MACHEREY-NAGEL
Chromatography application note

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PFAS Analysis According to EPA 533 and to EPA 537.1

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Abstract

This application note describes the determination of native per- and polyfluoroalkyl substances (PFAS) according to EPA directives 533 and 537.1. It demonstrates the chromatographic separation of PFAS using a NUCLEODUR® PFAS analytical HPLC column in combination with a NUCLEODUR® PFAS Delay column.

Introduction

Per- and polyfluoroalkyl substances (PFAS) belong to a large group of manufactured chemicals which are very persistent in the environment and which have been used as additives in consumer products like fire-fighting foam, fiber coating, cookware, paper finishing, food packaging (e. g. pizza cartons, paper cups), building material, (e. g. water resistant lacquer). These anthropogenic pollutants are characterized by a linear aliphatic backbone, a high degree of fluorination and often feature a carboxylic or sulfonic acid functionality and have the potential to accumulate in the environment and impact the food chain, affecting fish, birds, livestock, and humans.

PFAS present entail numerous analytical challenges, including their widespread presence in a variety of environmental samples, occurrence of isomers for some compounds and precursor transformations that may occur during preservation and storage of the samples. There is also evidence that exposure to PFAS can lead to adverse human health effects.

This is the reason why authorities in the US published variety of laws and regulations to protect public health and the environment [1]:

- Safe Drinking Water Act
- Toxic Substances Control Act (TSCA)
- Comprehensive Environmental Response, Compensation and Liability Act
- Clean Air Act

To protect environment and human health, the environmental protection agency (EPA) has published an action plan for identification and for understanding PFAS, e.g. new approaches to address current PFAS contamination, to prevent future contamination and to effectively communicate with the public about PFAS [2].

There is a need of robust and fast analytical methods to ensure accurate quantification of low levels. Therefore, EPA recommends two methods to detect PFAS from drinking water by solid phase extraction (SPE) and liquid chromatography/tandem mass spectrometry (LC/MS-MS) [3, 4].

This work presents the analysis of PFAS according to directives EPA method 533 and 537.1. It represents the chromatographic separation of PFAS using a NUCLEODUR® PFAS analytical HPLC column in combination with a NUCLEODUR® PFAS Delay column.

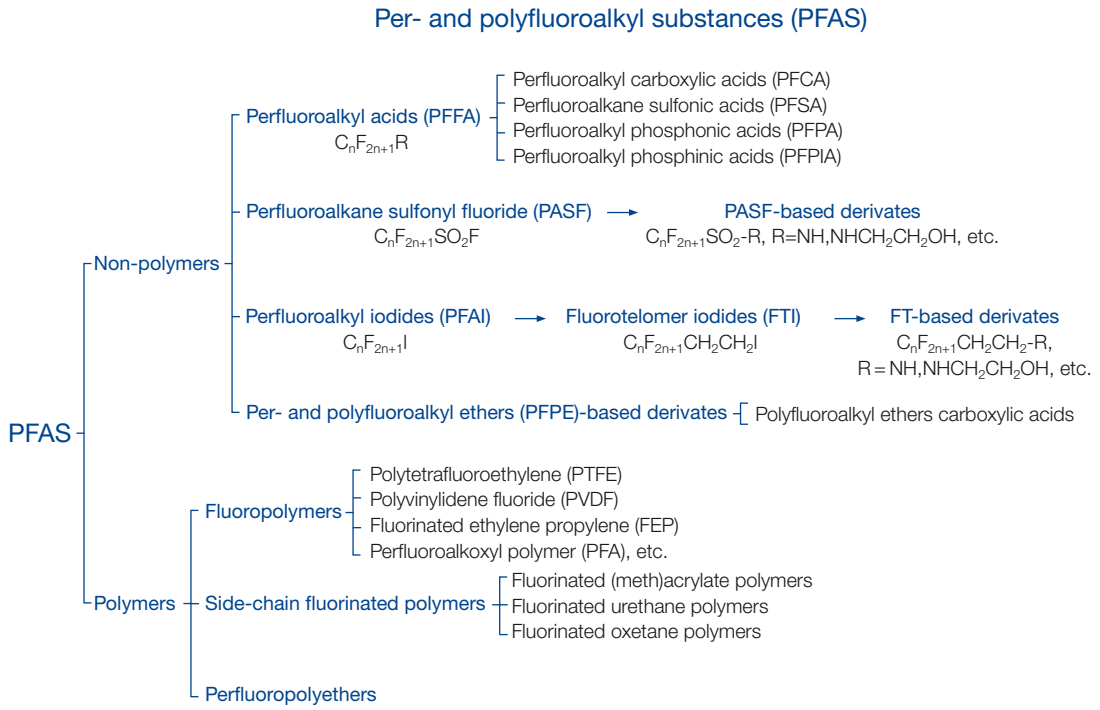

Figure 1: Classes of per- and polyfluoroalkyl substances (PFAS).

PFAS Analysis According to EPA 533 and to EPA 537.1

Analysis by HPLC-MS / MS

A: Chromatographic conditions
NUCLEODUR® PFAS, EC 50 x 2 mm, 3 µm

Delay column:	EC 50/2 NUCLEODUR® PFAS Delay , 5 µm (REF 760673.20)
Analytical column:	EC 50/2 NUCLEODUR® PFAS, 3 µm (REF 760663.20)
Eluent A:	5 mM ammonium acetate in water
Eluent B:	5 mM ammonium acetate in methanol
Gradient:	hold 40 % B for 0.5 min, in 4 min from 40 % B to 95 % B, hold 95 % B for 1.5 min, in 0.05 min to 40 % B, hold 40 % B for 1.45 min
Flow rate:	0.3 mL/min
Temperature:	40 °C
Injection volume:	2 µL
Sample solution:	Mixture of PFAS in methanol, concentration 1 ng/mL for each compound

B: Chromatographic conditions
NUCLEODUR® PFAS, EC 100 x 2 mm, 3 µm

Delay column:	EC 50/2 NUCLEODUR® PFAS Delay , 5 µm (REF 760673.20)
Analytical 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 1.0 min, in 8 min from 40 % B to 95 % B, hold 95 % B for 3.0 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
Sample solution:	Mixture of PFAS in methanol, concentration 1 ng/mL for each compound

MS conditions:

AB Sciex QTRAP 5500

Acquisition mode:	SRM	Ion spray voltage:	– 4500 V
Interface:	ESI	Temperature:	400 °C
Polarity:	negative	Ion source gas 1:	50 psig
Curtain gas:	30 psig	Ion source gas 2:	60 psig
Collision Gas:	medium	Detection window:	60 s

MRM transitions

Analyte	Abbreviation	CAS number	Q_1 mass [Da]	Q_3 mass [Da]	Retention time [min] A	Retention time [min] B
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	284.9	168.7	3.26	5.77
Perfluorobutanesulfonic acid	PFBS	375-73-5	298.9	98.9	2.37	4.20
Perfluorohexanoic acid	PFHxA	307-24-4	312.9	268.8	3.05	5.40
Perfluoroheptanoic acid	PFHpA	375-85-9	362.9	318.8	3.60	6.45
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4	376.9	250.7	3.66	6.58
Perfluorohexanesulfonic acid	PFHxS	355-46-4	398.9	79.8	3.62	6.49
Perfluorooctanoic acid	PFOA	335-67-1	412.9	369.0	3.99	7.26
Perfluorononanoic acid	PFNA	375-95-1	462.9	418.9	4.31	7.92
Perfluorooctanesulfonic acid	PFOS	1763-23-1	498.8	79.9	4.30	7.89
Perfluorodecanoic acid	PFDA	335-76-2	512.8	468.9	4.58	8.49
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF_3ONS	756426-58-1	530.8	350.7	4.48	8.25
Perfluoroundecanoic acid	PFUnA	2058-94-8	562.8	518.9	4.81	8.95
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9	569.8	418.9	4.71	8.78
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6	583.8	418.8	4.40	9.02
Perfluorododecanoic acid	PFDoA	307-55-1	612.8	568.9	5.02	9.33
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF_3OUdS	763051-92-9	630.7	451.0	4.93	9.15
Perfluorotridecanoic acid	PFTrDA	72629-94-8	662.8	618.9	5.19	9.66
Perfluorotetradecanoic acid	PFTA	376-06-7	712.8	668.8	5.34	9.94

Table 1: MRM transitions and retention times of PFAS according to EPA 537.1.

PFAS Analysis According to EPA 533 and to EPA 537.1

Analyte	Abbreviation	CAS number	Q_1 mass [Da]	Q_3 mass [Da]	Retention time [min] A	Retention time [min] B
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6	201.0	85.0	2.94	5.19
Perfluorobutanoic acid	PFBA	375-22-4	212.9	168.8	1.02	2.01
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1	228.9	85.0	1.37	2.64
Perfluoropentanoic acid	PFPeA	2706-90-3	262.9	219.0	2.14	3.90
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5	279.2	84.9	4.42	7.97
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	284.9	168.7	3.26	5.77
Perfluorobutanesulfonic acid	PFBS	375-73-5	298.9	98.9	2.37	4.20
Perfluorohexanoic acid	PFHxA	307-24-4	312.9	268.8	3.05	5.40
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7	315.1	135.1	2.76	4.85
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4	326.9	306.9	2,98	5.27
Perfluoropentanesulfonic acid	PFPeS	2706-91-4	348.8	80.0	3.13	5.54
Perfluoroheptanoic acid	PFHpA	375-85-9	362.9	318.8	3.60	6.45
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4	376.9	250.7	3.66	6.58
Perfluorohexanesulfonic acid	PFHxS	355-46-4	398.9	79.8	3.62	6.49
Perfluorooctanoic acid	PFOA	335-67-1	412.9	369.0	3.99	7.26
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2	426.9	406.9	3.99	7.24
Perfluoroheptanesulfonic acid	PFHpS	375-92-8	448.9	79.8	4.00	7.26
Perfluorononanoic acid	PFNA	375-95-1	462.9	418.9	4.31	7.92
Perfluorooctanesulfonic acid	PFOS	1763-23-1	498.8	79.9	4.30	7.89
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4	526.8	506.8	4.60	8.50
Perfluorodecanoic acid	PFDA	335-76-2	512.8	468.9	4.58	8.49
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF_3ONS	756426-58-1	530.8	350.7	4.48	8.25
Perfluoroundecanoic acid	PFUnA	2058-94-8	562.8	518.9	4.81	8.95
Perfluorododecanoic acid	PFDoA	307-55-1	612.8	568.9	5.02	9.33
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF_3OUdS	763051-92-9	630.7	451.0	4.93	9.15

Table 2: MRM transitions and retention times of PFAS according to EPA 533.

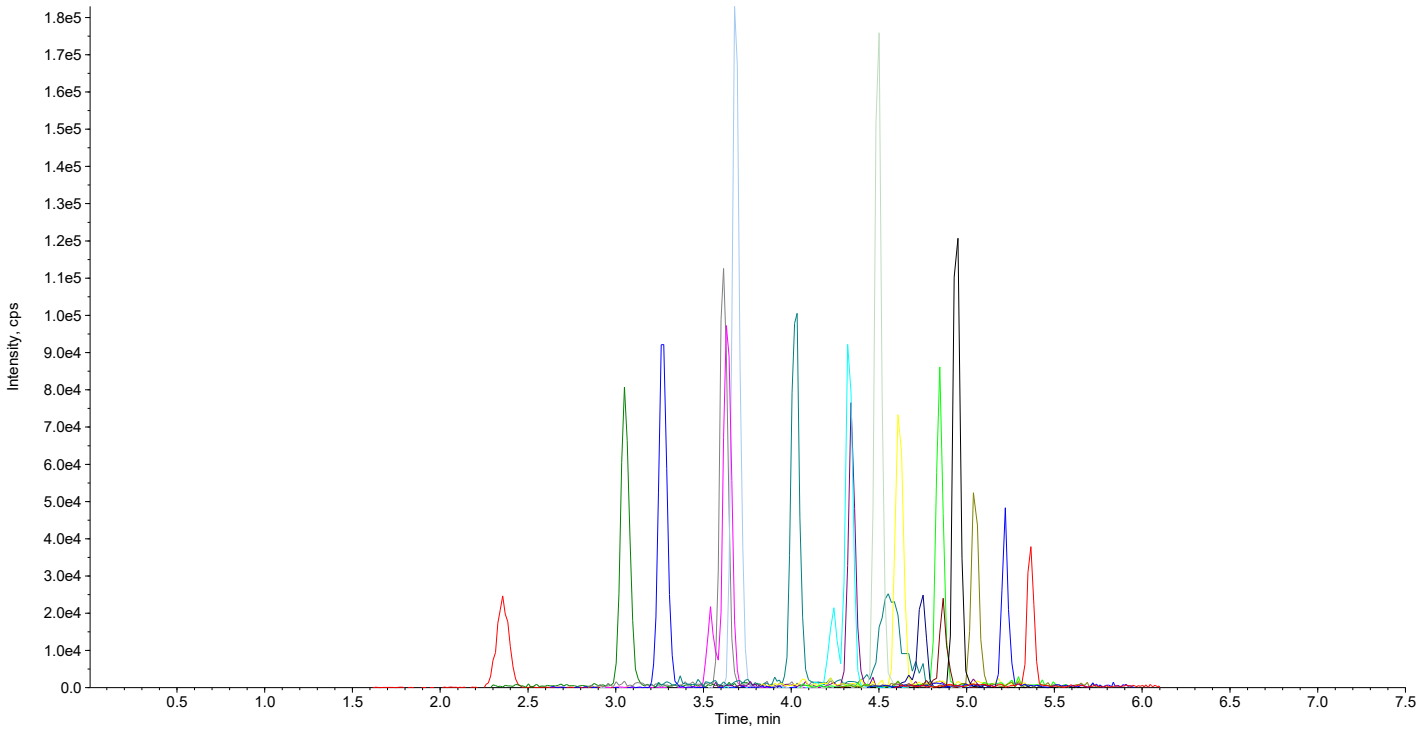

Figure 2: Chromatogram of PFAS according to EPA 537.1 on NUCLEODUR® PFAS EC 50 x 2 mm column (β = 1.0 ng/mL for each compound).

PFAS Analysis According to EPA 533 and to EPA 537.1

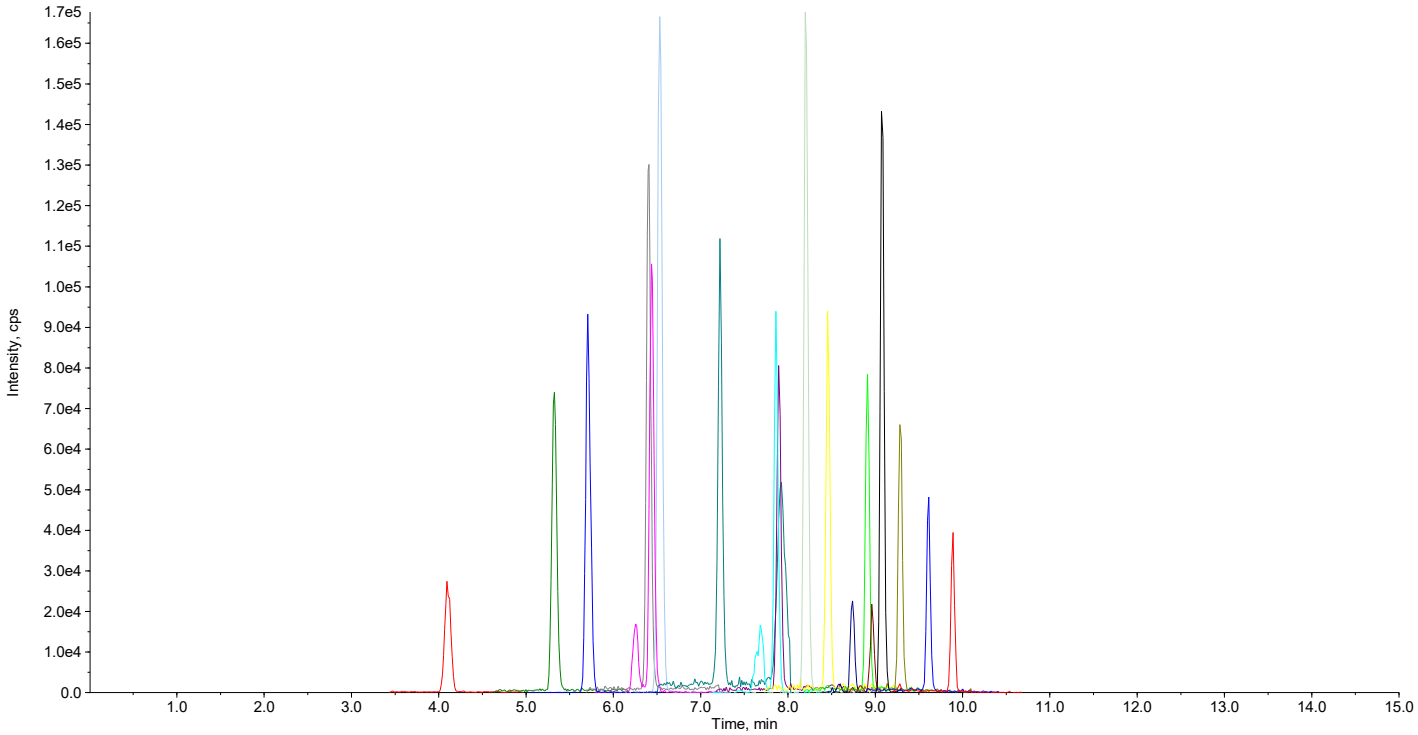

Figure 3: Chromatogram of PFAS according to EPA 537.1 on NUCLEODUR® PFAS EC 100 x 2 mm column (β = 1.0 ng/mL for each compound).

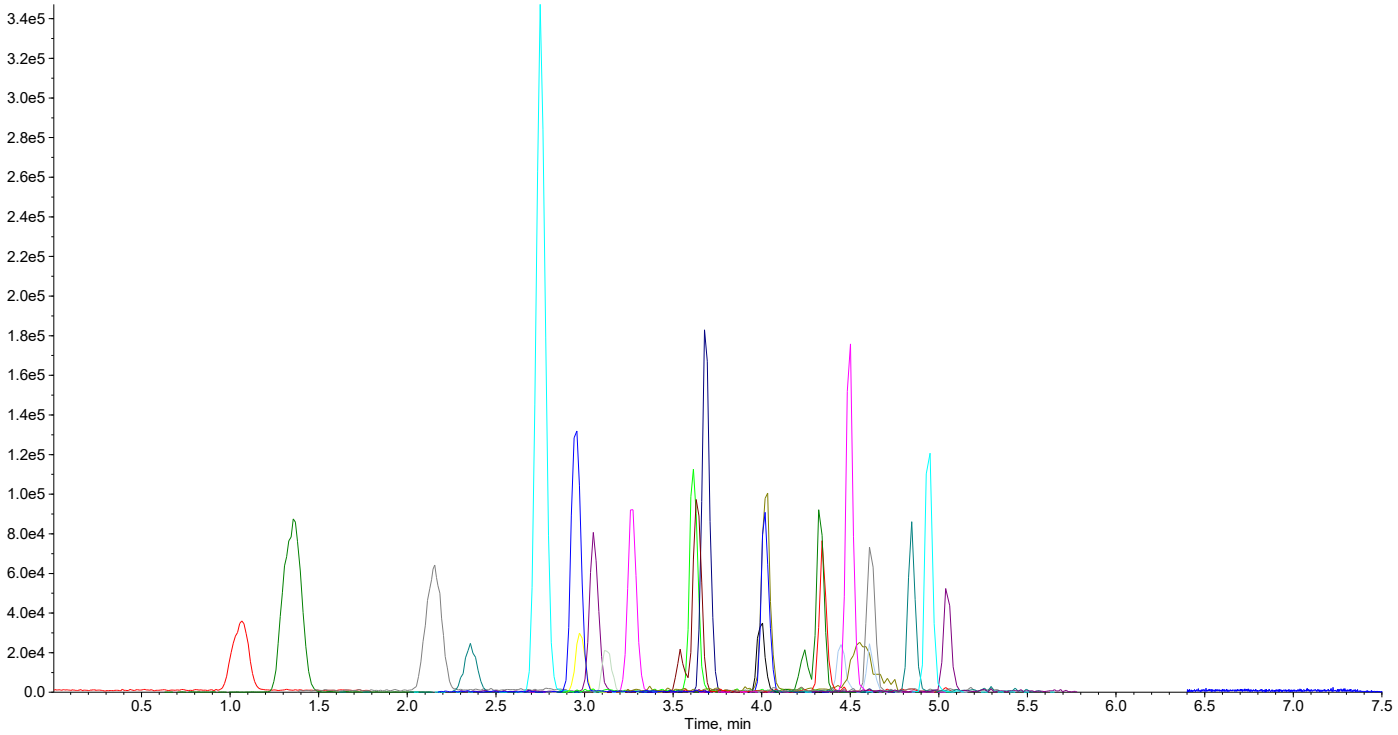

Figure 4: Chromatogram of PFAS according to EPA 533 on NUCLEODUR® PFAS EC 50 x 2 mm column (β = 1.0 ng/mL for each compound).

PFAS Analysis According to EPA 533 and to EPA 537.1

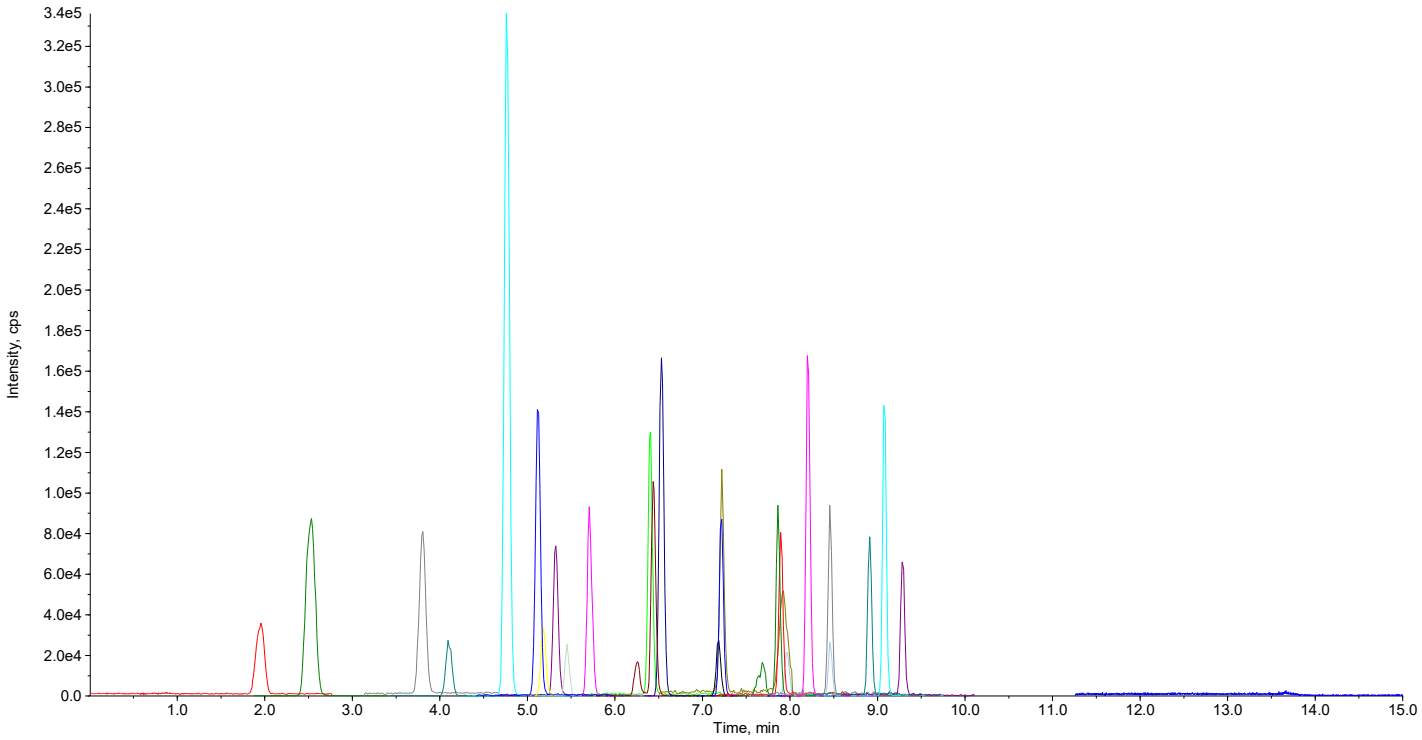

Figure 5: Chromatogram of PFAS according to EPA 533 on NUCLEODUR® PFAS EC 100 x 2 mm column (β = 1.0 ng/mL for each compound).

Excursus: The way of PFAS from manufacturing over waste and consumer products to environment and human contamination

PFAS are emitted into the environment by different pathways. For example, exhaust air from industrial sources can contain PFAS and thus are dispersed into nearby ground and water bodies. Rain and snow, for example, can eventually carry them from the air into the soil and surface waters. Particle accumulation can even cause them to travel long distances through the air. PFAS are therefore also found far from industrial production sites and human living areas, such as in sediments from the Bering Sea to the Arctic [5, 6].

Through volatilization from products (evaporation from carpets or home textiles treated with soil-repellent agents) or from waterproofing sprays, indoor air can also be contaminated.

Soils can also be directly contaminated, for example by firefighting foams. With the uptake of PFAS from contaminated soils and waters in vegetation and their accumulation in fish, these substances enter the human food chain. Consequently, humans absorb PFAS from the environment through food, water or air.

Schematic diagram

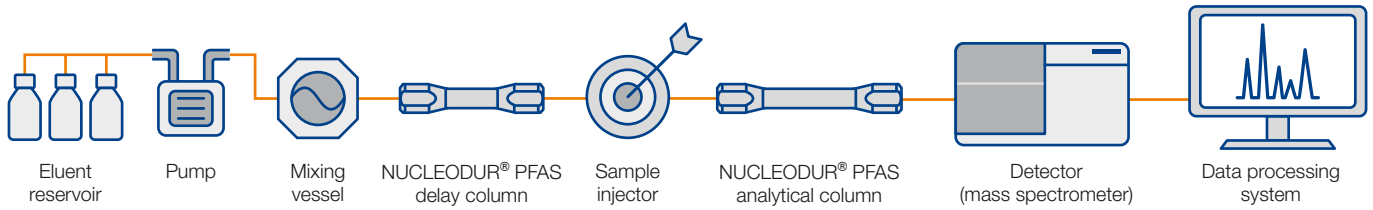

Figure 6: Installation and usage of NUCLEODUR® PFAS Delay column. The NUCLEODUR® PFAS Delay column is connected in flow direction between the mixing vessel and the sample injector.

PFAS Analysis According to EPA 533 and to EPA 537.1

Separation of instrument contaminants by using a delay column

The prevalence of PFOA is commonly observed as an instrument materials contaminant. The following figures 7 and 8 show the benefits of using a delay column for the combinations A and B (see chromatographic conditions).

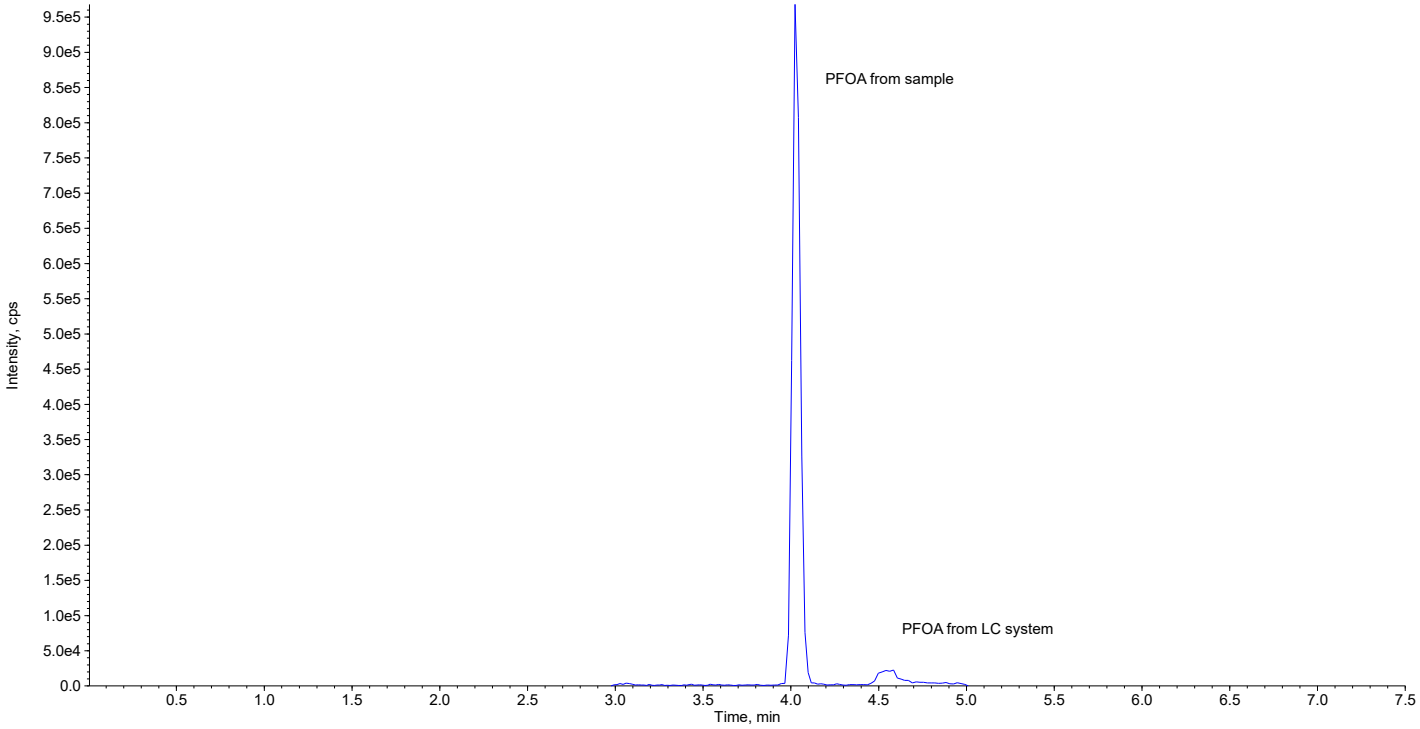

Figure 7: Chromatogram of PFOA resulted from the combination of NUCLEODUR® PFAS Delay EC 50/2 mm column x NUCLEODUR® PFAS EC 50/2 mm column (β = 10.0 ng/mL in methanol). The chromatogram shows the effectiveness of the NUCLEODUR® PFAS Delay column by impeding the instrument PFOA contamination from the sample by 0.5 minutes (RT 4.02 min PFOA from sample, RT 4.55 min PFOA from LC system).

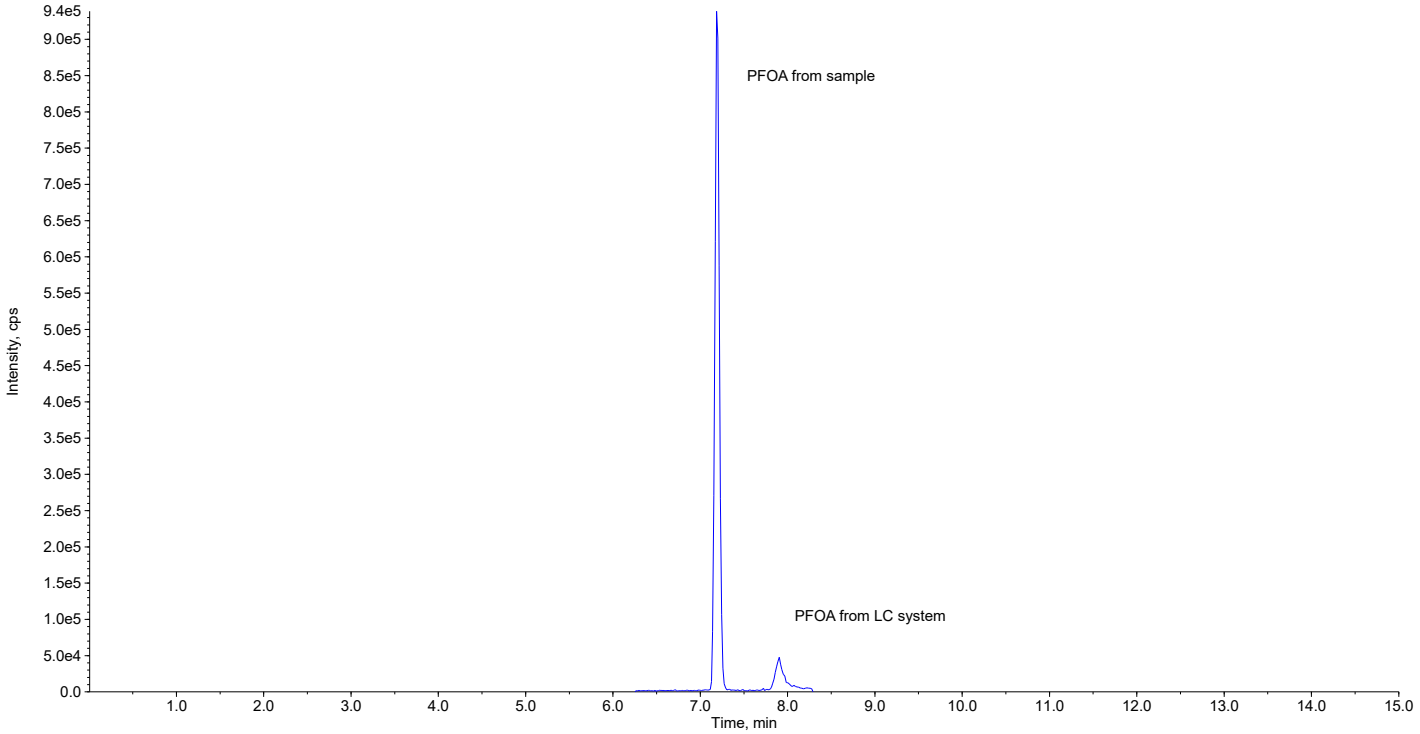

Figure 8: Chromatogram of PFOA resulted from the combination of NUCLEODUR® PFAS Delay EC 50/2 mm column x NUCLEODUR® PFAS EC 100/2 mm column (β = 10.0 ng/mL in methanol). The chromatogram shows the effectiveness of the NUCLEODUR® PFAS Delay column by impeding the instrument PFOA contamination from the sample by 0.7 minutes (RT 7.21 min PFOA from sample, RT 7.91 min PFOA from LC system).

PFAS Analysis According to EPA 533 and to EPA 537.1

Batch-to-batch reproducibility

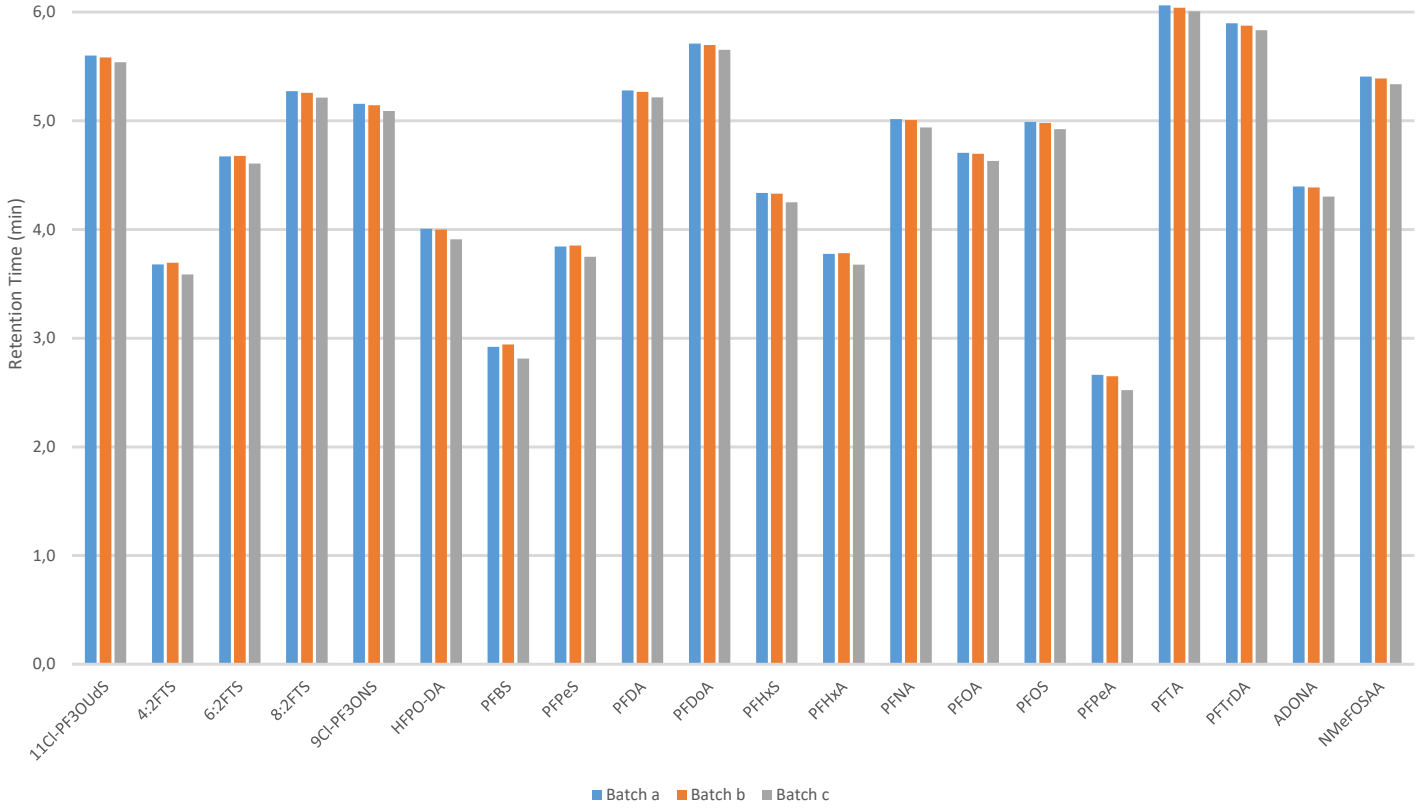

Figure 9: Comparison of retention times of PFAS on NUCLEODUR® PFAS EC 50 x 2 mm column using three batches (a-c).

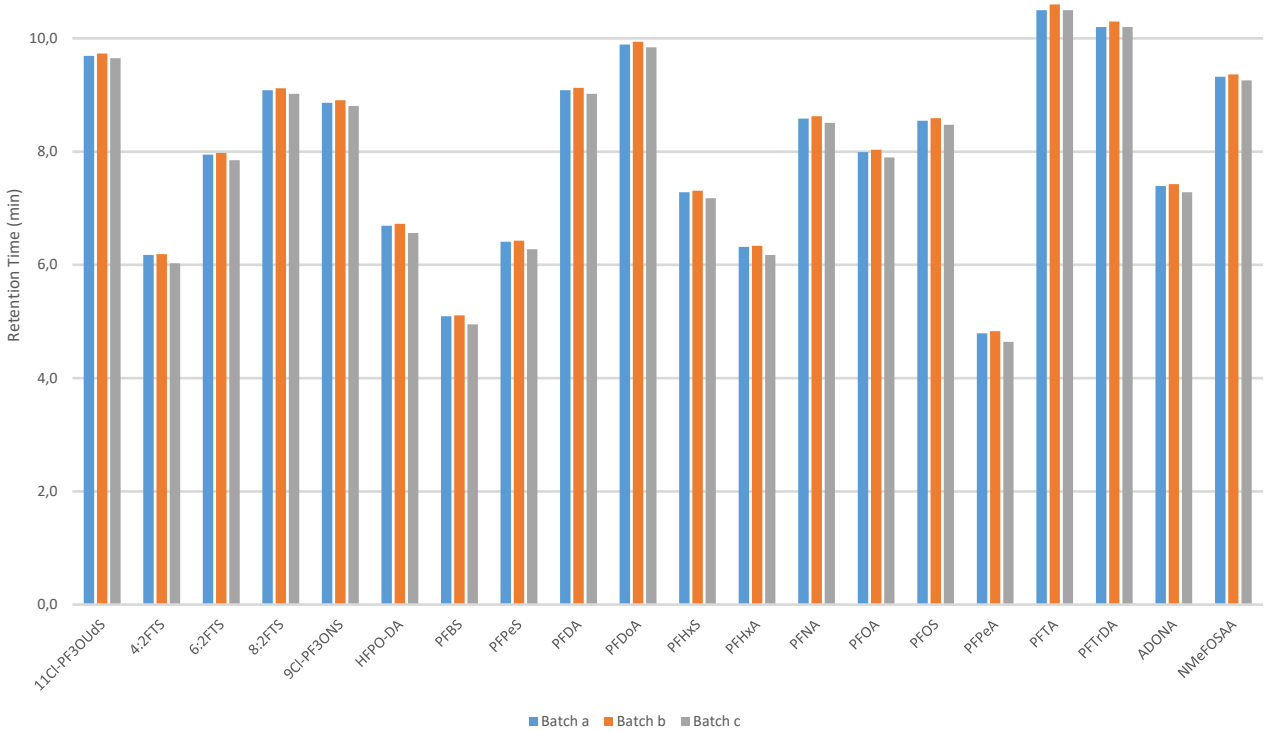

Figure 10: Comparison of retention times of PFAS on NUCLEODUR® PFAS EC 100 x 2 mm column using three batches (a-c).

PFAS Analysis According to EPA 533 and to EPA 537.1

Suitability of NUCLEODUR® PFAS columns for PFAS analysis

In the following tables the suitability of NUCLEODUR® PFAS for PFAS analysis is shown. There is a good correlation of the peaks, a high signal to noise ratio, sharp and narrow peaks with a large peak height which corresponds to high resolution and sensitivity.

		NUCLEODUR® PFAS EC 100 x 2 mm				NUCLEODUR® PFAS EC 50 x 2 mm			
Analyte	Abbreviation	Correlation	Area / Height	Width at 50%	Signal / Noise	Correlation	Area / Height	Width at 50%	Signal / Noise
Hexafluoropropylene oxide dimer acid	HFPO-DA	0.99862	4.08	0.06	1064	0.99913	3.51	0.06	1184
Perfluorobutanesulfonic acid	PFBS	0.99561	4.01	0.07	198	0.99888	4.91	0.08	351
Perfluorohexanoic acid	PFHxA	0.99918	4.05	0.06	266	0.99972	3.59	0.06	238
Perfluoroheptanoic acid	PFHpA	0.99905	3.68	0.06	307	0.99939	3.20	0.05	226
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	0.99918	3.85	0.06	2101	0.99851	3.12	0.05	989
Perfluorohexanesulfonic acid	PFHxS	0.99896	4.37	0.06	627	0.99959	3.95	0.05	421
Perfluorooctanoic acid	PFOA	0.99888	3.60	0.06	127	0.99941	3.27	0.05	142
Perfluorononanoic acid	PFNA	0.99698	3.46	0.05	186	0.99791	3.07	0.05	144
Perfluorooctanesulfonic acid	PFOS	0.99899	4.33	0.05	565	0.99911	3.85	0.05	420
Perfluorodecanoic acid	PFDA	0.99783	3.48	0.06	165	0.99788	3.30	0.05	133
9-Chlorohexadecafluoro-3-oxa-nonane-1-sulfonic acid	9Cl-PF_3ONS	0.99866	3.39	0.05	1867	0.99925	2.89	0.05	833
Perfluoroundecanoic acid	PFUnA	0.99841	3.24	0.05	185	0.99848	2.82	0.05	160
N-methyl perfluorooctanesulfonamido-acetic acid	NMeFOSAA	0.99831	4.00	0.06	216	0.99842	3.64	0.05	121
N-ethyl perfluorooctanesulfonamido-acetic acid	NEtFOSAA	0.99807	3.77	0.05	276	0.99899	3.27	0.05	169
Perfluorododecanoic acid	PFDoA	0.99845	3.26	0.05	179	0.99654	2.92	0.05	154
11-Chloroeicosafluoro-3-oxa-undecane-1-sulfonic acid	11Cl-PF_3OUdS	0.99864	3.25	0.05	1321	0.99796	2.88	0.04	629
Perfluorotridecanoic acid	PFTrDA	0.99302	2.63	0.04	112	0.99914	2.77	0.04	162
Perfluorotetradecanoic acid	PFTA	0.99673	3.05	0.05	230	0.99854	2.73	0.04	174

Table 3: Signal to noise ratio, area to height ratio of the peak, peak width at 50 % peak height and correlation of various sample concentrations for 18 PFAS analytes according to EPA 537.1.

		NUCLEODUR® PFAS EC 100 x 2 mm					NUCLEODUR® PFAS EC 50 x 2 mm				
Analyte	Abbreviation	Correlation	Area / Height	Retention time [min]	Width at 50%	Signal / Noise	Correlation	Area / Height	Retention time [min]	Width at 50%	Signal / Noise
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	0.99922	4.15	5.12	0.063	426	0.99949	3.77	2.95	0.06	417
Perfluorobutanoic acid	PFBA	0.99924	6.91	1.95	0.117	186	0.99937	6.51	1.06	0.11	192
Perfluoro-3-methoxypropanoic acid	PFMPA	0.99933	7.50	2.52	0.120	1023	0.99958	7.29	1.35	0.12	820
Perfluoropentanoic acid	PFPeA	0.99919	5.44	3.80	0.083	269	0.99954	6.22	2.15	0.10	188
Perfluoro-4-methoxybutanoic acid	PFMBA	0.99875	4.00	7.96	0.060	177	0.99588	3.28	4.45	0.05	214
Hexafluoropropylene oxide dimer acid	HFPO-DA	0.99862	4.08	5.71	0.063	1064	0.99913	3.51	3.27	0.06	1184
Perfluorobutanesulfonic acid	PFBS	0.99561	4.01	4.15	0.070	198	0.99888	4.91	2.36	0.08	351
Perfluorohexanoic acid	PFHxA	0.99918	4.05	5.32	0.060	266	0.99972	3.59	3.05	0.06	238
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	0.99886	4.25	4.76	0.070	1588	0.99950	3.95	2.75	0.06	2168
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	4:2FTS	0.99900	4.09	5.18	0.063	695	0.99952	3.60	2.97	0.06	442
Perfluoropentanesulfonic acid	PFPeS	0.99910	3.78	5.45	0.060	455	0.99925	3.50	3.13	0.05	242
Perfluoroheptanoic acid	PFHpA	0.99905	3.68	6.40	0.060	307	0.99939	3.20	3.61	0.05	226
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	0.99918	3.85	6.53	0.060	2102	0.99851	3.12	3.68	0.05	989
Perfluorohexanesulfonic acid	PFHxS	0.99896	4.37	6.44	0.057	627	0.99959	3.95	3.64	0.05	421
Perfluorooctanoic acid	PFOA	0.99888	3.60	7.22	0.050	127	0.99941	3.27	4.02	0.05	142
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2FTS	0.99891	3.72	7.18	0.057	654	0.99227	3.33	3.99	0.05	392
Perfluoroheptanesulfonic acid	PFHpS	0.99888	3.54	7.21	0.053	1183	0.99852	3.05	4.02	0.05	762
Perfluorononanoic acid	PFNA	0.99698	3.46	7.89	0.050	186	0.99791	3.07	4.34	0.05	144

PFAS Analysis According to EPA 533 and to EPA 537.1

Analyte	Abbreviation	NUCLEODUR® PFAS EC 100 x 2 mm					NUCLEODUR® PFAS EC 50 x 2 mm				
		Correlation	Area / Height	Retention time [min]	Width at 50%	Signal / Noise	Correlation	Area / Height	Retention time [min]	Width at 50%	Signal / Noise
Perfluorooctanesulfonic acid	PFOS	0.99899	4.33	7.86	0.053	565	0.99911	3.85	4.33	0.05	420
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	8:2FTS	0.99783	3.48	8.46	0.057	165	0.99788	3.30	4.62	0.05	133
Perfluorodecanoic acid	PFDA	0.99837	3.45	8.46	0.057	336	0.99765	3.19	4.61	0.05	113
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF_3ONS	0.99866	3.39	8.21	0.050	1867	0.99925	2.89	4.49	0.05	833
Perfluoroundecanoic acid	PFUnA	0.99841	3.24	8.91	0.047	185	0.99848	2.82	4.84	0.05	160
Perfluorododecanoic acid	PFDoA	0.99845	3.26	9.28	0.050	179	0.99654	2.92	5.05	0.05	154
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF_3OUdS	0.99864	3.25	9.08	0.050	1321	0.99796	2.88	4.94	0.04	629

Table 4: Signal to noise ratio, area to height of the peak, peak width at 50 % peak height and correlation of various sample concentrations for 25 PFAS analytes according to EPA 533.

Conclusion

This application note shows the reliable and successful separation of per- and polyfluoroalkyl substances (PFAS) on optimized PFAS HPLC columns. Figures 2–5 show chromatograms of PFAS standard mixtures according to EPA methods 533 and 537.1.

The NUCLEODUR® PFAS column is especially well suited for the analysis of PFAS compounds. This HPLC phase shows high retention for polar PFAS like PFBA and PBS and high MS intensity. Figure 9 and 10 present the excellent batch-to-batch reproducibility by comparing the retention times of several PFAS using three different batches. If the chromatographic issue is focused on the separation of linear and branched PFAS isomers NUCLEODUR® PFAS column should have a length of 100 mm.

This work points out the effectiveness of the NUCLEODUR® PFAS Delay column. This isolator column impedes the instrument contamination (especially PFOA) from the sample by 0.5 or 0.7 minutes respectively.

In summary, the presented application note describes a quick and convenient method for chromatographic separation of PFAS according to EPA Methods 537.1 and 533.

References

[1] United States Environmental Protection Agency, PFAS Laws and Regulations, https://www.epa.gov/pfas/pfas-laws-and-regulations.

[2] EPA's Per- and Polyfluoroalkyl Substances (PFAS) Action Plan, EPA $823R_1$8004, February 2019, www.epa.gov/pfas.

[3] Method 537.1: Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS). U.S. Environmental Protection Agency, Office of Research and Development, National Center for Environmental Assessment, Washington, DC, 2018.

[4] Method 533: Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry. U.S. Environmental Protection Agency, Office of Research and Development, National Center for Environmental Assessment, Washington, DC, 2019.

[5] Hanna Joerss, Zhiyong Xie, Charlotte C. Wagner, Wilken-Jon von Appen, Elsie M. Sunderland, and Ralf Ebinghaus: Transport of Legacy Perfluoroalkyl Substances and the Replacement Compound HFPO-DA through the Atlantic Gateway to the Arctic Ocean—Is the Arctic a Sink or a Source? Environ. Sci. Technol. 2020, Publication Date:July 29, 2020.

[6] Yan Lin, Jheng-Jie Jiang, Lisa A. Rodenburg, Minggang Cai, Zhai Wu, Hongwei Ke, Mahdi Chitsaz: Perfluoroalkyl substances in sediments from the Bering Sea to the western Arctic: Source and pathway analysis, Environment International, Volume 139, 2020.

Product information

The following MACHEREY-NAGEL products have been used in this application note:

REF 760673.20	EC 50/2 NUCLEODUR® PFAS Delay, 5 µm
REF 760666.20	EC 100/2 NUCLEODUR® PFAS, 3 µm
REF 760663.20	EC 50/2 NUCLEODUR® PFAS, 3 µm
REF 702402	Screw closure, N 9, PP, blue, center hole, silicone white/polyimide orange, 1.0 mm, fluorine-free
REF 702009	Screw neck vial, N 9, 11.6 x 32.0 mm, 0.3 mL, inner cone, PP transparent

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