



RAPID PFAS ANALYSIS USING FUSED-CORE® TECHNOLOGY

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Outline

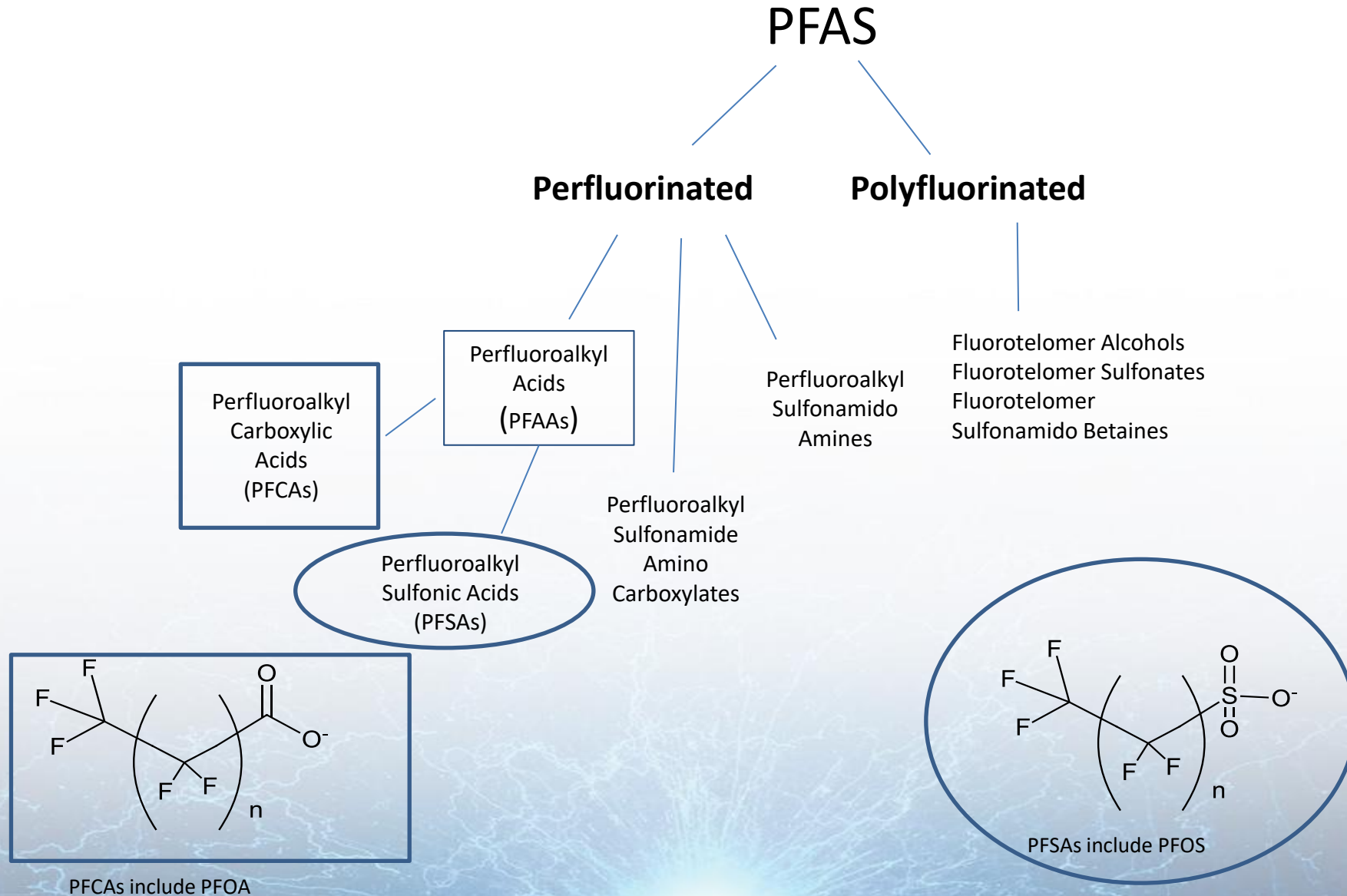
- PFAS:
 - a) What?
 - b) Where?
 - c) Why?
 - d) How?



- HALO[®] PFAS Solution



What: PFAS Chemicals

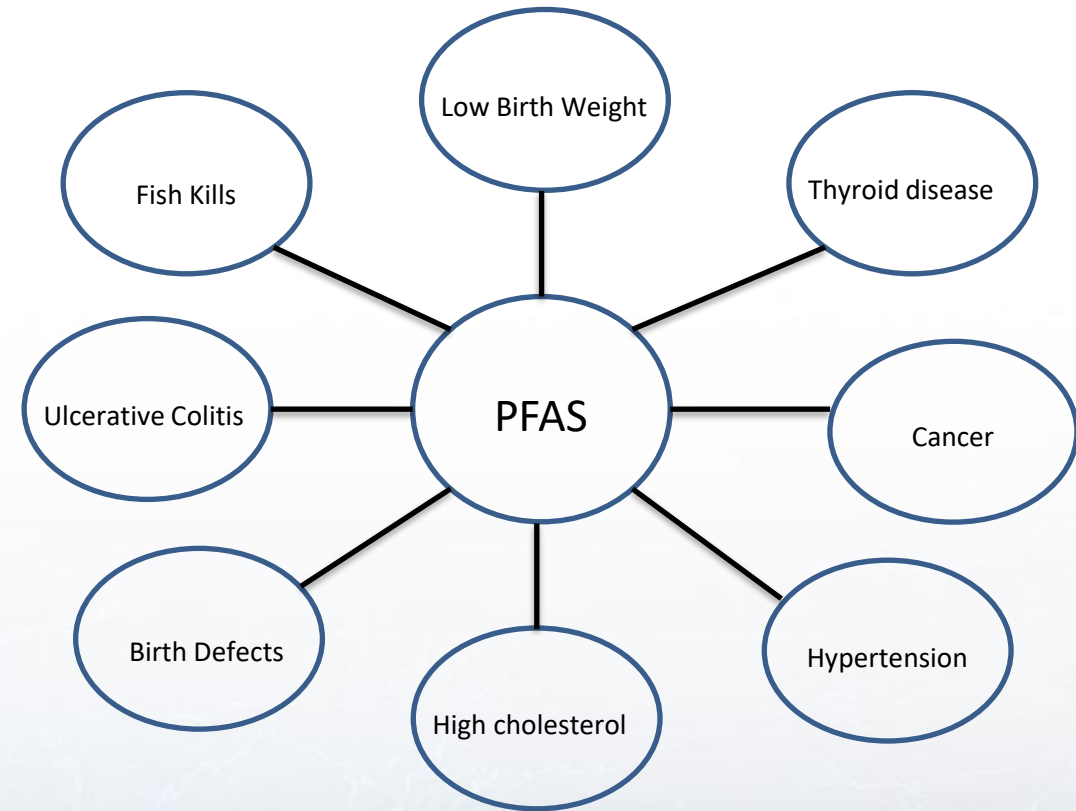
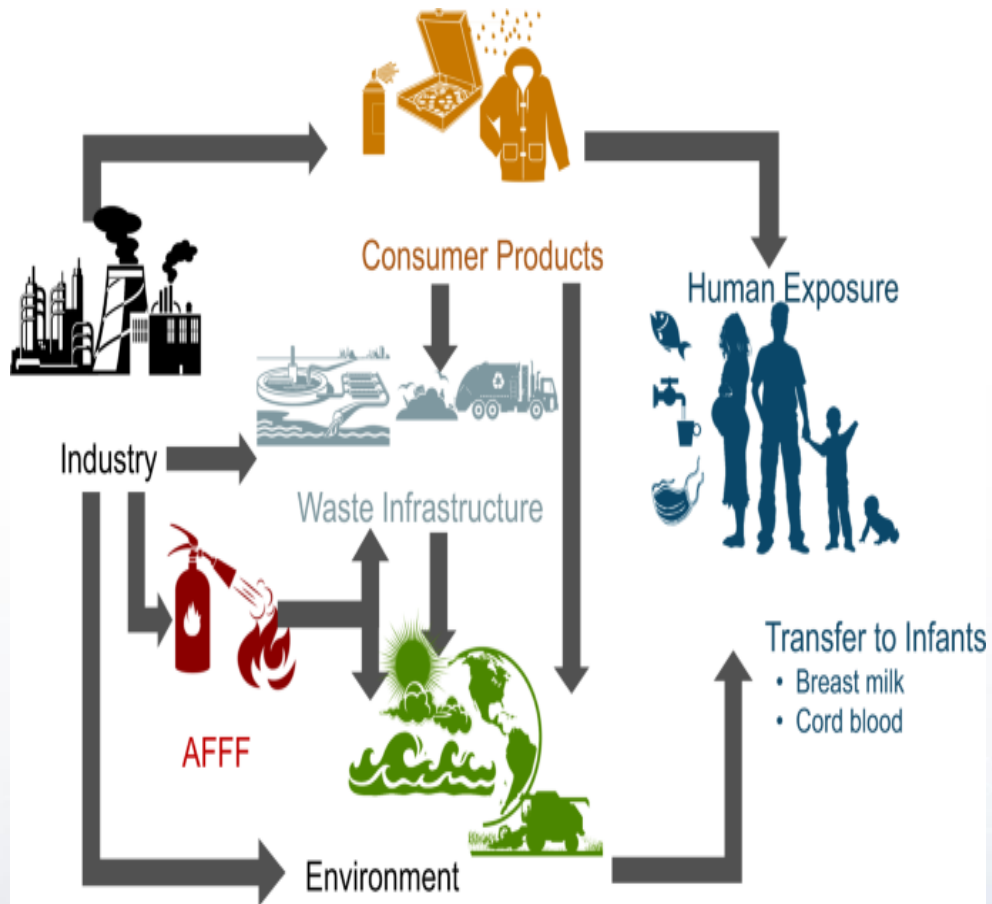


Where: PFAS Time Line 1947-present

- **1940s (post WWII):** 3M starts mass-producing PFOA
- **1950s:** DuPont starts using PFOA to make Teflon. Waterproof, or stain-resistant products using PFOA and PFOS produced—clothing, carpets..ect.
- **1960s:** Aqueous film forming foams (AFFF) — used as a firefighting foam containing PFOS and PFOA is developed
- **1970s:** AFFF used worldwide. Research finds that the PFOA and PFOS are toxic.
- **2000s:** new “short-chain” PFAS. PFOA is a “likely” human carcinogen. PFOS and PFOA are estimated to be in the blood serum of > 95% of Americans.
- **2012:** The EPA directs large public water systems to test for PFAS.
- **2016:** The EPA issues lifetime health advisory level for PFOA and PFOS in drinking water: 70 parts per trillion.
- **Present:** No Ban on PFAS production, voluntary phase out. Human exposure points: Long chain PFAS are lipophilic and thus accumulate in the fat. Short chain pass through the filters meant to filter PFAS out of drinking water



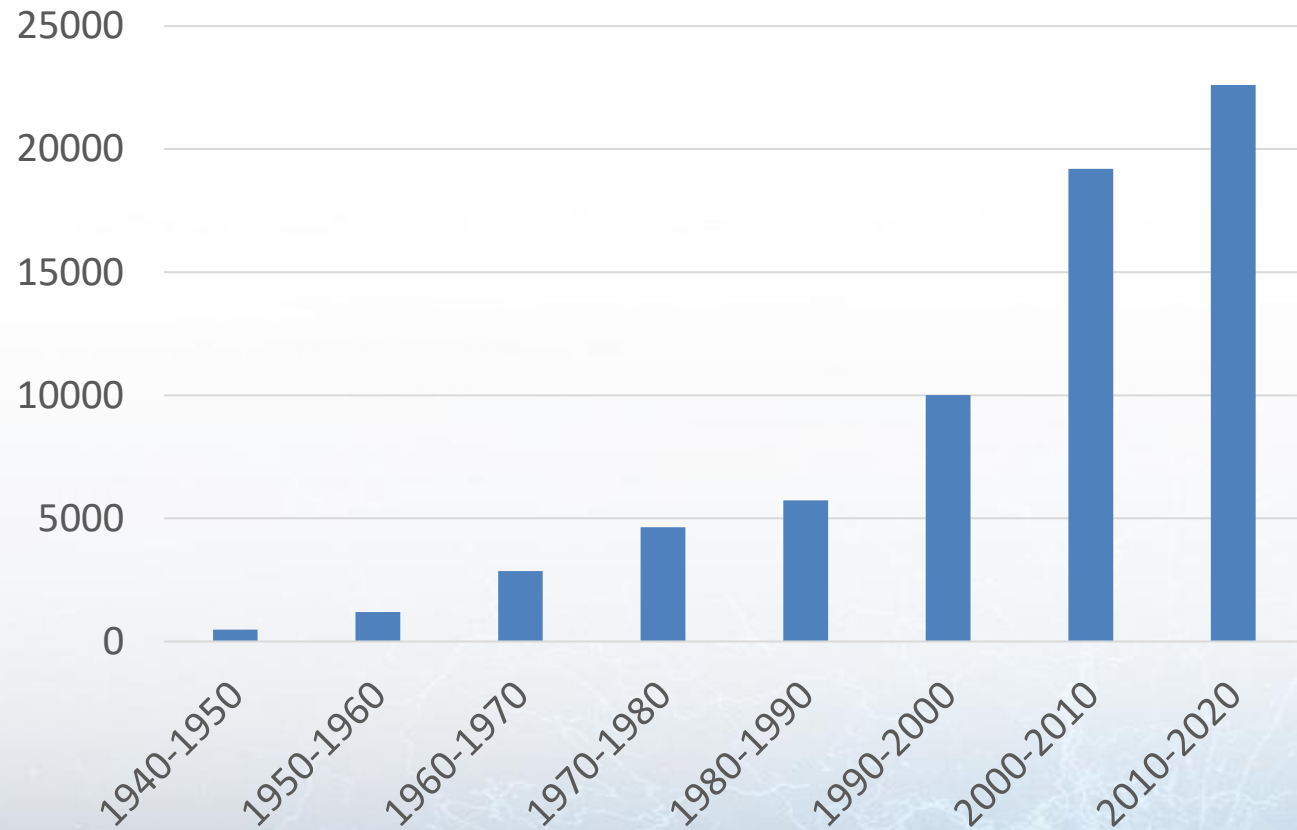
Why: PFAS Exposure Sources and Health Effects



Sunderland, E.M., Hu, X.C., Dassuncao, C. et al. *J Expo Sci Environ Epidemiol* **29**, 131–147 (2019).

Publications show increased interest in PFAS

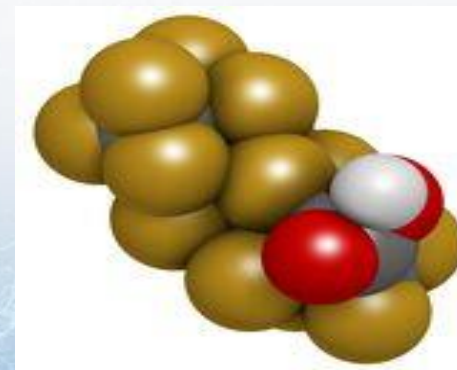
Number of PFAS publications



How: PFAS Contamination in the United States

EPA method 537 (2009) EPA method 537.1 (2018) EPA method 533 (2019)

EPA method 8327 (2019)



Potential Technical Issues

- **CONTAMINATION**

- PFASs from fluoropolymers and coatings are ubiquitous in common sampling and analytical equipment
- Laboratory contamination is almost impossible to eliminate once it happens
- Result: false positives and high reporting levels due to background

- **Recovery**

- Non-standard concentration methods may not extract all PFAS components, especially short-chain
- Long-chain compounds may stick to water sampling containers*.

	“Short Chain” PFAS	“Long Chain” PFAS
Number of units	≤7 for Carboxylates ≤6 for Sulfonates	≥8 for Carboxylates ≥7 for Sulfonates

*Powley, CR et al., 2006, Organohalogen Compounds, 68, 1688

HALO[®] PFAS Solution

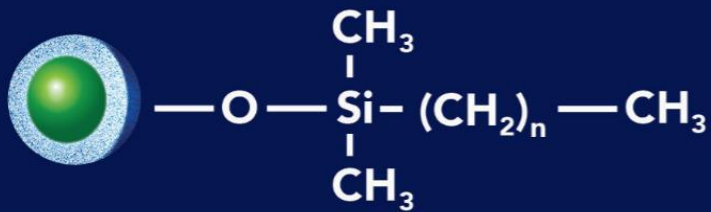


HALO[®]
PFAS Delay
Column

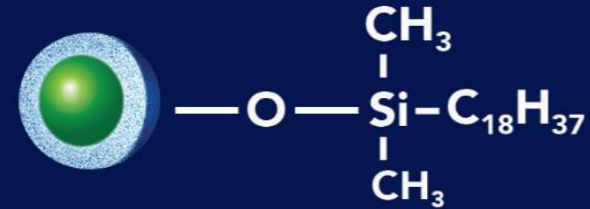


HALO[®] PFAS
Analytical Column

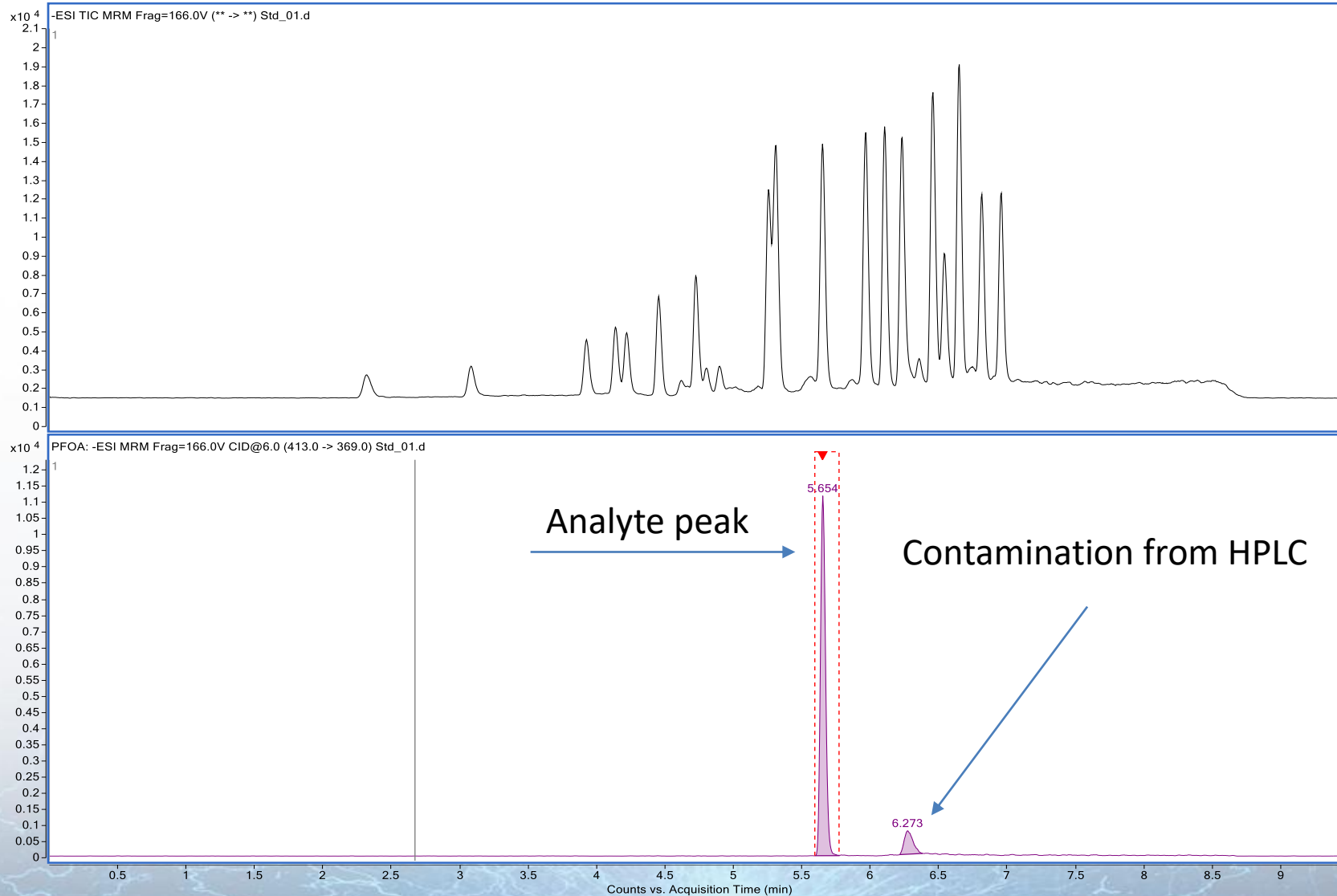
HALO[®] PFAS DELAY



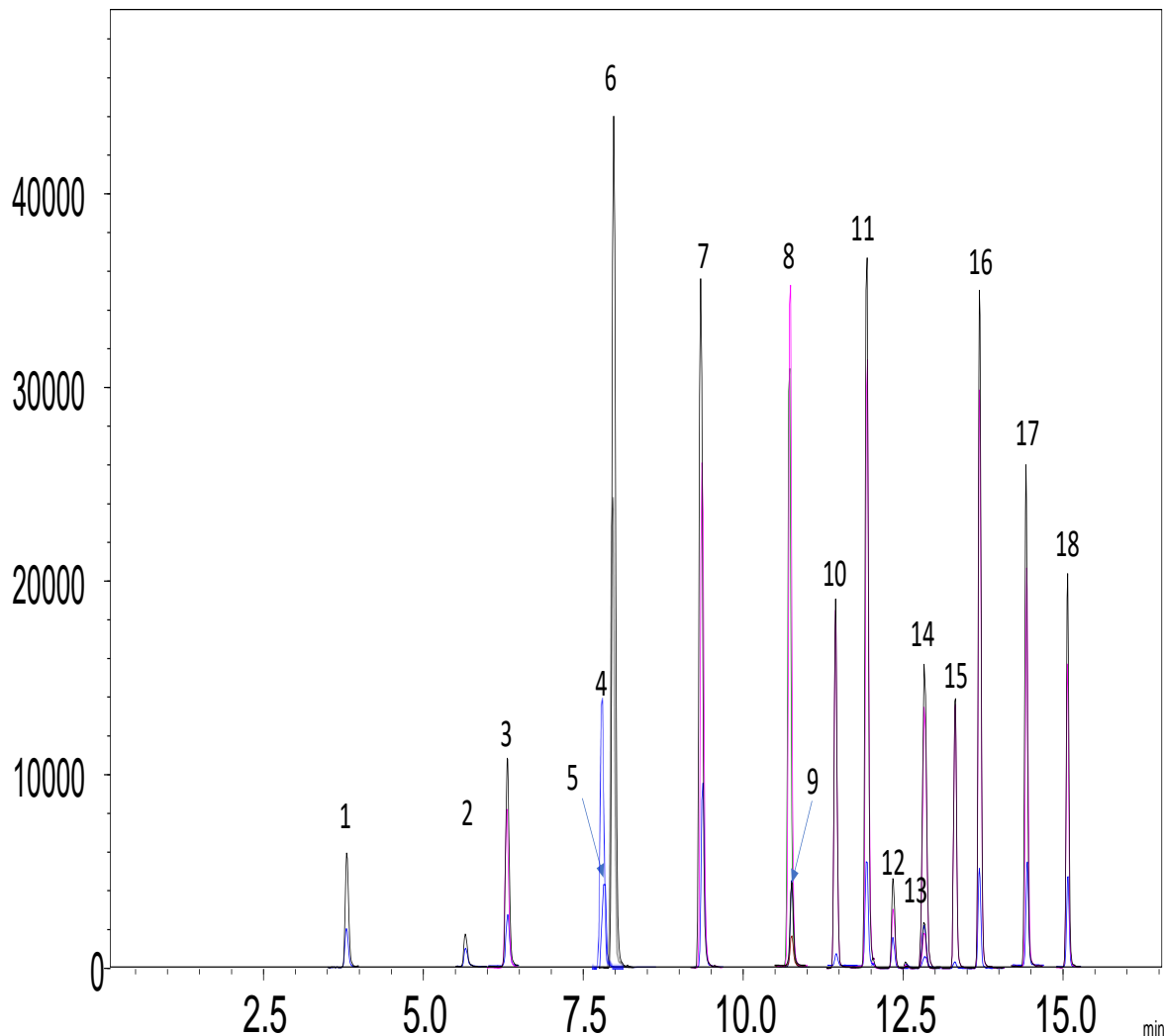
HALO[®] PFAS



Delay column effectiveness showing the delay of PFOA contamination by 0.7 minutes



EPA 537.1 - Drinking Water



Analytical Column: HALO®
 PFAS, 2.7 µm, 2.1 x 100
 mm
 Part Number: 92812-613
 Delay Column: HALO® PFAS
 Delay, 3.0 x 50 mm
 Part Number: 92113-415
 Mobile Phase A: 10 mM
 Ammonium Acetate
 Mobile Phase B: Methanol

Gradient:

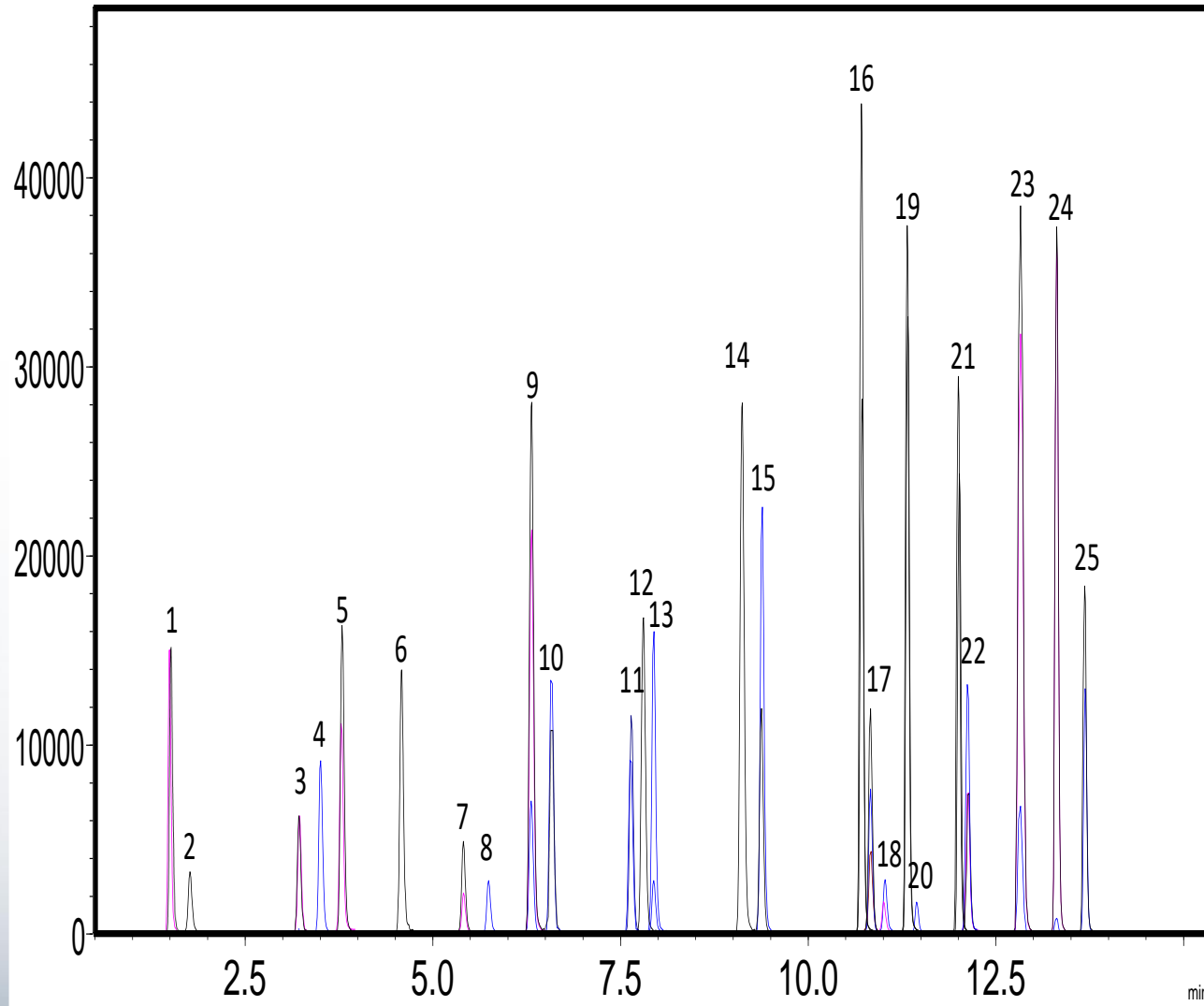
Time	%B
0.0	33
18.0	98
18.1	100
21.0	100
21.1	33
26.0	End

Flow Rate: 0.4 mL/min
 Pressure: 485 bar
 Temperature: 35 °C
 Injection Volume: 2.0 µL
 Sample Solvent: Methanol
 (96%) Water (4%)

Peak Number	Compound
1	PFBS
2	PFHxA
3	HFPO-DA
4	PFHpA
5	PFHxS
6	ADONA
7	PFOA
8	PFNA
9	PFOS
10	9Cl-PF3ONS
11	PFDA
12	N- MeFOSAA
13	PFUnA
14	N-EtFOSAA
15	11Cl- PF3OUdS
16	PFDoA
17	PFTTrDA
18	PFTeDA



EPA 533 - Short Chain PFAS, Drinking Water



Analytical Column: HALO® PFAS, 2.7 µm, 2.1 x 100 mm
 Part Number: 92812-613
 Delay Column: HALO® PFAS Delay, 3.0 x 50 mm
 Part Number: 92113-415
 Mobile Phase A: 10 mM Ammonium Acetate
 Mobile Phase B: Methanol

Gradient:

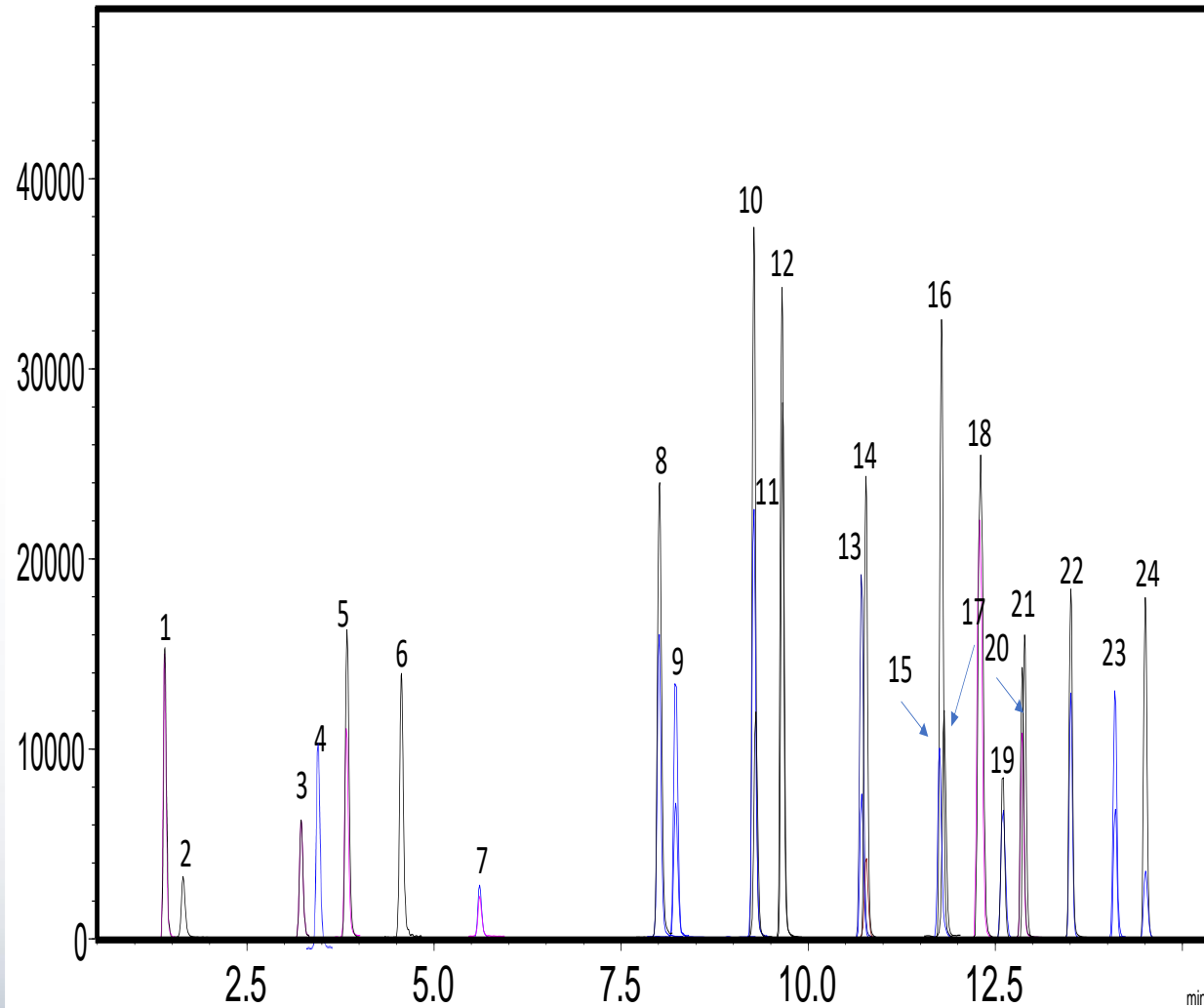
Time (min)	%B
0.0	33
18.0	98
18.1	100
21.0	100
21.1	33
26.0	End

Flow Rate: 0.4 mL/min
 Pressure: 485 bar
 Temperature: 35 °C
 Injection Volume: 2.0 µL
 Sample Solvent: Methanol (96%) Water (4%)

Peak number	Compound
1	PFBA
2	4:2FTS
3	PFPeA
4	PFBS
5	PFHpS
6	PFPeS
7	PFMPA
8	PFHxA
9	PFEESA
10	HFPO-DA
11	PFHpA
12	PFHxS
13	ADONA
14	PFOA
15	PFMBA
16	PFNA
17	PFOS
18	9Cl-PF3ONS
19	PFDA
20	8:2FTS
21	6:2FTS
22	NFDHA
23	PFUnA
24	11Cl-PF3OUds
25	PFDoA



EPA 8327 - Non Potable Water



Analytical Column: HALO®
 PFAS, 2.7 µm, 2.1 x 100
 mm
 Part Number: 92812-613
 Delay Column: HALO® PFAS
 Delay, 3.0 x 50 mm
 Part Number: 92113-415
 Mobile Phase A: 10 mM
 Ammonium Acetate
 Mobile Phase B: Methanol

Gradient:

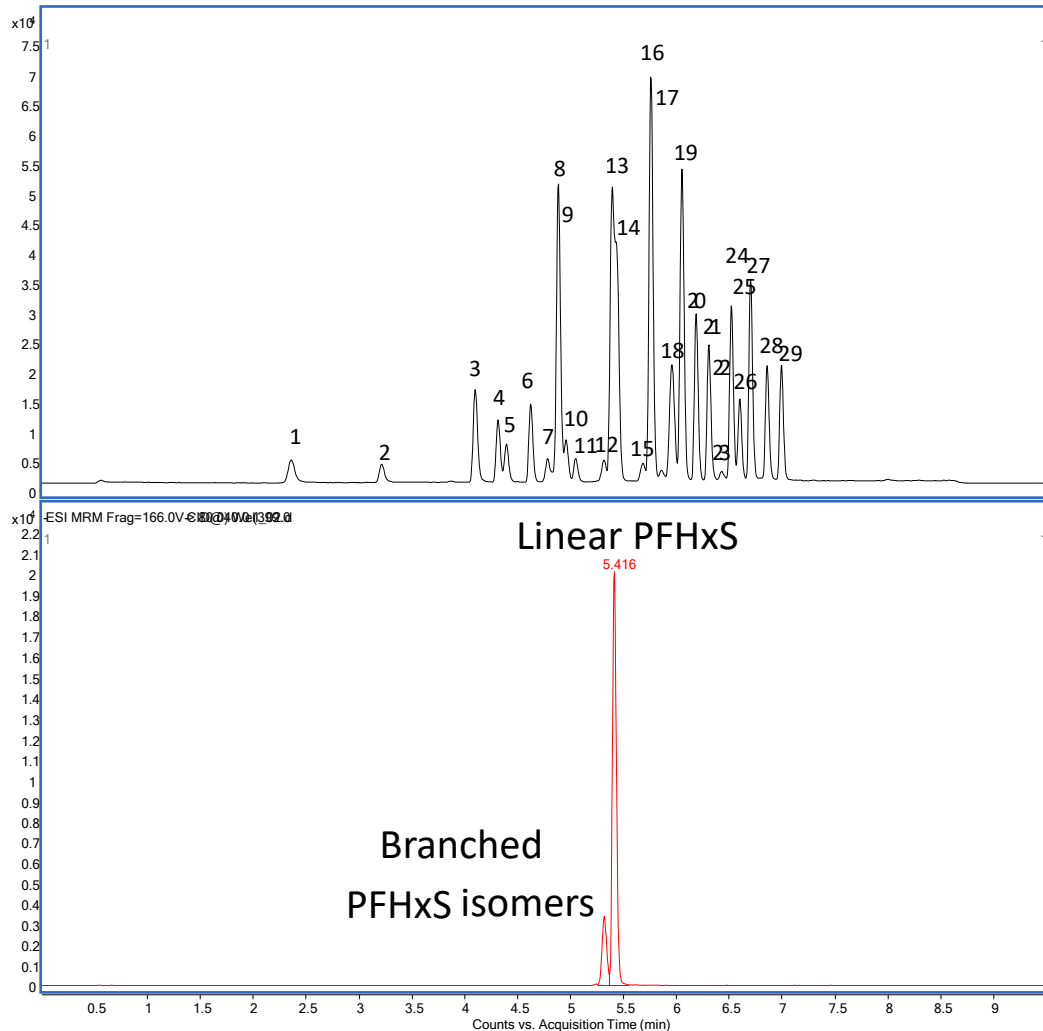
Time	%B
0.0	33
18.0	98
18.1	100
21.0	100
21.1	33
26.0	End

Flow Rate: 0.4 mL/min
 Pressure: 485 bar
 Temperature: 35 °C
 Injection Volume: 2.0 µL
 Sample Solvent: Methanol
 (96%) Water (4%)

Peak number	Compound
1	PFBA
2	4:2FTS
3	PFPeA
4	PFBS
5	PFHpS
6	PFPeS
7	PFHxA
8	PFHpA
9	PFHxS
10	FOSA
11	PFOA
12	PFDS
13	PFNA
14	PFOS
15	PFNS
16	PFDA
17	8:2FTS
18	N- MeFOSAA
19	6:2FTS
20	PFUnA
21	N-EtFOSAA
22	PFDoA
23	PFTTrDA
24	PFTeDA



Applications- Branched and Linear PFAS Isomers in Spiked Well Water

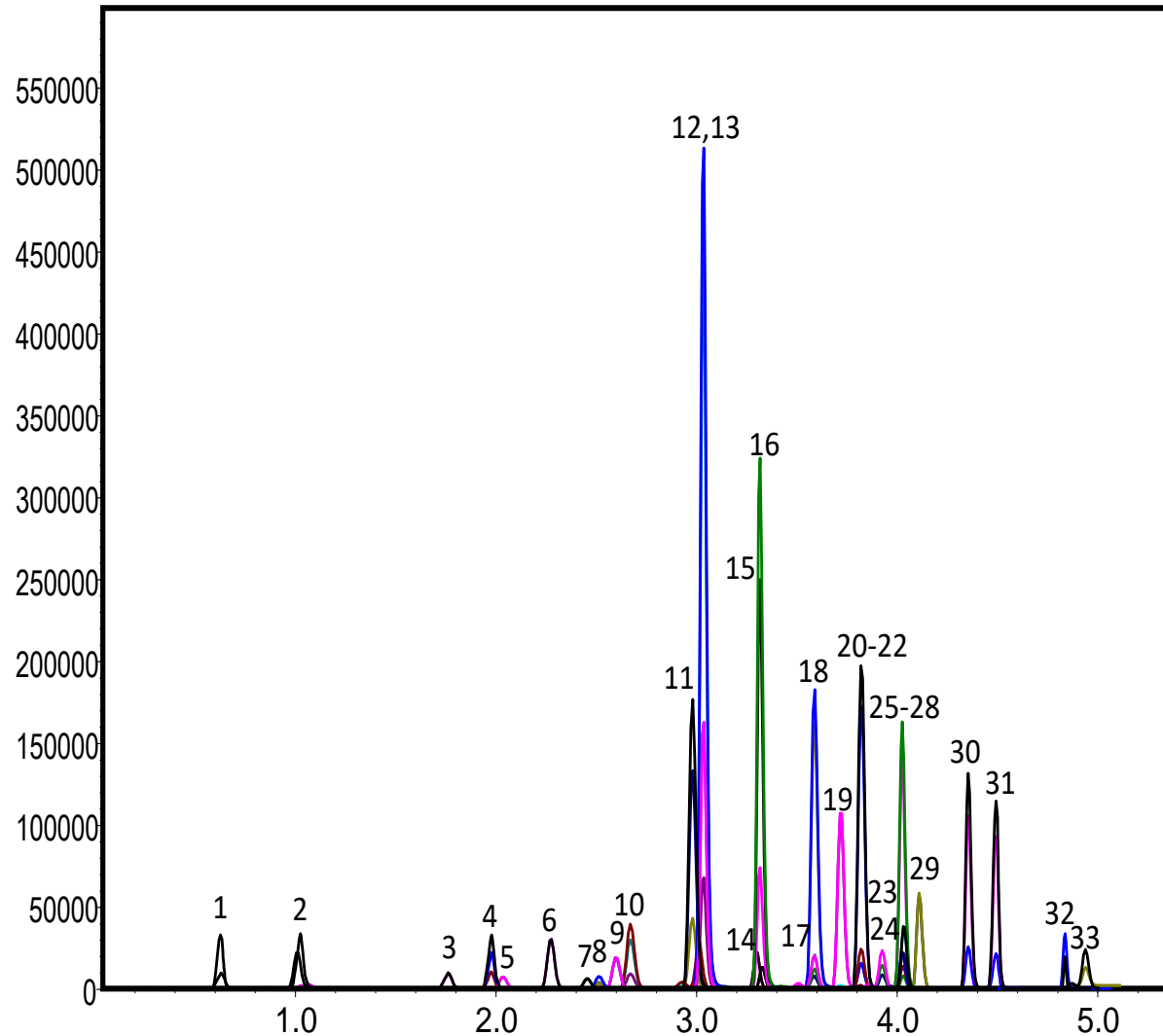


TEST CONDITIONS:
 Analytica Column:
 HALO 90 Å PFAS, 2.7
 µm 2.1 x 100 mm
 Delay Column: HALO®
 PFAS, 3.0 x 50 mm
 Part Number:
 Mobile Phase A: 20
 mM Ammonium
 Acetate
 mobile phase B:
 Methanol
 Gradient:
 Time %B
 0.0 20
 5.5 90
 7.5 90
 8.0 20
 10.5 End
 Flow Rate: 0.4 mL/min
 Pressure: 505 bar
 Temperature: 44 °C
 Detection: -ESI
 Injection Volume: 2.0
 µL
 Sample Solvent:
 Methanol (96%) Water
 (4%)
 LC System: Agilent MS
 6400

Peak number	Compound
1	PFBA
2	PFMPA
3	PFPeA
4	PFBS
5	PFMBA
6	PFEESA
7	NFDHA
8	4-2FTS
9	PFHxA
10	PFPeS
11	HFPO-DA
12	PFHpA
13	PFHxS
14	ADONA
15	6-2FTS
16	PFHpS
17	PFOA
18	PFOS
19	PFNA
20	9Cl-PF3ONS
21	PFDA
22	8-2FTS
23	NMeFOSAA
24	PFUnA
25	NEtFOSAA
26	11Cl-PF3OUdS
27	PFDoA
28	PFTTrA
29	PFTA



How Can Fused-Core® Benefit PFAS Separations? = Speed



Analytical Column:
 HALO® PFAS, 2.7 µm, 2.1 x 100 mm
 Part Number: 92812-613
 Delay Column: HALO®
 PFAS Delay, 3.0 x 50 mm
 Part Number: 92113-415
 Mobile Phase A: 10 mM
 Ammonium Acetate
 Mobile Phase B:
 Methanol

Gradient:

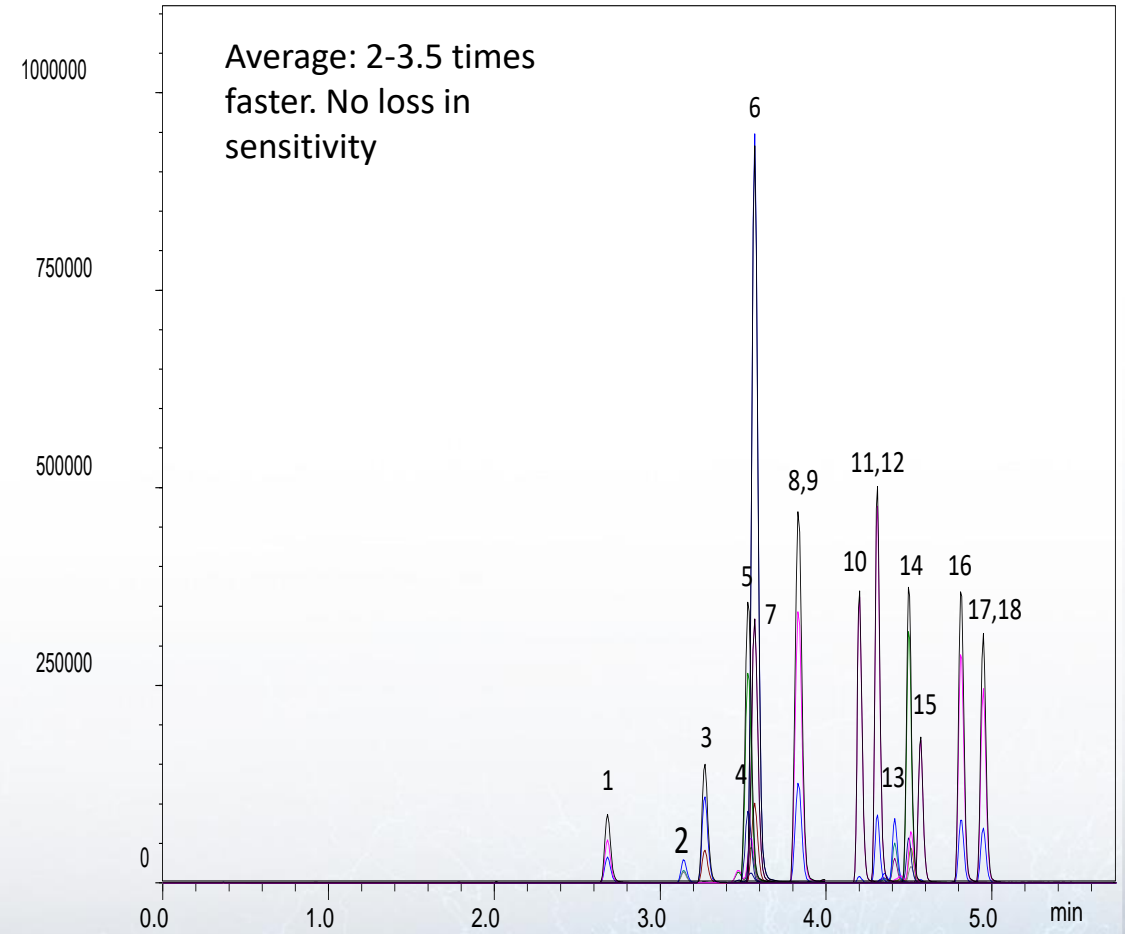
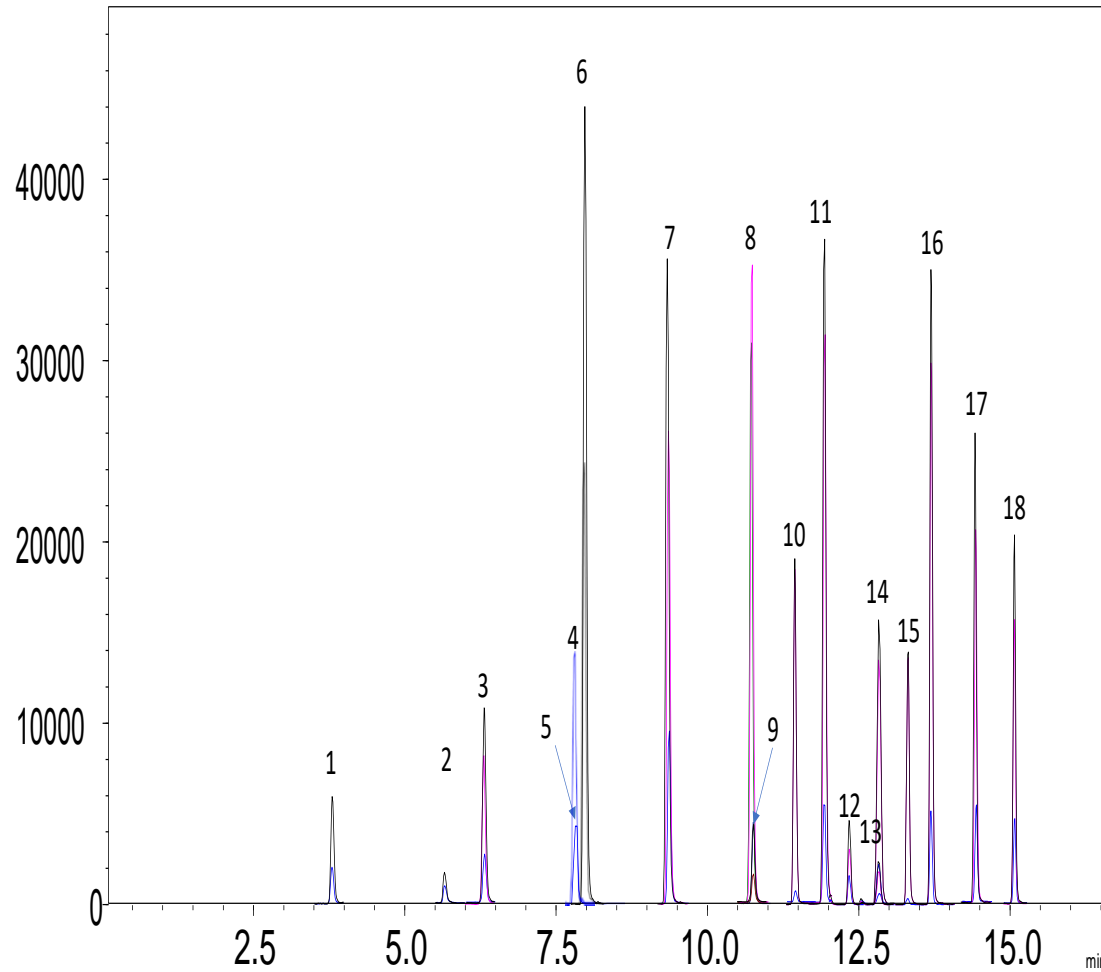
Time %B
 0.0 33
 4.0 98
 4.10 100
 6.00 100
 6.10 33
 7.50 End

Flow Rate: 0.4 mL/min
 Pressure: 389 bar
 Temperature: 35 °C
 Injection Volume: 2.0 µL
 Sample Solvent:
 Methanol (96%) Water
 (4%)

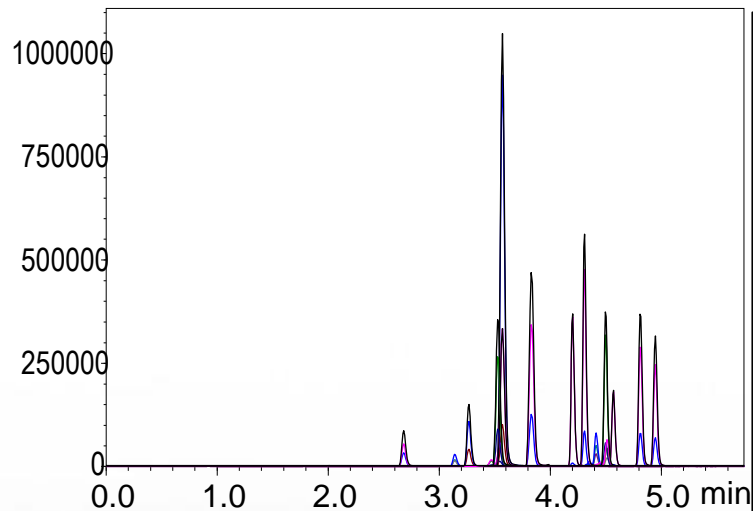
Peak Number	Compound
1	PFBA
2	4:2FTS
3	PFPeA
4	PFBS
5	PFHpS
6	PFPeS
7	PFMPA
8	PFHxA
9	PFEESA
10	HFPO-DA
11	PFHxS
12	ADONA
13	NaDONA
14	FOSA
15	PFOA
16	PFMBA
17	PFHpA
18	PFOS
19	9Cl-PF3ONS
20	8:2FTS
21	PFNS
22	PFDA
23	PFNA
24	N-MeFOSAA
25	NFDHA
26	PFUnA
27	N-EtFOSAA
28	6:2FTS
29	11Cl-PF3OUdS
30	PFTTrDA
31	PFDoA
32	PFTeDA
33	PFDS

The separation of 33 PFAS species found in EPA 537.1, EPA 533, and EPA 8327, completed in under 5 minutes.

Comparison: 18 min Gradient to 5 min Gradient for EPA 537.1



EPA 537.1 - Reproducibility



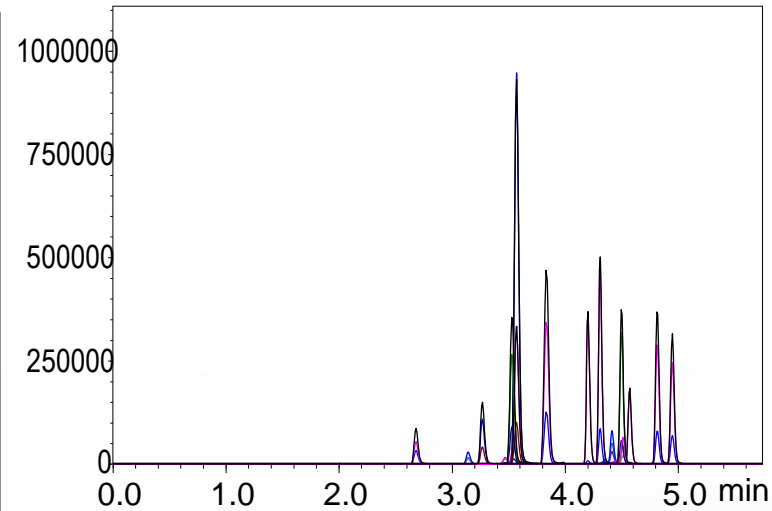
Injection 1

Peak Number	Compound	Retention Time (min)
1	PFBS	2.682
2	PFHxA	3.141
3	HFPO-DA	3.269
4	PFHpA	3.472
5	PFHxS	3.531
6	ADONA	3.569
7	PFOA	3.57
8	PFNA	3.834
9	PFOS	3.911
10	9Cl-PF3ONS	4.201
11	PFDA	4.309
12	N-MeFOSAA	4.355
13	PFUnA	4.451
14	N-EtFOSAA	4.499
15	11Cl-PF3OUdS	4.57
16	PFDoA	4.814
17	PFTTrDA	4.948
18	PFTeDA	4.978

Analytical Column:
HALO® PFAS, 2.7 µm, 2.1 x 100 mm
Part Number: 92812-613
Delay Column: HALO® PFAS Delay, 3.0 x 50 mm
Part Number: 92113-415
Mobile Phase A: 10 mM Ammonium Acetate
Mobile Phase B: Methanol

Gradient:
Time %B
0.0 33
4.0 98
4.10 100
6.00 100
6.10 33
7.50 End

Flow Rate: 0.4 mL/min
Pressure: 389 bar
Temperature: 35 °C
Injection Volume: 2.0 µL
Sample Solvent: Methanol (96%) Water (4%)



Injection 300

Peak Number	Compound	Retention Time (min)
1	PFBS	2.702
2	PFHxA	3.212
3	HFPO-DA	3.254
4	PFHpA	3.488
5	PFHxS	3.583
6	ADONA	3.590
7	PFOA	3.591
8	PFNA	3.801
9	PFOS	3.899
10	9Cl-PF3ONS	4.195
11	PFDA	4.319
12	N-MeFOSAA	4.401
13	PFUnA	4.461
14	N-EtFOSAA	4.510
15	11Cl-PF3OUdS	4.569
16	PFDoA	4.822
17	PFTTrDA	4.910
18	PFTeDA	4.931

Summary

- HALO[®] PFAS and PFAS Delay columns are application verified columns well suited for challenging environmental sample analysis.
- HALO[®] columns enable fast, efficient separations of PFAS compounds including problematic compounds like branched and linear isomers.
- The lifetime and stability of HALO[®] columns is demonstrated using the analysis of EPA 537.1 using 300 injections.

Acknowledgements

- Charles Powley – STRIDE Center for PFAS Solutions



Questions

