Analysis of long-chain PFASs in biological sample matrix

PFASs are a group of synthetic organofluorine chemical compounds used in the production of fluoropolymer coatings and products such as furniture, adhesives, personal care products, food packaging, mobile phone screens, etc. Many PFASs pose health and environmental concerns because they are classified as persistent organic pollutants. The long-chain PFASs are due to higher bioaccumulative potential frequently detected in living organisms. This application shows a robust method of extraction and determination of the PFAS compounds in fish biota samples, which can be a relevant exposure pathway for humans.

MSPE with 15 mg of C18 stationary phase and nylon frits was used to analyze 14 PFAS compounds in fish tissue samples. The MSPE sample preparation approach was compared with the reference extraction method without SPE based on Grabicova et al. 2018 [1]. Both sets of samples were fortified to 1 ng/g of PFAS. The MSPE method enhances the response of most of the analytes between 80–130 % (Fig. 1). Although sample preparation is more demanding than the reference method, the MSPE offers relevant signal improvement.

References

[1] Grabicova K, Vojs Staňová A, Koba Ucun O, Borik A, Randak T, Grabic R. Development of a robust extraction procedure for the HPLC-ESI-HRPS determination of multi-residual pharmaceuticals in biota samples. Anal Chim Acta. 2018 Aug 31;1022:53-60. doi: 10.1016/j.aca.2018.04.011. Epub 2018 Apr 18. PMID: 29729738.

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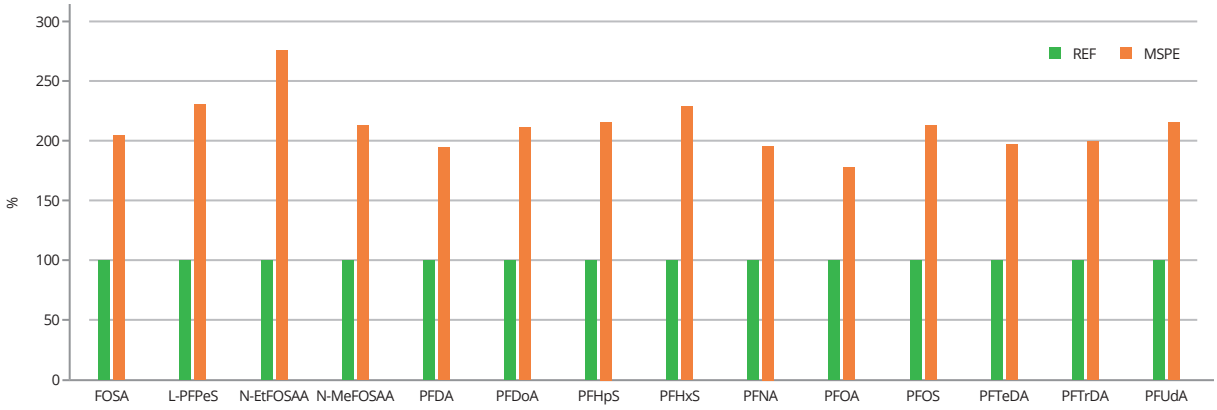
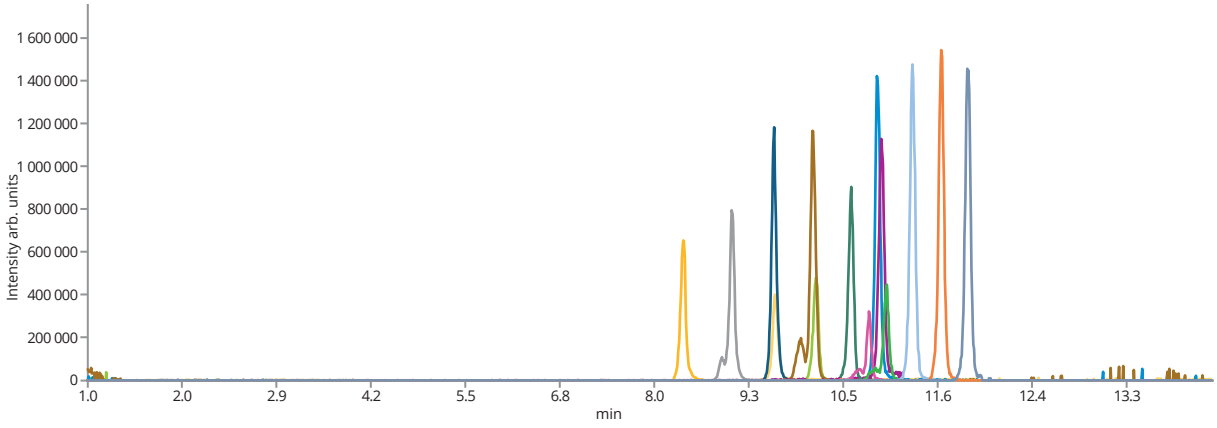


Fig 1. MSPE method relative signal improvement in comparison to the reference extraction method without SPE



Mixture of PFASs on ASTRA® column

This application was developed in cooperation with the Faculty of Fisheries and Protection of Waters, Research Institute of Fish Culture and Hydrobiology, Laboratory of Environmental Chemistry and Biochemistry.

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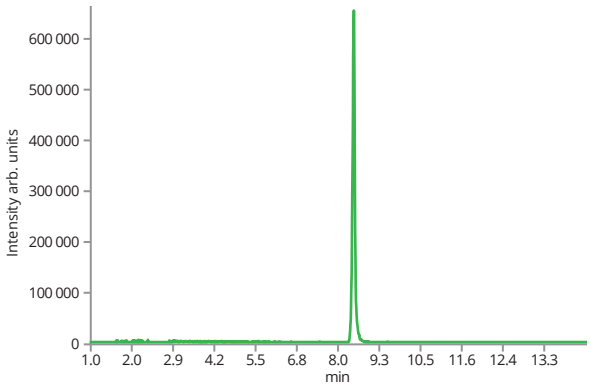
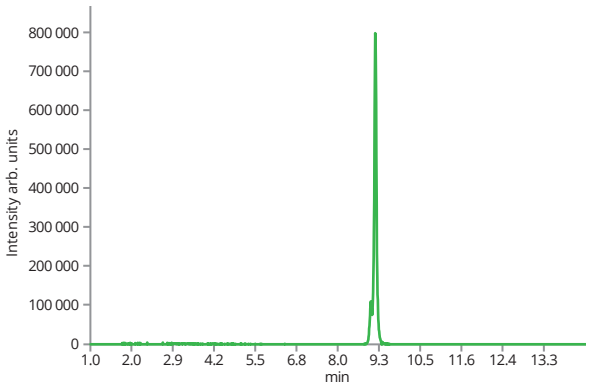
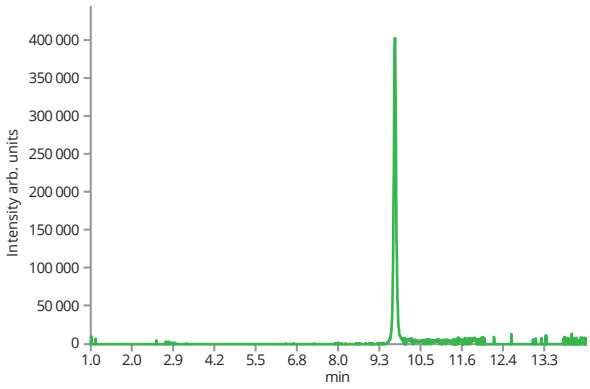


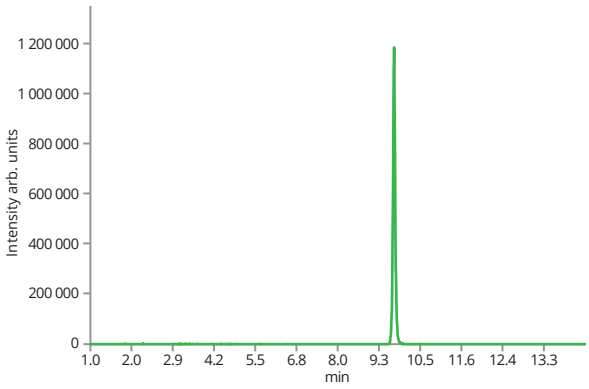
L-PFPeS RT: 8.44, m/z 348.9398



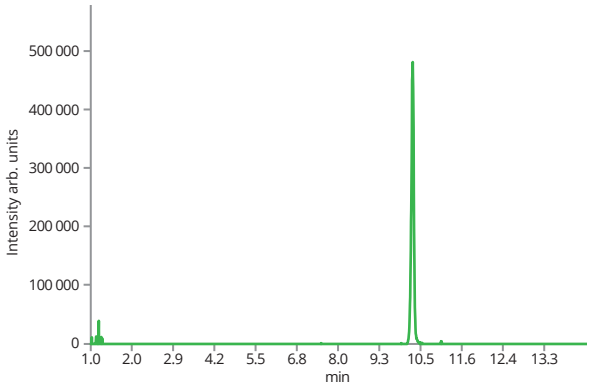
PFHxS RT: 9.09, m/z 398.9366



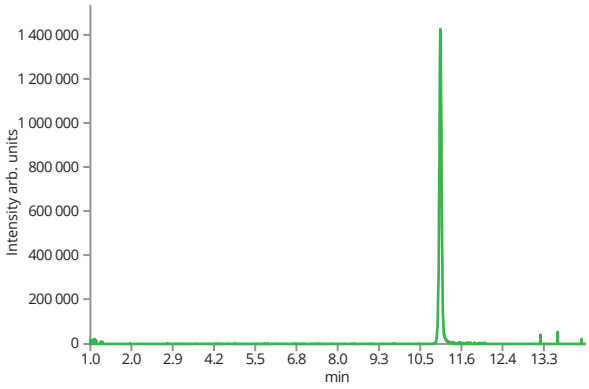
PFOA RT: 9.68, m/z 412.9664



PFHpS RT: 9.66, m/z 448.9334



PFNA RT: 10.17, m/z 462.9632



FOSA RT: 10.93, m/z 497.9462

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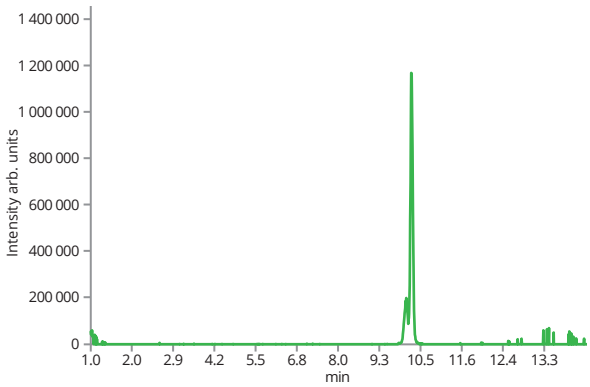
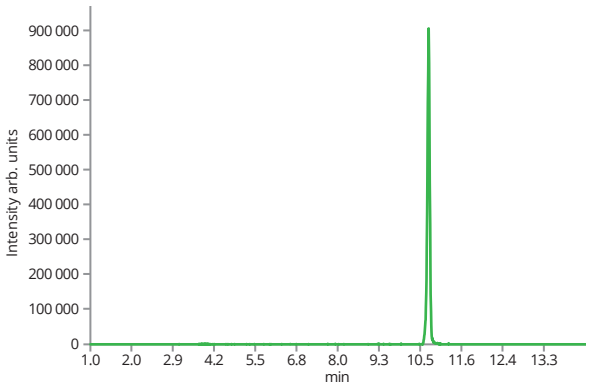
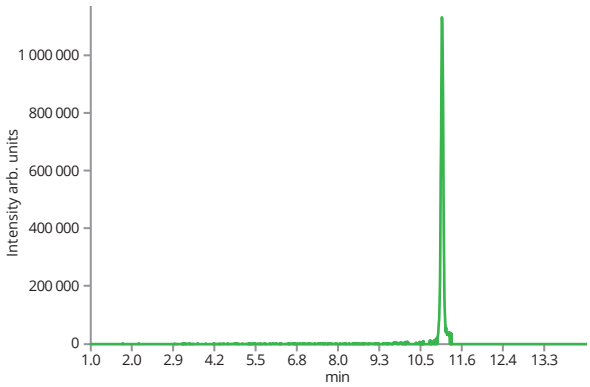


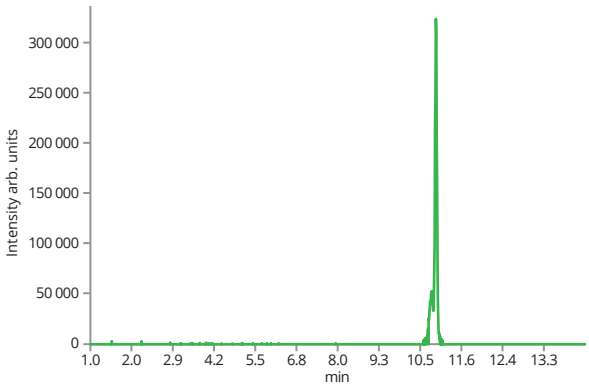
PFOS RT: 10.13, m/z 498.9302



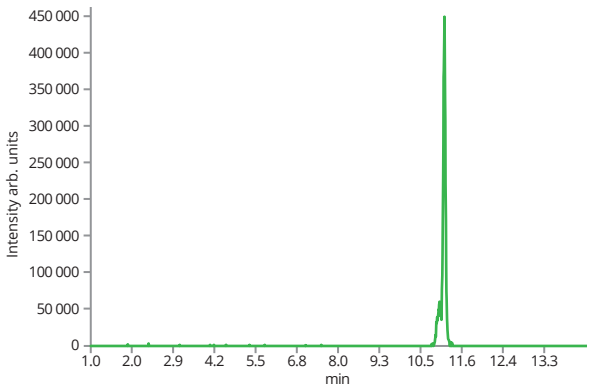
PFDA RT: 10.6, m/z 512.960



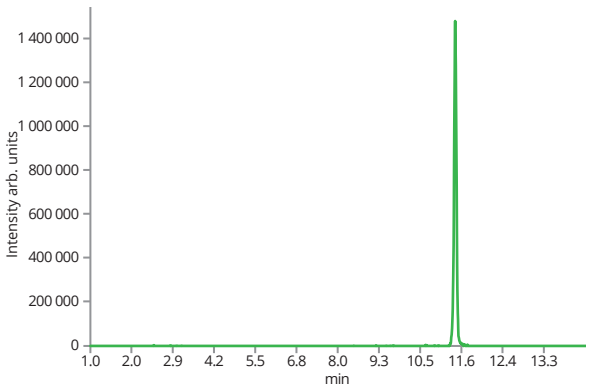
PFUdA RT: 10.9, m/z 562.9568



N-MeFOSAA RT: 10.9, m/z 569.9673



N-EtFOSAA RT: 11.03, m/z 583.9829



PFDoA RT: 11.3, m/z 612.9536

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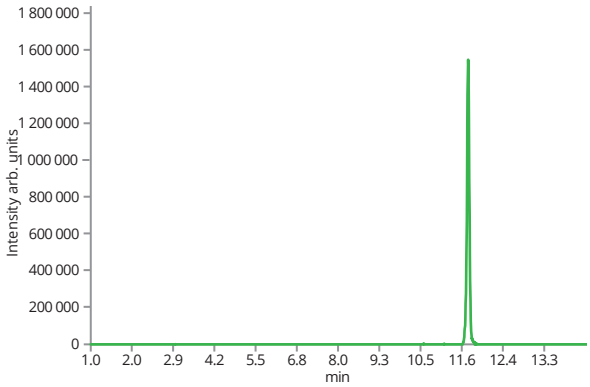
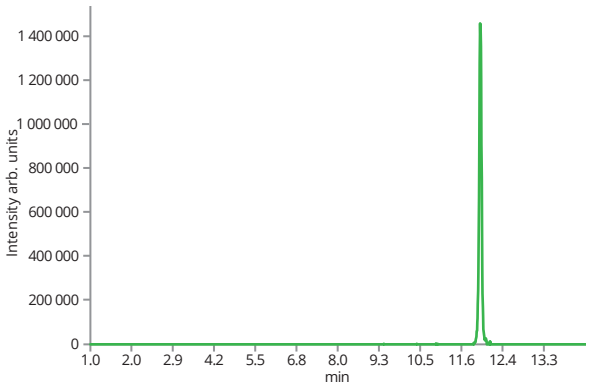
Analysis of long-chain PFASs in biological sample matrix



PFTrDA RT: 11.6, m/z 662.9505



PFTeDA RT: 11.85, m/z 712.9472

MSPE Method

MSPE Column	Micro Spin SpeExtra™ MSPE C18 column with 0.22 µm Nylon membrane, 15 mg
Homogenization	TissueLyser II, Quiagen, Germany, 1800 min^{-1} for 10 min
Extraction	ACN + 0.1% FA
Centrifugation	Mini spin, Eppendorf, Germany; 4000 rpm for 2 min
Reconstitution	DDW + 0.1% FA
Dissolution	Ultrasonic bath 5 min
MSPE Activation	DDW + MeOH (50:50)
MSPE Equilibration	DDW + 0.1% FA
MSPE Extraction of analytes	500 µL of sample
MSPE Elution of analytes	200 µL ACN + 0.1% FA

Analysis of long-chain PFASs in biological sample matrix

LC-HRMS Method

Column	ASTRA® C18-HE, 3 µm			
Dimensions	75 mm × 2.1 mm			
Part number	AST-5732-IH21			
Mobile phase	A: DDW + 10mM Ammonium Acetate B: Methanol + 10mM Ammonium Acetate			
Gradient elution	**Time**	**A (%)**	**B (%)**	**Flow rate (µL/min)**
	0	90	10	350
	0.5	90	10	350
	6	40	60	350
	11	0	100	400
	14	0	100	400
	14.01	90	10	350
	16	90	10	350
Temperature	23 °C			
Injection volume	10 µL			
Detection	Full scan (resolving power 120 000, m/z 200), negative mode			

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Analysis of long-chain PFASs in biological sample matrix

Analytes

1. L-PFPeS (perfluoropentanesulfonic acid),
CAS number 2706-91-4
2. PFHxS (perfluorohexanesulfonic acid),
CAS number 355-46-4
3. PFOA (perfluorooctanoic acid),
CAS number 335-67-1
4. PFHpS (perfluoroheptanesulfonic acid),
CAS number 375-92-8
5. PFNA (perfluorononanoic acid),
CAS number 375-95-1
6. FOSA (perfluorooctanesulfonamide),
CAS number 754-91-6
7. PFOS (perfluorooctanesulfonic acid),
CAS number 1763-23-1
8. PFDA (perfluorodecanoic acid),
CAS number 335-76-2
9. PFUdA (perfluoroundecanoic acid),
CAS number 2058-94-8
10. N-MeFOSAA
(2-(N-methylperfluorooctanesulfonamido)acetic acid),
CAS number 2355-31-9
11. N-EtFOSAA
(N-Ethyl-N-[(heptadecafluorooctyl)sulphonyl]glycine),
CAS number 2991-50-6
12. PFDoA (perfluorododecanoic acid),
CAS number 307-55-1
13. PFTrDA (perfluorotridecanoic acid),
CAS number 72629-94-8
14. PFTeDA (perfluorotetradecanoic acid),
CAS number 376-06-7

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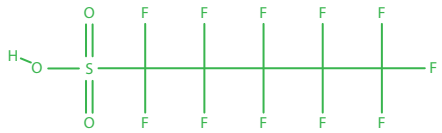
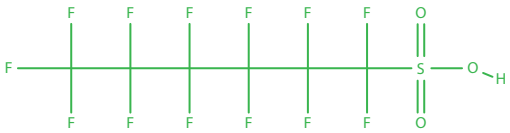
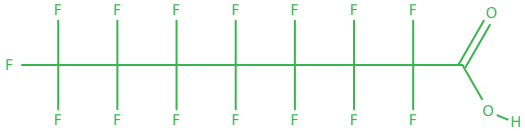


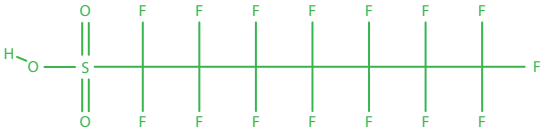
L-PFPeS



PFHxS



PFOA



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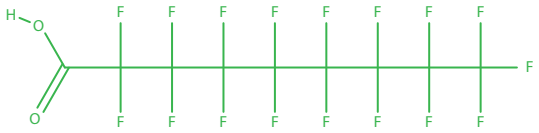
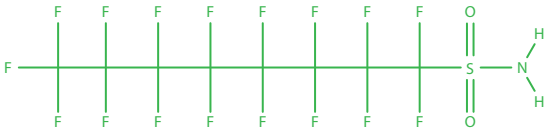
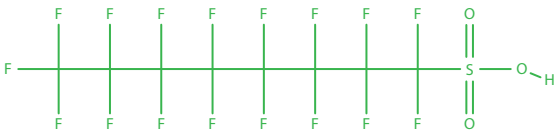


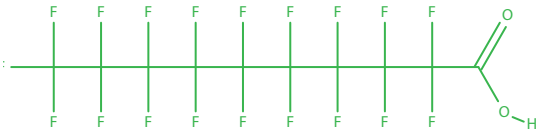
PFNA



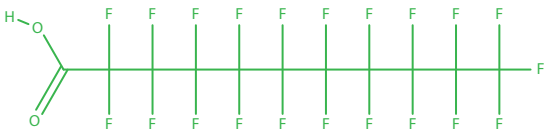
FOSA



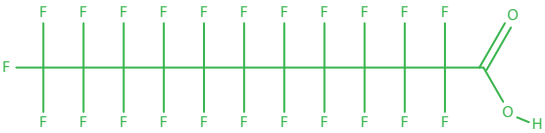
PFOS



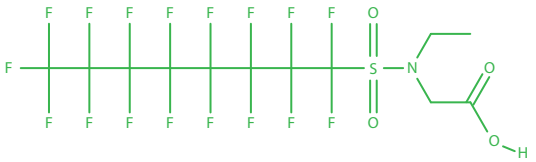
PFDA



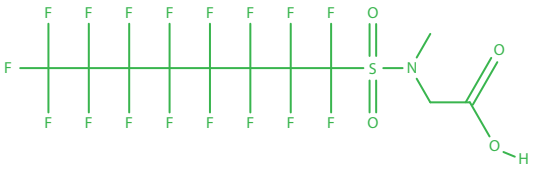
PFUdA



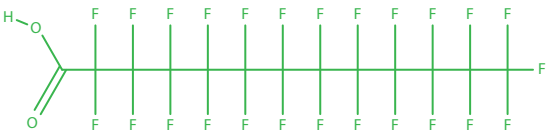
PFDoA



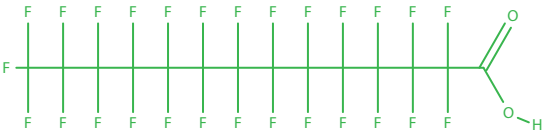
N-EtFOSAA



N-MeFOSAA



PFTrDA



PFTeDA