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DETECTING AND REGULATING PFSA CONTAMINATION IN WATER: ADVANCEMENTS IN LC-MS/MS METHODOLOGY

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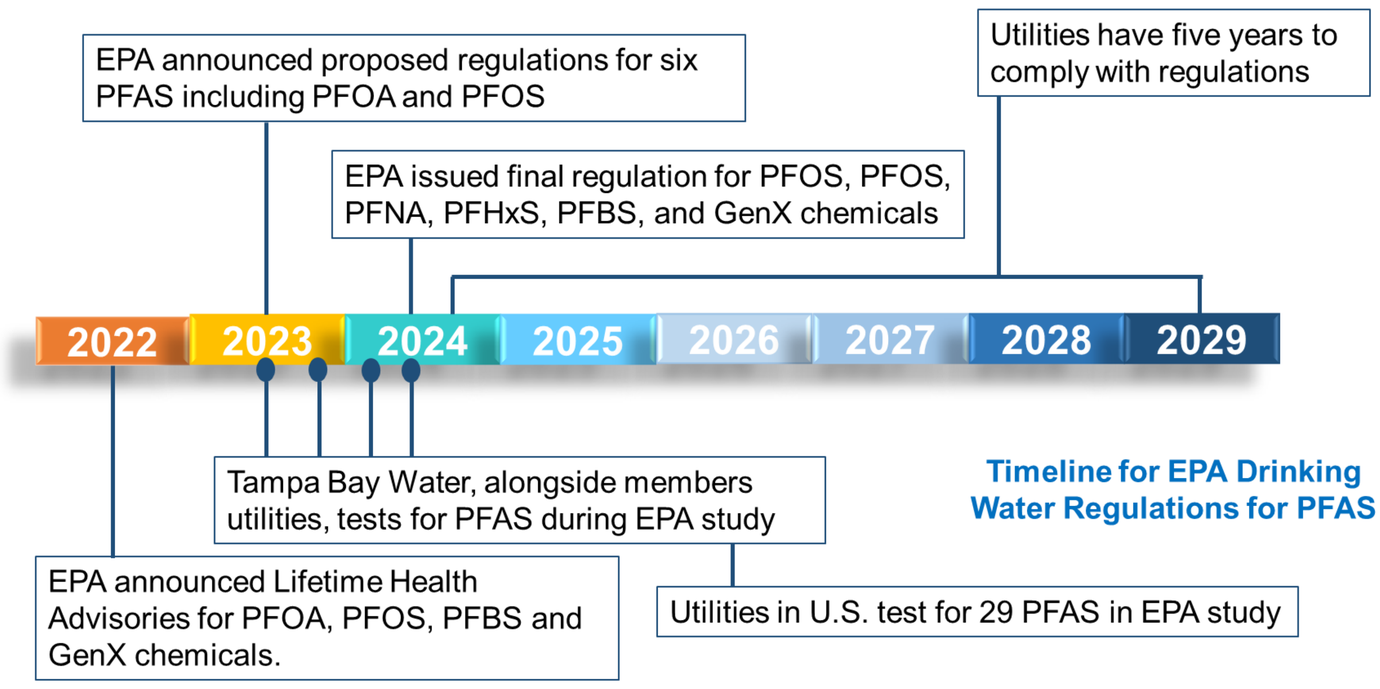
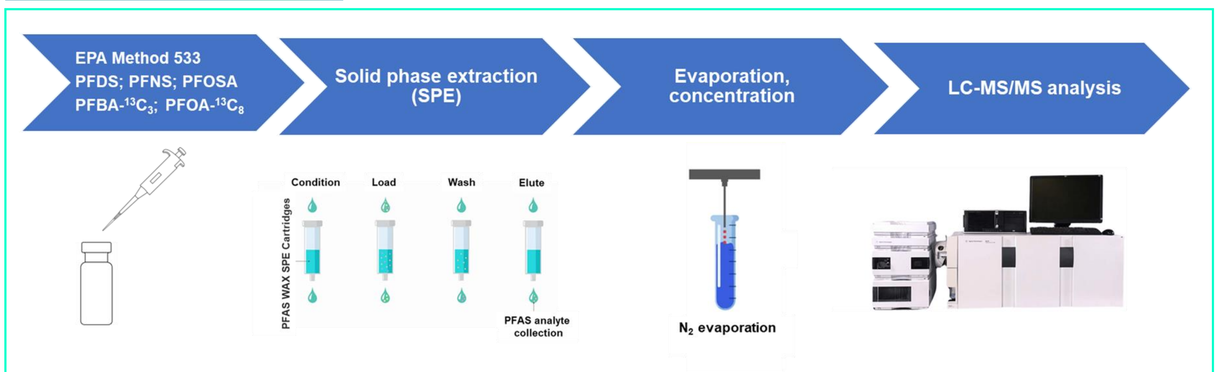
Background

- **PFAS Overview:** Synthetic chemicals linked to environmental and health issues.
- **Regulatory Focus:** EPA's timeline for drinking water regulations targeting PFOS and PFOA.
- **Analytical Demand:** Increased need for effective detection methods for PFAS.
- **Detection Techniques:** Essential use of LC-MS/MS for accurate PFAS measurement.
- **Public Health Impact:** Need for advanced methods to meet standards and reduce contamination risks.
- **Aim of the Work:** Development and validation of a new LC-MS/MS method for PFAS detection in drinking water.

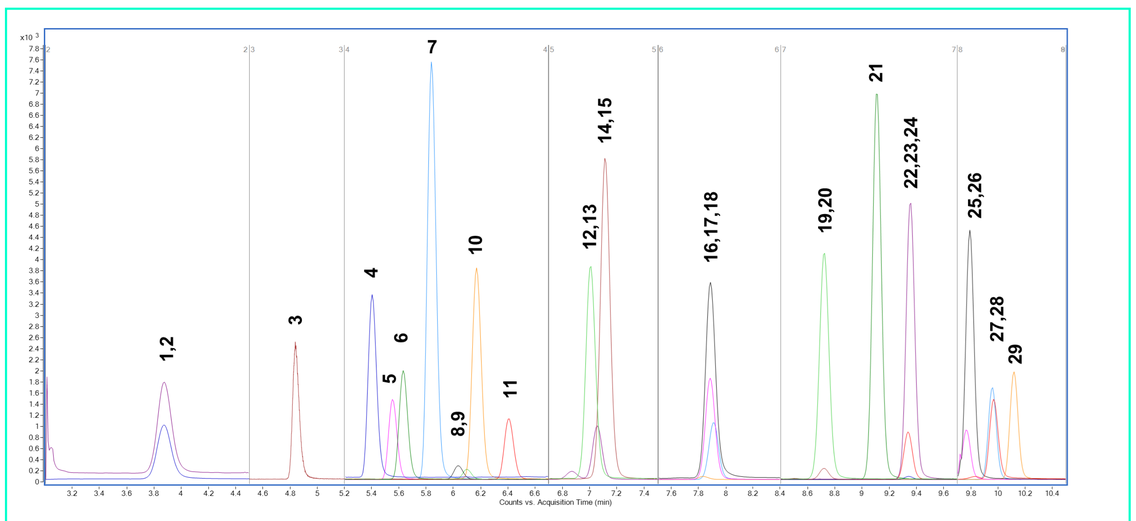


Timeline for EPA Drinking Water Regulations for PFAS

Methodology



Chromatogram



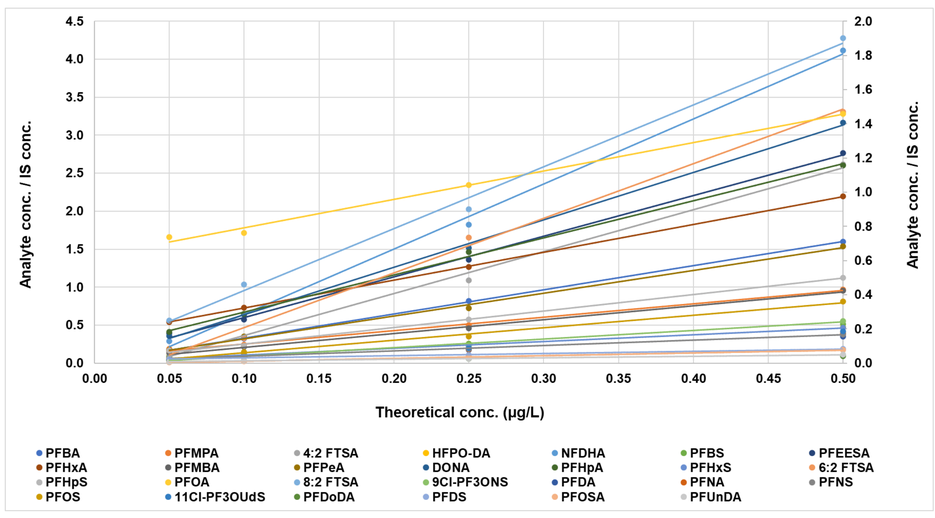
Calibration standard with PFAS and labelled IS sstandards, at 500 ng/L (in sample concentration after 500x sample pre-concentration during sample preparation

LC conditions

- Chromatographic column:Zorbabax eclipse XDB-C18
- Colum temperature: 30°C
- Injection volume: 20 µl
- Mobile phase: 5 mM ammonium acetate/ MeOH
- Flow: 0.4 mL/min
- Sample solvent: water
- Elution mode: gradient program
- Chromatographic run-time: 20 min

Time (min)	A (%)	B (%)
0.00	85	15
1.00	75	25
1.50	45	55
5.50	30	70
9.00	0	100
10.0	0	100
10.1	85	15

Method performance



Performance parameters for PFAS analysis in drinking water using LC-MS/MS

No.	Analyte	LOD (µg/L)	LOQ (µg/L)	Linearity	Concentration coefficient	Matrix effects (%)	Intra-day precision (%)	Inter-day precision (%)
1	PFBA	0.01	0.03	0.05 - 0.50	500	102 ± 6.3	3.2	9.4
2	PFMPA					100 ± 4.8	3.7	9.2
3	4:2 FTSA					98 ± 7.4	4.1	10.6
4	PFEESA					103 ± 4.2	4.8	10.2
5	PFHxA					108 ± 6.3	6.9	12.3
6	PFBS					102 ± 6.3	5.7	11.7
7	NFDHA					96 ± 6.4	6.3	10.4
8	HFPO-DA					94 ± 7.8	8.1	13.8
9	PFMBA					99 ± 7.0	4.9	10.2
10	PFPeA					103 ± 7.1	5.7	10.9
11	PFHxS					96 ± 8.0	6.4	11.7
12	DONA					98 ± 6.6	8.3	12.4
13	PFHpA					104 ± 8.7	5.6	11.7
14	PFHpS					107 ± 8.5	7.1	12.8
15	6:2 FTSA					93 ± 6.0	5.8	11.4
16	PFOA					90 ± 8.3	6.9	12.3
17	PFNS					105 ± 6.1	5.8	12.2
18	9Cl-PF_3ONS					96 ± 5.4	7.4	13.5
19	8:2 FTSA					94 ± 4.5	7.6	13.8
20	PFDA					97 ± 8.9	5.3	10.7
21	PFOS					105 ± 5.0	6.4	11.2
22	PFNA					102 ± 6.6	6.8	12.4
23	11Cl-PF_3OUdS					106 ± 7.6	7.2	13.5
24	PFDoDA					94 ± 7.8	6.9	12.6
25	PFDS					97 ± 9.1	8.2	13.9
26	PFUnDA					92 ± 8.4	6.5	11.8
27	PFOSA					103 ± 6.4	7.3	12.7

MS conditions

No.	Analyte	r_T	Precursor ion	Product ion	Dwel time	Fragmentor Voltage (V)	Collision Energy (V)	Cell Accelerator Voltage (V)	ESI source
1	PFBA-$^{13}C_3$	3.87	216	172	250	80	2	5	Negative
2	PFBA	3.88	213	169	250	60	4	0	Negative
3	PFMPA	4.84	229	85	10	60	12	4	Negative
4	4:2 FTSA	6.13	327	307	60	30	15	4	Negative
5	PFEESA	6.43	315	135	60	110	24	4	Negative
6	PFHxA	6.06	313	269	60	80	3	6	Negative
7	PFBS	5.57	299	80	60	90	45	6	Negative
8	NFDHA	5.86	295	201	60	120	0	4	Negative
9	HFPO-DA	6.20	285	169	60	75	4	4	Negative
10	PFMBA	5.66	279	85	60	70	12	4	Negative
11	PFPeA	5.43	263	219	60	71	3	5	Negative
12	PFHxS	7.15	399	80	170	120	50	4	Negative
13	DONA	7.04	377	251	170	90	5	4	Negative
14	PFHpA	7.08	363	319	170	75	1	4	Negative
15	PFHpS	7.87	449	80	120	135	50	4	Negative
16	6:2 FTSA	7.95	427	81	120	90	50	5	Negative
17	PFOA $^{13}C_8$	7.92	421	376	120	83	4	3	Negative
18	PFOA	7.94	413	369	120	90	0	2	Negative
19	PFNS	9.37	549	80	80	135	60	6	Negative
20	9Cl-PF_3ONS	9.14	531	351	80	145	28	4	Negative
21	8:2 FTSA	9.38	527	81	80	90	40	5	Negative
22	PFDA	8.77	513	469	80	90	10	0	Negative
23	PFOS	9.37	499	80	80	140	30	5	Negative
24	PFNA	8.76	463	419	80	75	2	1	Negative
25	11Cl-PF_3OUdS	9.98	631	451	100	60	20	4	Negative
26	PFDoDA	10.14	613	569	100	30	10	4	Negative
27	PFDS	9.80	599	80	100	135	60	6	Negative
28	PFUnDA	9.99	563	519	100	80	10	0	Negative
29	PFOSA	9.82	498	78	100	135	45	5	Negative

Conclusions

- **Effective Detection:** LC-MS/MS achieved LODs as low as 0.01 µg/L for 27 PFAS, meeting EPA and Ord. 7/2023 requirements.
- **Reliable Performance:** The method demonstrated strong linearity and excellent precision, ensuring accurate quantification for routine monitoring.
- **Minimal Interference:** The analysis exhibited low matrix effects (90-108%), confirming robustness in complex water samples.
- **Comprehensive Analysis:** Detection of 27 PFAS highlights the method's applicability for regulatory compliance and risk assessment.
- **Public Health Impact:** This analytical approach supports effective monitoring, enhancing protections against PFAS contamination in drinking water.

Acknowledgments

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Disemination

Pascu, L.F.; Petre, V.A.; Cimpean, I.A.; Paun, I.; Pirvu, F.; Chiriac, F.L. Managing PFAS in Sewage Sludge: Exposure Pathways, Impacts, and Treatment Innovations. J. Xenobiot. 2025, 15, 135. https://doi.org/10.3390/jox15040135..