

Column-switching LC-MS/MS analysis of PFAS in human serum using YMC-Triart C18

U210519AE

Poly- and perfluoroalkyl substances (PFAS) have been widely used because they have various properties such as water repellency, heat resistance etc. However, they have been researched and regulated worldwide in recent years due to concern over their adverse health effects.

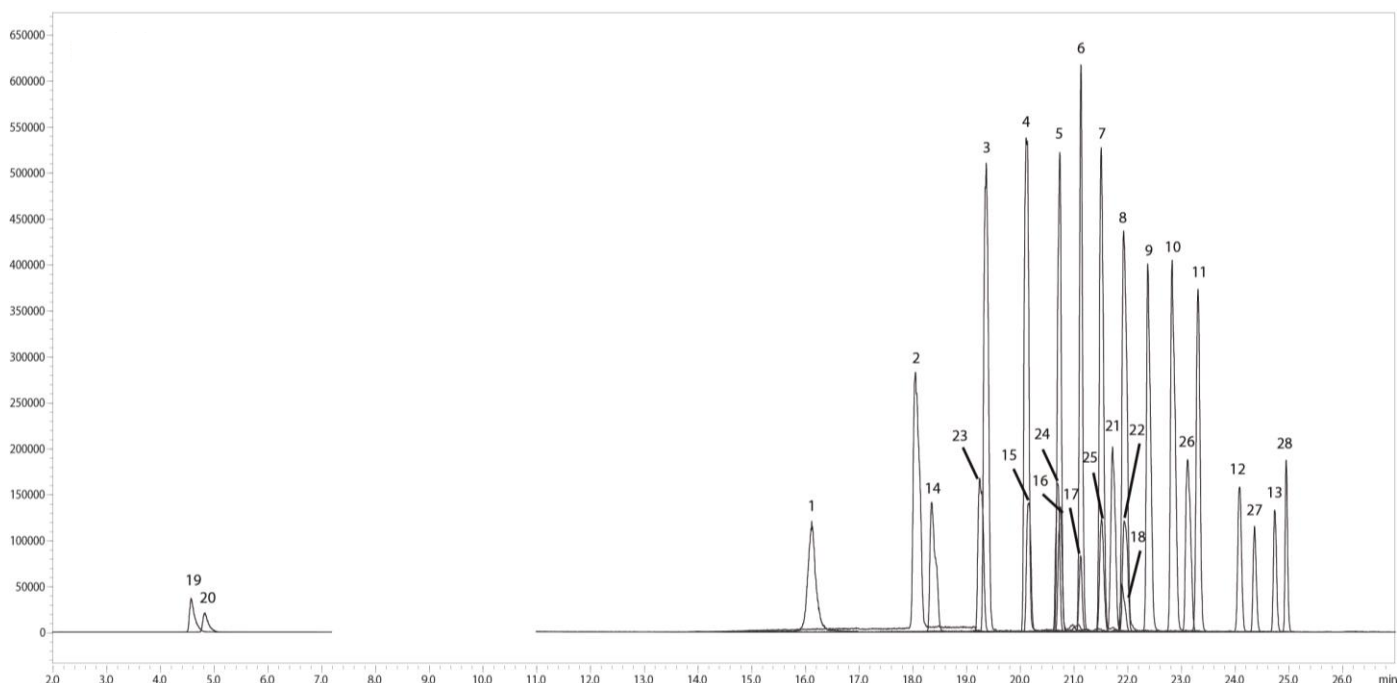
Recently, the highly sensitive and high-throughput method for the analysis of 28 PFAS in human serum was reported by Nakayama et al¹). An automated solid phase extraction (SPE) coupled with the on-line SPE column-switching LC-MS/MS method using YMC-Triart C18 as analytical column was employed.

Target PFAS

- | | |
|---|--|
| 1. Perfluorobutanoic acid (PFBA) | 17. Perfluorooctane sulphonic acid (PFOS) |
| 2. Perfluoropentanoic acid (PFPA) | 18. Perfluorodecane sulphonic acid (PFDS) |
| 3. Perfluorohexanoic acid (PFHxA) | 19. <i>N</i> -Methyl perfluorooctane sulphonamide (MeFOSA-M) |
| 4. Perfluoroheptanoic acid (PFHpA) | 20. <i>N</i> -Ethyl perfluorooctane sulphonamide (EtFOSA-M) |
| 5. Perfluorooctanoic acid (PFOA) | 21. <i>N</i> -Methyl perfluorooctane sulphonamido acetic acid (MeFOSA-A) |
| 6. Perfluorononanoic acid (PFNA) | 22. <i>N</i> -Ethyl perfluorooctane sulphonamido acetic acid (EtFOSA-A) |
| 7. Perfluorodecanoic acid (PFDA) | 23. 4:2 Fluorotelomer sulphonic acid (4:2 FTS) |
| 8. Perfluoroundecanoic acid (PFUnA) | 24. 6:2 Fluorotelomer sulphonic acid (6:2 FTS) |
| 9. Perfluorododecanoic acid (PFDoA) | 25. 8:2 Fluorotelomer sulphonic acid (8:2 FTS) |
| 10. Perfluorotridecanoic acid (PFTrDA) | 26. 6:2 Polyfluoroalkyl phosphoric acid diester (6:2 diPAP) |
| 11. Perfluorotetradecanoic acid (PFTeDA) | 27. 8:2 Polyfluoroalkyl phosphoric acid diester (8:2 diPAP) |
| 12. Perfluorohexadecanoic acid (PFHxDA) | 28. Perfluorooctane sulphonamido ethanol-based phosphate ester (diSAM PAP) |
| 13. Perfluorooctadecanoic acid (PFODA) | |
| 14. Perfluorobutane sulphonic acid (PFBS) | |
| 15. Perfluorohexane sulphonic acid (PFHxS) | |
| 16. Perfluoroheptane sulphonic acid (PFHpS) | |

Chromatogram

Neutral matrices (#19, 20) were analyzed using eluent with near neutral pH, and ionization matrices were analyzed by switching to alkaline eluent. YMC-Triart C18 have great durability and enable stable analysis even under alkaline condition.



LC condition

Column : YMC-Triart C18 (3 μ m, 12 nm)
 [Guard column] 10 X 2.1 mmI.D.
 [Analytical column] 100 X 2.1 mmI.D.

Temperature : 40°C

Detection : negative ESI

Injection : 400 μ L

Eluent

Sample : standard solution (4 ng/mL)

LC Pump A : 2.5 mM ammonium acetate

Sample load

LC Pump B1 : methanol

LC pump D1 : water

LC Pump B2 : 2.5 mM ammonium acetate in
95% methanol

On-line SPE column wash

LC pump D2 : 0.5% ammonia in 90% methanol

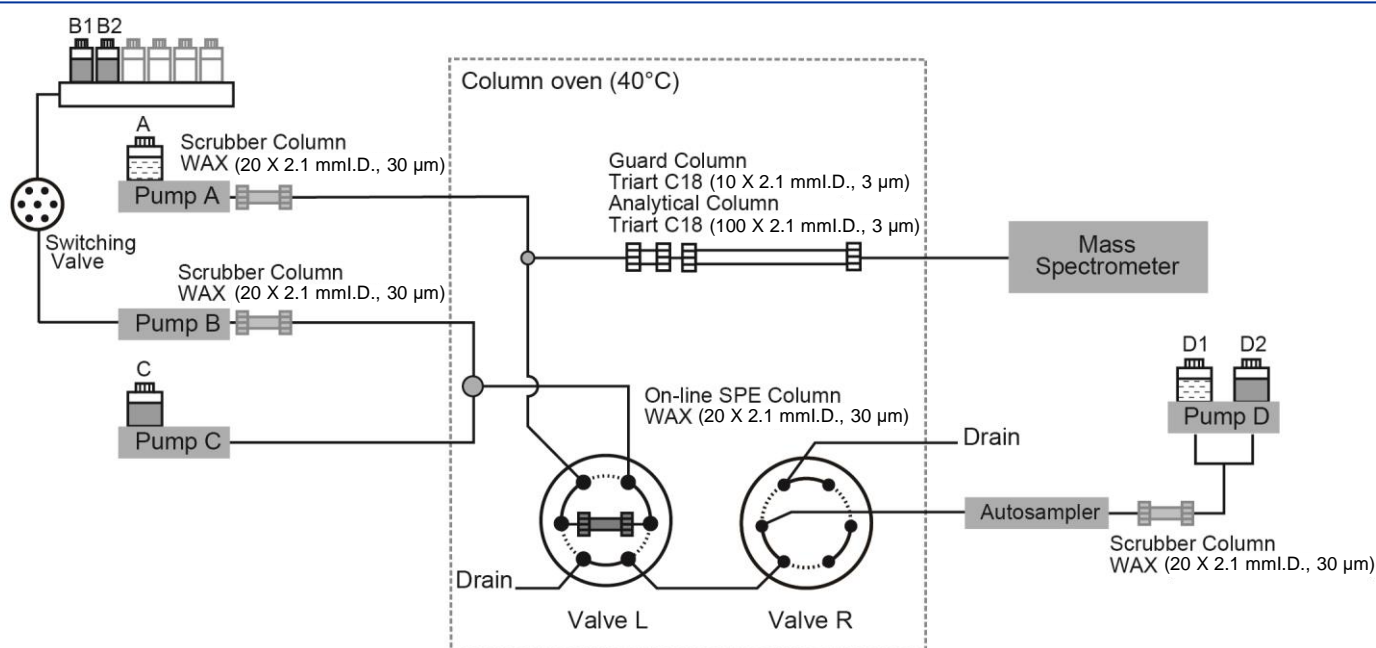
LC Pump C : 0.1% ammonia in methanol

System : LCMS-8060 (Shimadzu Corporation)

Time (min)	Ternary gradient				Flow rate	Binary gradient		Flow rate	Valve position	
	Pump A (%)	Pump B1 (%)	Pump B2 (%)	Pump C (%)	Pump A+B+C (ml/min)	Pump D1 (%)	Pump D2 (%)	Pump D (ml/min)	Valve L	Valve R
0.00	12.5	87.5	0.0	0.0	0.3	100	0.0	1.0	1	0
2.00	12.5	87.5	0.0	0.0	0.3	100	0.0	1.0	0	1
2.01	12.5	87.5	0.0	0.0	0.3	0.0	0.0	0.0	0	1
4.00	12.5	87.5	0.0	0.0	0.3	0.0	0.0	0.0	0	1
5.50	12.5	87.5	0.0	0.0	0.3	0.0	0.0	0.0	1	0
5.51	12.5	0.0	87.5	0.0	0.3	0.0	0.0	0.0	1	0
7.00	12.5	0.0	87.5	0.0	0.3	0.0	0.0	0.0	1	0
7.01	92.5	0.0	0.0	7.5	0.5	0.0	0.0	0.0	1	0
7.50	92.5	0.0	0.0	7.5	0.5	0.0	0.0	0.0	0	1
10.00	92.5	0.0	0.0	7.5	0.5	0.0	0.0	0.0	0	1
10.01	92.5	0.0	0.0	7.5	0.3	0.0	0.0	0.0	0	1
18.00	45	0.0	47.5	7.5	0.3	0.0	0.0	0.0	0	1
20.00	27.5	0.0	65	7.5	0.3	0.0	0.0	0.0	1	0
20.01	*a	0.0	*a	12.5	0.3	0.0	100	1.0	1	0
24.00	*a	0.0	*a	12.5	0.3	0.0	100	1.0	1	0
24.01	*a	0.0	*a	12.5	0.3	100	0.0	1.0	1	0
25.00	0.0	0.0	87.5	12.5	0.3	100	0.0	1.0	1	0
25.01	12.5	87.5	0.0	0.0	0.3	100	0.0	1.0	1	0
27.00	12.5	87.5	0.0	0.0	0.3	100	0.0	1.0	1	0

*a 20.00–25.00 min linear gradient (Pump A + Pump B2)

Instrumental configuration



Courtesy of Dr. Shoji F. Nakayama, National Institute for Environmental Studies

Reference 1)

Shoji F. Nakayama et al. Poly- and perfluoroalkyl substances in maternal serum: Method development and application in Pilot Study of the Japan Environment and Children's Study, Journal of Chromatography A 1618 (2020) 460933