run dry and ensure it is always immersed in water.)

Bottle Rinse:

Rinse the walls of the reservoir with 5 mL reagent water (twice) followed by 5 mL of 1:1 0.1M formic acid/methanol.

Washing step:

Pass those rinses through the cartridge using vacuum. Discard the rinse solution.

Drying step:

Dry the cartridge by pulling air through for 15 seconds.

Elution:

Rinse the inside of the sample bottle and the SPE reservoir with 5 mL of 1 % methanolic ammonium hydroxide. Use vacuum to pull the elution solvent through the cartridge and into the collection tubes.

Clean-up:

Add 25 µL of concentrated acetic acid to each sample eluted in the collection tubes and vortex to mix. Add 10 mg of carbon (GCB) to each sample. Immediately vortex (30 seconds) and centrifuge at 2800 rpm for 10 minutes.

Membrane Filtration:

Filter eluate through a syringe filter (25-mm filter, 0.2µm nylon membrane, REF 729212) into polypropylene vial.

Analysis by HPLC-MS / MS

MN Appl. No. 129370

Chromatographic conditions

DELAY Column	EC 50/2 NUCLEODUR® PFAS Delay (REF 760673.20)
Column	EC 100/2 NUCLEODUR® PFAS, 3 µm (REF 760666.20)
Eluent A	5 mM ammonium acetate in water
Eluent B	5 mM ammonium acetate in methanol
Gradient	hold 40 % B for 1 min, in 8 min from 40 % B to 95 % B, hold 95 % B for 3 min, in 0.1 min to 40 % B, hold 40 % B for 2.9 min
Flow rate	0.3 mL/min
Temperature	40 °C
Injection volume	2 µL
MS conditions	
Acquisition mode	SRM
Interface	ESI
Polarity	negative
Curtain Gas	30
Collision Gas	medium
Ionspray Voltage	- 4500 V
Temperature	400 °C
Ion Source Gas 1	50
Ion Source Gas 2	60
Detection Window	60 sec



MRM transitions

Analyte	Abbreviation	CAS number	Q1 mass [Da]	Q3 mass [Da]	Retention time [min]
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1	229.00	85.00	1.96
Perfluoro- <i>n</i> -butanoic acid	PFBA	375-22-4	212.90	168.80	2.01
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5	241.00	177.00	3.33
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5	279.00	85.00	3.64
Perfluoro- <i>n</i> -pentanoic acid	PFPeA	2706-90-3	262.88	219.00	3.90
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7	315.00	135.00	4.14
Perfluoro- <i>n</i> -butanesulfonic acid	PFBS	375-73-5	298.93	98.90	4.20
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6	295.00	201.00	4.48
1 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorohexane sulfonic acid	4:2FTS	757124-72-4	326.94	306.90	5.27
Perfluoro- <i>n</i> -hexanoic acid	PFHxA	307-24-4	312.91	268.80	5.40
Perfluoropentanesulfonic acid	PFPeS	2706-91-4	348.85	80.00	5.54
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	284.99	168.70	5.77
2 <i>H</i> ,2 <i>H</i> ,3 <i>H</i> ,3 <i>H</i> -Perfluorooctanoic acid	5:3FTCA	914637-49-3	341.00	237.00	6.33
Perfluoro- <i>n</i> -heptanoic acid	PFHpA	375-85-9	362.93	318.80	6.45
Perfluoro- <i>n</i> -hexanesulfonic acid	PFHxS	355-46-4	398.94	79.80	6.49
4,8-Dioxa-3 <i>H</i> -perfluorononanoic acid	ADONA	919005-14-4	376.90	250.70	6.58
1 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorooctane sulfonic acid	6:2FTS	27619-97-2	426.93	406.90	7.24
Perfluoro- <i>n</i> -heptanesulfonic acid	PFHpS	375-92-8	448.93	79.80	7.26
Perfluoro- <i>n</i> -octanoic acid	PFOA	335-67-1	412.91	369.00	7.26
Perfluoro- <i>n</i> -octanesulfonic acid	PFOS	1763-23-1	498.84	79.90	7.89
Perfluoro- <i>n</i> -nonanoic acid	PFNA	375-95-1	462.89	418.90	7.92
3-Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4	441.00	317.00	8.00
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	73606-19-6	530.75	350.70	8.25
Perfluorononanesulfonic acid	PFNS	68259-12-1	548.81	79.90	8.45
Perfluoro- <i>n</i> -decanoic acid	PFDA	335-76-2	512.84	468.90	8.49
1 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorodecane sulfonic acid	8:2FTS	39108-34-4	526.00	506.80	8.50
<i>N</i> -methyl perfluorooctanesulfonamidoacetic acid	<i>N</i> -MeFOSAA	2355-31-9	569.80	418.90	8.78
Perfluorooctanesulfonamide	FOSA	754-91-6	497.87	77.80	8.84
Perfluoro- <i>n</i> -decanesulfonic	PFDS	335-77-3	598.79	79.90	8.90
Perfluoro- <i>n</i> -undecanoic acid	PFUnDA	2058-94-8	562.80	518.90	8.95
<i>N</i> -ethyl perfluorooctanesulfonamidoacetic acid	<i>N</i> -EtFOSAA	2991-50-6	583.81	418.80	9.02
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9	630.74	451.00	9.15
Perfluoro- <i>n</i> -dodecanoic acid	PFDoDA	307-55-1	612.79	568.90	9.33
<i>N</i> -methyl perfluorooctanesulfonamide	<i>N</i> -MeFOSA	31506-32-8	512.00	169.00	9.55
Perfluoro- <i>n</i> -tridecanoic acid	PFTriDA	72629-94-8	662.77	618.90	9.66
Perfluorododecanesulfonic acid	PFDoS	79780-39-5	698.72	80.00	9.70
<i>N</i> -ethyl perfluorooctanesulfonamide	<i>N</i> -EtFOSA	4151-50-2	526.00	169.00	9.80
<i>N</i> -methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7	616.00	59.00	9.82
Perfluoro- <i>n</i> -tetradecanoic acid	PFTeDA	376-06-7	712.77	668.80	9.94
<i>N</i> -ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1961-99-2	630.00	59.00	10.04
Surrogates					
Perfluoro-(2,3,4- ¹³ C ₃)butanoic acid	M3PFBA		216.00	172.00	1.91
Perfluoro-(¹³ C ₄)butanoic acid	M4PFBA		216.94	171.90	2.01
Perfluoro-(¹³ C ₅)pentanoic acid	M5PFPeA		267.97	222.90	3.92
Sodium perfluoro-(2,3,4- ¹³ C ₃)butanesulfonate	M3PFBS		301.89	98.90	4.22

Analysis of PFAS in Aqueous Samples according to EPA Draft Method 1633

Analyte	Abbreviation	CAS number	Q1 mass [Da]	Q3 mass [Da]	Retention time [min]
Perfluoro-(1,2- ¹³ C ₂)hexanoic acid	MPFHxA		315.00	270.00	5.05
Sodium 1 <i>H</i> ,1 <i>H</i> ,2 <i>H</i> ,2 <i>H</i> -perfluoro(1,2- ¹³ C ₂)hexanesulfonate	M2-4:2FTS		328.97	81.00	5.26
Perfluoro-(1,2,3,4,6- ¹³ C ₅)hexanoic acid	M5PFHxA		318.00	272.80	5.40
Tetrafluoro-2-heptafluoropropoxy- ¹³ C ₃ -propanoic acid	M3HPFO-DA		287.00	169.00	5.44
Perfluoro-1-hexane(¹⁸ O ₂)sulfonic acid	MPFHxS		403.00	103.00	6.28
Perfluoro-(1,2,3,4- ¹³ C ₄)heptanoic acid	M4PFHpA		366.95	321.80	6.45
Sodium perfluoro-(1,2,3- ¹³ C ₃)hexanesulfonate	M3PFHxS		401.90	79.90	6.50
Perfluoro-(1,2,3,4- ¹³ C ₄)octanoic acid	MPFOA		417.00	372.00	7.08
Sodium 1 <i>H</i> ,1 <i>H</i> ,2 <i>H</i> ,2 <i>H</i> -perfluoro(1,2- ¹³ C ₂)octanesulfonate	M2-6:2FTS		428.94	81.00	7.23
Perfluoro-(¹³ C ₈)octanoic acid	M8PFOA		420.95	376.00	7.27
Perfluoro-(1,2,3,4,5- ¹³ C ₅) nonanoic acid	MPFNA		468.00	423.00	7.77
Perfluoro-(1,2,3,4- ¹³ C ₄)octanesulfonic acid	MPFOS		503.00	99.00	7.78
Sodium perfluoro-(¹³ C ₈)octanesulfonate	M8PFOS		506.91	98.90	7.89
Perfluoro-(¹³ C ₉)nonanoic acid	M9PFNA		471.94	427.00	7.92
Perfluoro-(1,2- ¹³ C ₂)decanoic acid	MPFDA		515.00	470.00	8.36
Perfluoro-(1,2,3,4,5,6- ¹³ C ₆)decanoic acid	M6PFDA		518.92	474.00	8.49
Sodium 1 <i>H</i> ,1 <i>H</i> ,2 <i>H</i> ,2 <i>H</i> -perfluoro(1,2- ¹³ C ₂)decanesulfonate	M2-8:2FTS		528.94	80.90	8.50
<i>N</i> -methyl-d ₃ -perfluorooctanesulfonamidoacetic acid	d3- <i>N</i> -MeFOSAA		572.89	419.00	8.78
Perfluoro-(¹³ C ₈)octanesulfonamide	M8FOSA		505.98	77.90	8.84
Perfluoro-(1,2,3,4,5,6,7- ¹³ C ₇)undecanoic acid	M7PFUdA		569.95	525.00	8.95
<i>N</i> -ethyl-d ₅ -perfluorooctanesulfonamidoacetic acid	d5- <i>N</i> -EtFOSAA		588.85	418.80	9.02
Perfluoro-(1,2- ¹³ C ₂)dodecanoic acid	MPFDoA		614.95	569.90	9.33
<i>N</i> -methyl-d ₃ -perfluoro-1-octanesulfonamide	d3- <i>N</i> -MeFOSA		515.00	169.00	9.77
<i>N</i> -methyl-d ₇ -perfluorooctanesulfonamidoethanol	d7- <i>N</i> -MeFOSE		623.00	59.00	9.80
Perfluoro-(1,2- ¹³ C ₂)tetradecanoic acid	M2PFTeDA		714.94	670.00	9.94
<i>N</i> -ethyl-d ₅ -perfluoro-1-octanesulfonamide	d5- <i>N</i> -EtFOSA		531.00	169.00	10.02
<i>N</i> -ethyl-d ₉ -perfluorooctanesulfonamidoethanol	d9- <i>N</i> -EtFOSE		639.00	59.00	10.03

Table 2: MRM transitions and retention times of native PFAS and isotopically labeled PFAS analytical standards.

Chromatogramms

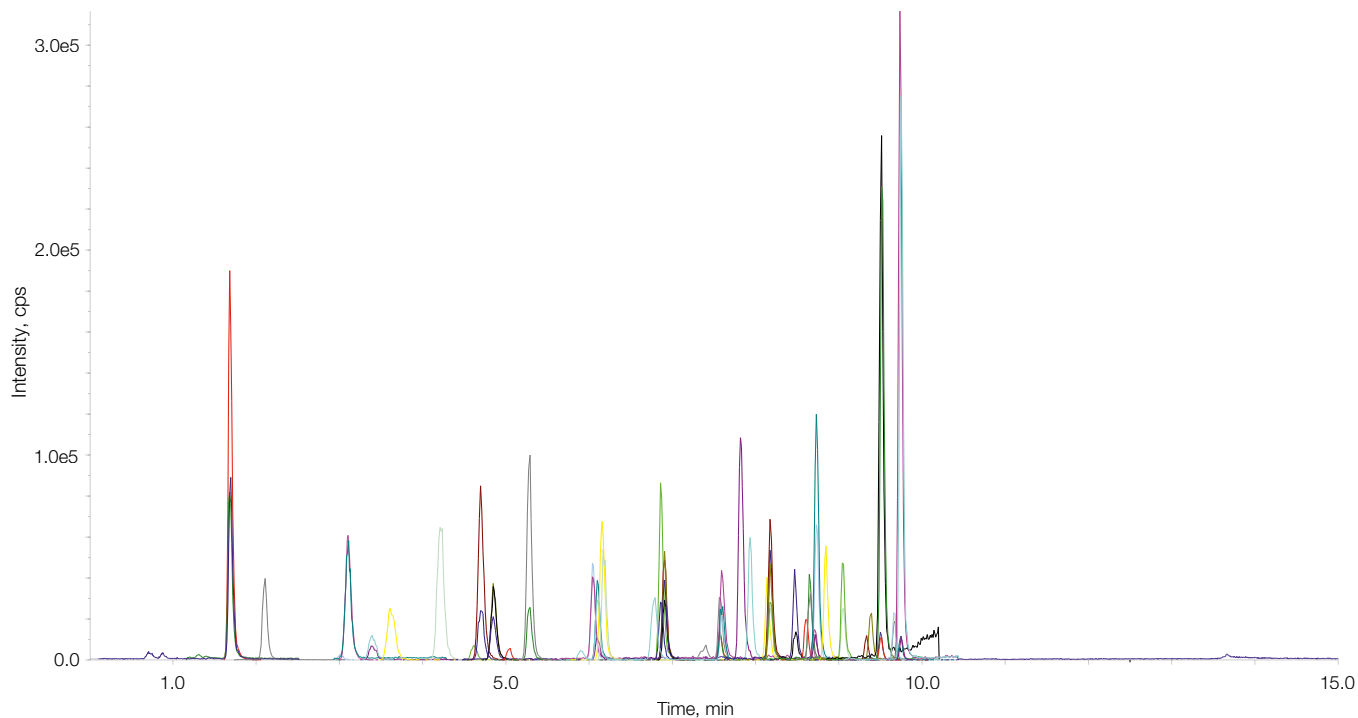


Figure 2: Chromatogram of a standard solution (concentration, $\beta = 0.5$ ng/mL).

Recovery rates

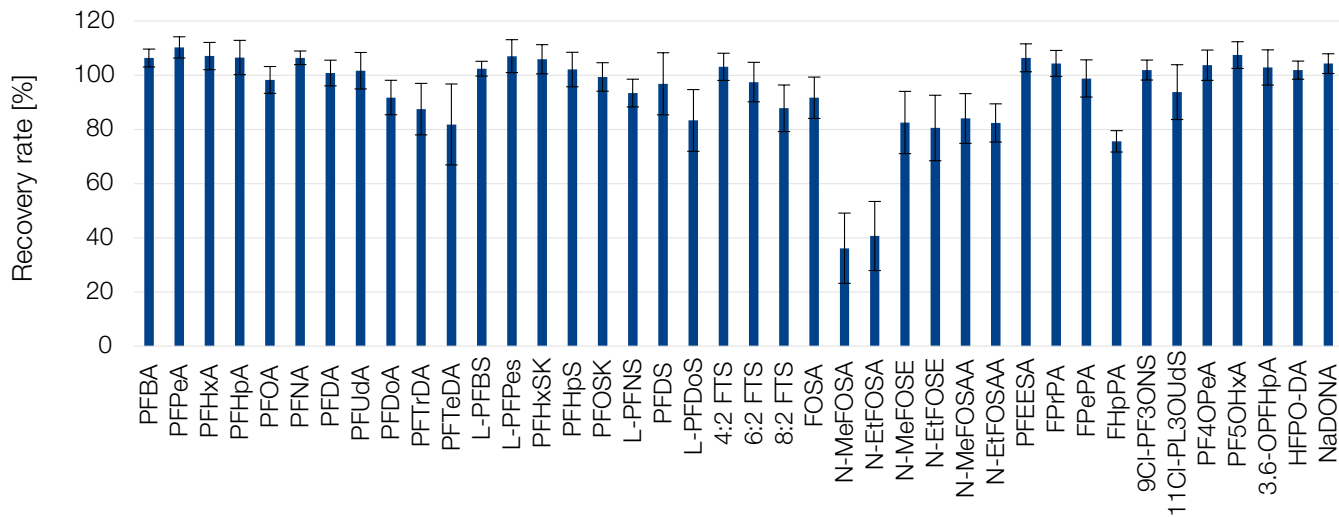


Figure 3: Recovery rate from water sample (concentration, $\beta = 5$ ng/L, n=5)

Analysis of PFAS in Aqueous Samples according to EPA Draft Method 1633

Analyte	Abbreviation	Recovery rate (%) ± RSD (%)
Perfluoro-3-methoxypropanoic acid	PFMPA	103.7 ± 5.6
Perfluoro- <i>n</i> -butanoic acid	PFBA	106.3 ± 3.3
3-Perfluoropropyl propanoic acid	3:3FTCA	104.3 ± 4.8
Perfluoro-4-methoxybutanoic acid	PFMBA	107.4 ± 4.9
Perfluoro- <i>n</i> -pentanoic acid	PFPeA	110.3 ± 3.9
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	106.4 ± 5.2
Perfluoro- <i>n</i> -butanesulfonic acid	PFBS	102.4 ± 2.7
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	102.9 ± 6.5
1 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorohexane sulfonic acid	4:2FTS	103.1 ± 5.0
Perfluoro- <i>n</i> -hexanoic acid	PFHxA	107.1 ± 5.0
Perfluoropentanesulfonic acid	PFPeS	107.0 ± 6.1
Hexafluoropropylene oxide dimer acid	HFPO-DA	101.9 ± 3.4
2 <i>H</i> ,2 <i>H</i> ,3 <i>H</i> ,3 <i>H</i> -Perfluorooctanoic acid	5:3FTCA	98.8 ± 6.9
Perfluoro- <i>n</i> -heptanoic acid	PFHpA	106.5 ± 6.3
Perfluoro- <i>n</i> -hexanesulfonic acid	PFHxS	105.9 ± 5.4
4,8-Dioxa-3 <i>H</i> -perfluorononanoic acid	ADONA	104.3 ± 3.6
1 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorooctane sulfonic acid	6:2FTS	97.5 ± 7.3
Perfluoro- <i>n</i> -heptanesulfonic acid	PFHpS	102.1 ± 6.4
Perfluoro- <i>n</i> -octanoic acid	PFOA	98.2 ± 5.0
Perfluoro- <i>n</i> -octanesulfonic acid	PFOS	99.3 ± 5.3
Perfluoro- <i>n</i> -nonanoic acid	PFNA	106.4 ± 2.5
3-Perfluoroheptyl propanoic acid	7:3FTCA	75.6 ± 3.9
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	101.9 ± 3.7
Perfluorononanesulfonic acid	PFNS	93.4 ± 5.1
Perfluoro- <i>n</i> -decanoic acid	PFDA	100.8 ± 4.7
1 <i>H</i> ,1 <i>H</i> , 2 <i>H</i> , 2 <i>H</i> -Perfluorodecane sulfonic acid	8:2FTS	87.8 ± 8.6
<i>N</i> -methyl perfluorooctanesulfonamidoacetic acid	<i>N</i> -MeFOSAA	84.0 ± 9.2
Perfluorooctanesulfonamide	FOSA	91.7 ± 7.6
Perfluoro- <i>n</i> -decanesulfonic acid	PFDS	96.8 ± 11.5
Perfluoro- <i>n</i> -undecanoic acid	PFUnDA	101.7 ± 6.7
<i>N</i> -ethyl perfluorooctanesulfonamidoacetic acid	<i>N</i> -EtFOSAA	82.4 ± 7.0
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	98.8 ± 10.1
Perfluoro- <i>n</i> -dodecanoic acid	PFDoDA	91.8 ± 6.4
<i>N</i> -methyl perfluorooctanesulfonamide	<i>N</i> -MeFOSA	36.1 ± 13.0
Perfluoro- <i>n</i> -tridecanoic acid	PFTrDA	87.5 ± 9.5
Perfluorododecanesulfonic acid	PFDoS	83.3 ± 11.4
<i>N</i> -ethyl perfluorooctanesulfonamide	<i>N</i> -EtFOSA	40.7 ± 12.7
<i>N</i> -methyl perfluorooctanesulfonamidoethanol	NMeFOSE	82.5 ± 11.5
Perfluoro- <i>n</i> -tetradecanoic acid	PFTeDA	81.8 ± 14.9
<i>N</i> -ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	80.5 ± 12.1
Surrogates		
Perfluoro-(2,3,4- ¹³ C ₃)butanoic acid	M3PFBA	104.3 ± 1.0
Perfluoro-(¹³ C ₄)butanoic acid	M4PFBA	106.4 ± 2.7
Perfluoro-(¹³ C ₅)pentanoic acid	M5PFPeA	113.4 ± 2.4
Sodium perfluoro-(2,3,4- ¹³ C ₃)butanesulfonate	M3PFBS	106.5 ± 1.9
Perfluoro-(1,2- ¹³ C ₂)hexanoic acid	MPFHxA	108.1 ± 5.7
Sodium 1 <i>H</i> ,1 <i>H</i> ,2 <i>H</i> ,2 <i>H</i> -perfluoro(1,2- ¹³ C ₂)hexanesulfonate	M2-4:2FTS	102.9 ± 1.7
Perfluoro-(1,2,3,4,6- ¹³ C ₅)hexanoic acid	M5PFHxA	101.8 ± 1.8
Tetrafluoro-2-heptafluoropropoxy- ¹³ C ₃ -propanoic acid	M3HPFO-DA	105.5 ± 3.3

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Analyte	Abbreviation	Recovery rate (%) ± RSD (%)
Perfluoro-1-hexane(¹⁸ O ₂)sulfonic acid	MPFHxS	105.0 ± 1.5
Perfluoro-(1,2,3,4- ¹³ C ₄)heptanoic acid	M4PFHpA	106.4 ± 2.4
Sodium perfluoro-(1,2,3- ¹³ C ₃)hexanesulfonate	M3PFHxS	108.0 ± 2.7
Perfluoro-(1,2,3,4- ¹³ C ₄)octanoic acid	MPFOA	104.5 ± 6.5
Sodium 1 <i>H</i> ,1 <i>H</i> ,2 <i>H</i> ,2 <i>H</i> -perfluoro(1,2- ¹³ C ₂)octanesulfonate	M2-6:2FTS	98.3 ± 3.8
Perfluoro-(¹³ C ₈)octanoic acid	M8PFOA	107.4 ± 1.0
Perfluoro-(1,2,3,4,5- ¹³ C ₅)nonanoic acid	MPFNA	104.9 ± 1.3
Perfluoro-(1,2,3,4- ¹³ C ₄)octanesulfonic acid	MPFOS	103.4 ± 2.4
Sodium perfluoro-(¹³ C ₈)octanesulfonate	M8PFOS	101.0 ± 0.8
Perfluoro-(¹³ C ₉)nonanoic acid	M9PFNA	107.8 ± 1.0
Perfluoro-(1,2- ¹³ C ₂)decanoic acid	MPFDA	98.5 ± 3.2
Perfluoro-(1,2,3,4,5,6- ¹³ C ₆)decanoic acid	M6PFDA	99.7 ± 4.2
Sodium 1 <i>H</i> ,1 <i>H</i> ,2 <i>H</i> ,2 <i>H</i> -perfluoro(1,2- ¹³ C ₂)decanesulfonate	M2-8:2FTS	89.9 ± 4.5
<i>N</i> -methyl-d ₃ -perfluorooctanesulfonamidoacetic acid	d3- <i>N</i> -MeFOSAA	85.8 ± 3.9
Perfluoro-(¹³ C ₈)octanesulfonamide	M8FOSA	92.8 ± 8.1
Perfluoro-(1,2,3,4,5,6,7- ¹³ C ₇)undecanoic acid	M7PFUdA	104.0 ± 3.5
<i>N</i> -ethyl-d ₅ -perfluorooctanesulfonamidoacetic acid	d5- <i>N</i> -EtFOSAA	90.9 ± 7.3
Perfluoro-(1,2- ¹³ C ₂)dodecanoic acid	MPFD _o A	93.9 ± 7.7
<i>N</i> -methyl-d ₃ -perfluoro-1-octanesulfonamide	d3- <i>N</i> -MeFOSA	90.9 ± 7.3
<i>N</i> -methyl-d ₇ -perfluorooctanesulfonamidoethanol	d7- <i>N</i> -MeFOSE	86.3 ± 9.1
Perfluoro-(1,2- ¹³ C ₂)tetradecanoic acid	M2PFTeDA	88.3 ± 13.0
<i>N</i> -ethyl-d ₅ -perfluoro-1-octanesulfonamide	d5- <i>N</i> -EtFOSA	43.9 ± 12.0
<i>N</i> -ethyl-d ₉ -perfluorooctanesulfonamidoethanol	d9- <i>N</i> -EtFOSE	85.4 ± 11.6

Recovery rates for the presented SPE method using CHROMABOND® WAX, 150 mg, 6 mL, n=5.

Conclusion

This application note presents the reliable and successful determination of 40 PFAS according to EPA Draft 1633 from drinking water. By using the SPE column, CHROMABOND® WAX, it was possible to achieve high recovery rates for with good reproducibility especially for short chain PFAS. CHROMABOND® WAX was optimized for PFAS analysis and provides various strong ionic interaction types like ionic, hydrophobic, hydrogen bonds and dipole-dipole interactions for the enrichment of a broad spectrum of PFAS. The sorbent is specially recommended for PFAS analysis because of its very low blind value levels. Most of the PFAS show recovery rates between 80 % to 110 %.

The neutralization of the eluate leads to very high recovery rates for neutral substances such as FOSA, *N*-MeFOSE and *N*-EtFOSE. Without the enrichment of sample concentration by eluent exchange, the analysis requires LC-MSMS systems with very good performance and high sensitivity. The membrane filtration was done without PFAS losses using a syringe filter with nylon membrane (25 mm filter, 0.2 µm). The identification and the quantification of PFAS in water were finally carried out by ESI mass spectrometry on a NUCLEODUR® PFAS column.

References

- [1] Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS; August 2021.
- [2] National Pollutant Discharge Elimination System (NPDES), 832-F-12-033.NTP
- [3] MONOGRAPH ON IMMUNOTOXICITY ASSOCIATED WITH EXPOSURE TO PERFLUOROOCTANOIC ACID (PFOA) OR PERFLUOROOCTANE SULFONATE (PFOS), September, 2016, Office of Health Assessment and Translation Division of the National Toxicology Program National Institute of Environmental Health Sciences National Institutes of Health.

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