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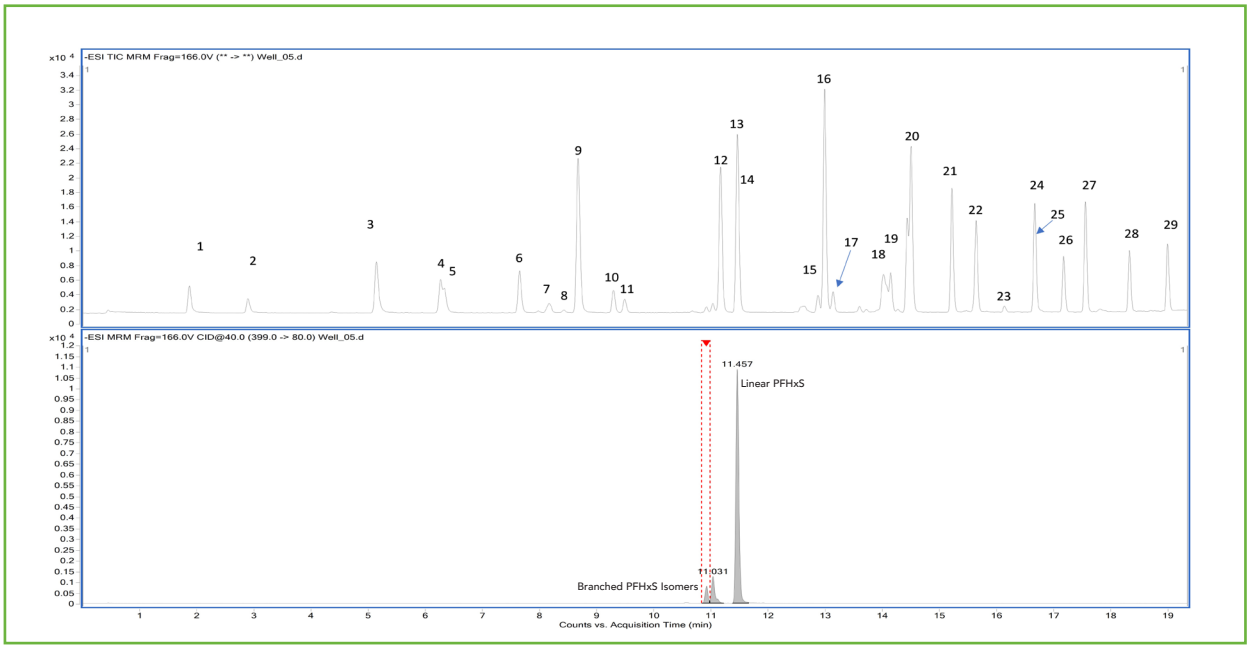
ENVIRONMENTAL

Analysis of PFAS in Well Water Spiked with Standards

243-PF



Peak #	Compound	t_R (min)	Transition	Peak #	Compound	t_R (min)	Transition
1	PFBA	1.88	213.0>169.0	16	PFOA	12.99	413.0>369.0
2	PFMPA	2.90	229.0>85.0	17	PFHpS	13.14	449.0>80.0
3	PFPeA	5.15	263.0>219.0	18	PFNA	14.43	463.0>419.0
4	PFBS	6.27	299.0>80.0	19	PFOS	14.50	499.0>80.0
5	PFMBA	6.34	279.0>85.0	20	9Cl-PF3ONS	15.22	531.0>351.0
6	PFEESA	7.66	315.0>135.0	21	8:2FTS	15.59	527.0>507.0
7	NFDHA	8.18	295.0>201.0	22	PFDA	15.64	513.0>469.0
8	4:2FTS	8.43	327.0>307.0	23	NMeFOSAA	16.13	570.0>419.0
9	PFHxA	8.67	313.0>269.0	24	NEtFOSAA	16.66	584.0>419.0
10	PFPeS	9.29	349.0>80.0	25	PFUnA	16.67	563.0>519.0
11	HFPO-DA	9.49	285.0>169.0	26	11Cl-PF3OUdS	17.17	631.0>451.0
12	PFHpA	11.17	363.0>319.0	27	PFDoA	17.55	613.0>569.0
13	PFHxS	11.46	399.0>80.0	28	PFTrA	18.32	663.0>619.0
14	ADONA	11.47	377.0>251.0	29	PFTA	18.99	713.0>669.0
15	6:2FTS	12.87	427.0>407.0				

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TEST CONDITIONS:

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: 20 mM Ammonium Acetete
B: Methanol
Gradient:

Time	%B
0.0	20
15	90
20	90

Flow Rate: 0.4 mL/min
Pressure: 505 bar
Temperature: 44 °C
Detection: -ESI MRM
Injection Volume: 2.0 µL
Sample Solvent: Methanol (96%) Water (4%)
LC System: Agilent Triple Quadrupole LC/MS 6400

MS Conditions:

Gas Temp: 130 °C
Nebulizer: 25 psi
Gas Flow: 11 L/min
Sheath Gas Heater: 250 °C
Capillary: 3500 V
Data courtesy of STRIDE Center for PFAS Solutions

In 2019 EPA method 533 was introduced and focused on "short chain" PFAS, those PFAS with carbon chain lengths of four to 12. Method 533 complements EPA Method 537.1 and can be used to test for 11 additional PFAS species. PFAS analysis, however, is an evolving area of study, and with nearly 5,000 different types of PFAS, undoubtedly more methods will be developed to include additional compounds. As PFAS science progresses, Advanced Materials Technology offers both PFAS delay and analytical columns, to further mitigate the effects of PFAS contamination from instrumentation, and provide a more accurate analysis. Here we show a clear separation of the branched and linear isomers of PFAS species PFHxS, found in a well water sample spiked with standards.

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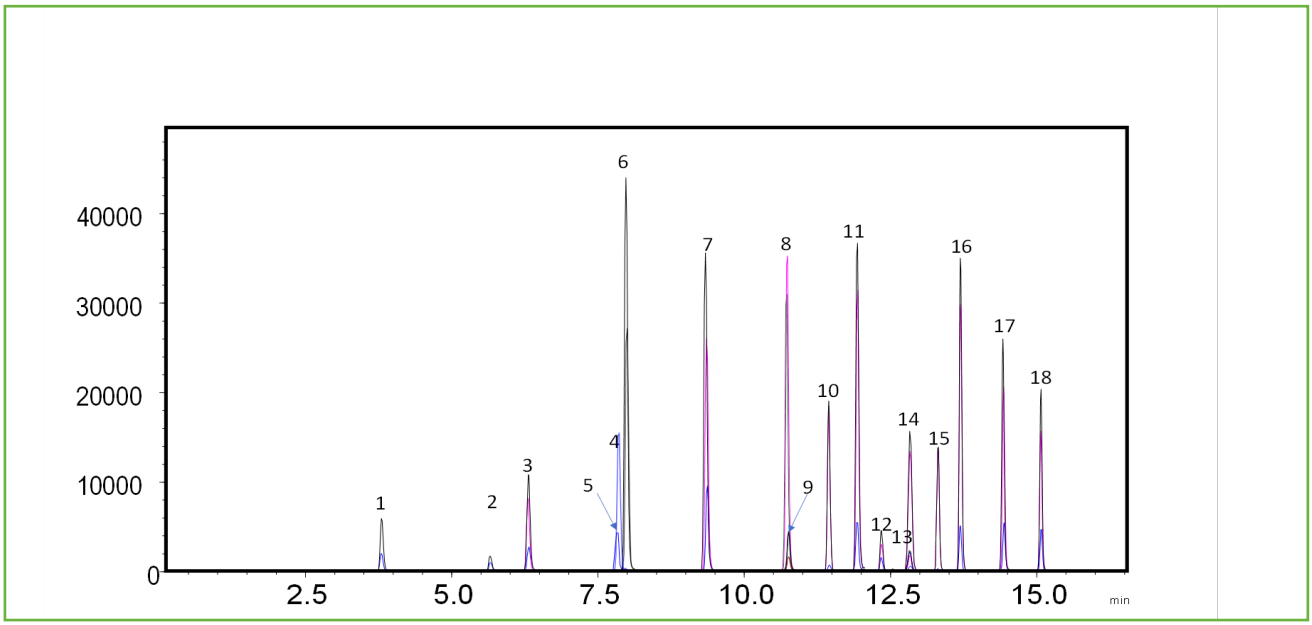
PFAS Analysis According to EPA 537.1

244-PF



Peak #	Compound	Transition	t_R (min)	Peak #	Compound	Transition	t_R (min)
1	PFBS	299.0000>80.0000	3.789	10	9Cl-PF3ONS	530.9000>351.0000	11.439
2	PFHxA	313.0000>269.0000	5.639	11	PFDA	513.0000>469.0000	11.857
3	HFPO-DA	285.0000>169.0000	6.307	12	N-MeFOSAA	570.0000>419.0000	12.336
4	PFHpA	363.0000>319.0000	7.723	13	PFUnA	563.0000>519.0000	12.822
5	PFHxS	399.0000>80.0000	7.936	14	N-EtFOSAA	584.0000>419.0000	12.827
6	ADONA	377.0000>250.9000	7.978	15	11Cl-PF3OUdS	630.7000>451.0000	13.311
7	PFOA	413.0000>369.0000	9.368	16	PFDoA	613.0000>569.0000	13.690
8	PFNA	463.0000>419.0000	10.715	17	PFTrDA	663.0000>619.0000	14.435
9	PFOS	499.0000>80.0000	10.762	18	PFTeDA	713.0000>669.0000	15.083

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TEST CONDITIONS:

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 Acetete
Mobile Phase B: Methanol

Gradient:

Time	%B
0.0	33
18	98
18.1	100
21.0	100
21.1	33
26.0	End

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

EPA Method 537.1 is used for the quantitation of 18 PFAS in drinking water, using solid phase extraction (SPE) and liquid chromatography/tandem mass spectrometry (LC/MS/MS). The method stipulates two columns be used for chromatography, one to be used as a delay column to mitigate PFAS contamination from the HPLC, and the other to be used as the analytical column and perform the separation. Here we present this high resolution separation on the HALO® PFAS delay column and the HALO® PFAS analytical column.

MS Conditions:

Detection: -ESI MS/MS
LC System: Shimadzu Nexera X2
ESI LCMS System: Shimadzu LCMS-8040
Spray Voltage: -2.0 kV
Nebulizing Gas: 2 L/min
Drying Gas: 15 L/min
DL Temperature: 250 ˚C
Heat Block: 400 ˚C

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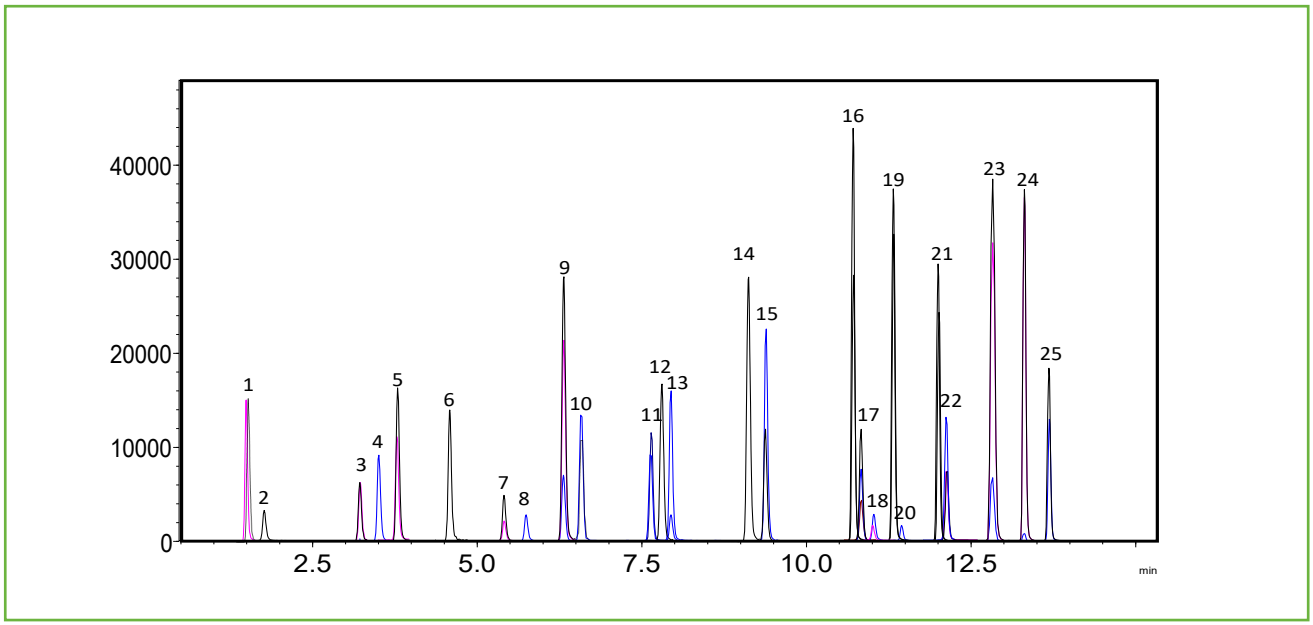
PFAS Analysis According to EPA 533

245-PF



Peak #	Compound	Transition	t_R (min)
1	PFBA	213.0000>169.0000	1.358
2	4:2FTS	229.0000>85.0000	1.890
3	PFPeA	263.0000>219.0000	3.219
4	PFBS	299.0000>80.0000	3.810
5	PFHpS	279.0000>85.0000	3.967
6	PFPeS	315.0000>135.0000	4.791
7	PFMPA	327.0000>307.0000	5.431
8	PFHxA	313.0000>269.0000	5.684
9	PFEESA	349.0000>80.0000	6.099
10	HFPO-DA	285.0000>169.0000	6.335
11	PFHpA	363.0000>319.0000	7.763
12	PFHxS	399.0000>80.0000	7.985
13	ADONA	377.0000>250.9000	8.012

Peak #	Compound	Transition	t_R (min)
14	PFOA	413.0000>369.0000	9.398
15	PFMBA	449.0000>80.0000	9.512
16	PFNA	463.0000>419.0000	10.751
17	PFOS	499.0000>80.0000	10.793
18	9Cl-PF3ONS	530.9000>351.0000	11.459
19	PFDA	513.0000>469.0000	11.885
20	8:2FTS	549.0000>80.0000	11.897
21	6:2FTS	498.0000>78.0000	12.680
22	NFDHA	599.0000>80.0000	12.847
23	PFUnA	563.0000>519.0000	12.862
24	11Cl-PF3OUdS	630.7000>451.0000	13.329
25	PFDoA	613.0000>569.0000	13.708

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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 Acetete
Mobile Phase B: Methanol

Gradient:

Time	%B
0.0	33
18	98
18.1	100
21.0	100
21.1	33
26.0	End

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

MS Conditions:

Detection: -ESI MS/MS
LC System: Shimadzu Nexera X2
ESI LCMS System: Shimadzu LCMS-8040
Spray Voltage: -2.0 kV
Nebulizing Gas: 2 L/min
Drying Gas: 15 L/min
DL Temperature: 250 ˚C
Heat Block: 400 ˚C

In 2019 EPA method 533 was introduced and focused on "short chain" PFAS, those PFAS with carbon chain lengths of four to 12. Method 533 complements EPA Method 537.1 and can be used to test for 11 additional PFAS species. Here we present this high resolution separation on the HALO® PFAS delay column and the HALO® PFAS analytical column.

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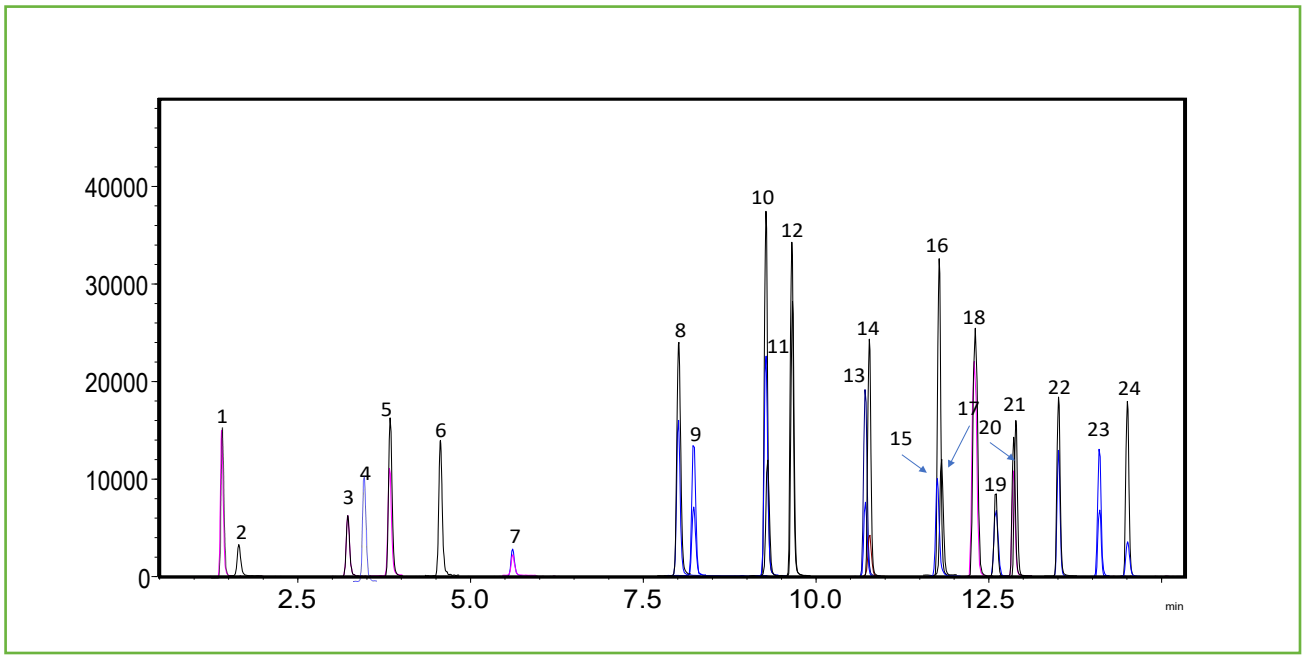
PFAS Analysis According to EPA 8327

246-PF



Peak #	Compound	Transition	t_R (min)	Peak #	Compound	Transition	t_R (min)
1	PFBA	213.0000>169.0000	1.358	13	PFNA	463.0000>419.0000	10.751
2	4:2FTS	229.0000>85.0000	1.890	14	PFOS	499.0000>80.0000	10.793
3	PFPeA	263.0000>219.0000	3.219	15	PFNS	527.0000>507.0000	11.843
4	PFBS	299.0000>80.0000	3.810	16	PFDA	513.0000>469.0000	11.885
5	PFHpS	279.0000>85.0000	3.967	17	8:2FTS	549.0000>80.0000	11.897
6	PFPeS	315.0000>135.0000	4.791	18	N-MeFOSAA	570.0000>419.0000	12.366
7	PFHxA	313.0000>269.0000	5.684	19	6:2FTS	498.0000>78.0000	12.680
8	PFHpA	363.0000>319.0000	7.763	20	PFUnA	563.0000>519.0000	12.862
9	PFHxS	399.0000>80.0000	7.985	21	N-EtFOSAA	584.0000>419.0000	12.865
10	FOSA	427.0000>407.0000	9.304	22	PFDoA	613.0000>569.0000	13.708
11	PFOA	413.0000>369.0000	9.398	23	PFTrDA	663.0000>619.0000	14.446
12	PFDS	295.0000>201.0000	9.695	24	PFTeDA	713.0000>669.0000	15.103

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TEST CONDITIONS:

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 Acetete
Mobile Phase B: Methanol

Gradient:

Time	%B
0.0	33
18	98
18.1	100
21.0	100
21.1	33
26.0	End

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

MS Conditions:

Detection: -ESI MS/MS
LC System: Shimadzu Nexera X2
ESI LCMS System: Shimadzu LCMS-8040
Spray Voltage: -2.0 kV
Nebulizing Gas: 2 L/min
Drying Gas: 15 L/min
DL Temperature: 250 ˚C
Heat Block: 400 ˚C

In 2019, the EPA validated method 8327 for non-potable water testing, which includes the analysis of 24 total PFAS compounds in a variety of aquatic matrices with 14 compounds being common across this method and EPA 537.1. Here we present this high resolution separation on the HALO® PFAS delay column coupled with the HALO® PFAS analytical column.

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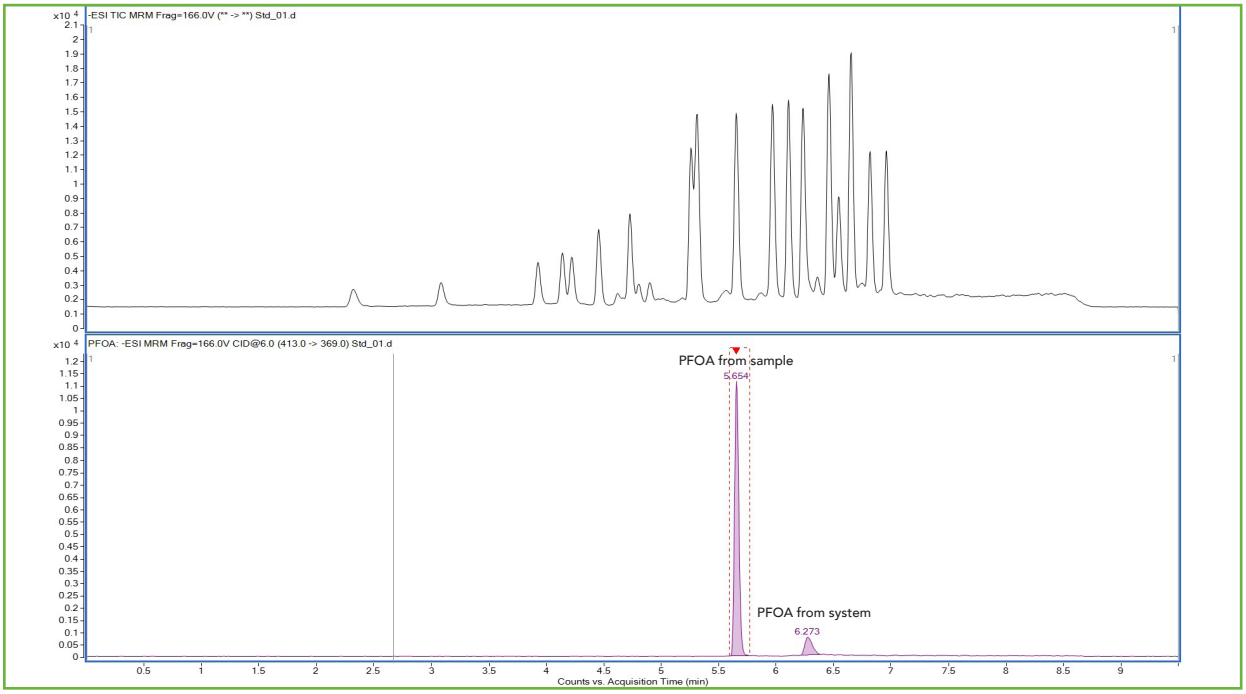
Demonstration of the HALO® PFAS Delay Column

247-PF



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Part Number: 92812-613
Delay Column: HALO® PFAS Delay, 3.0 x 50 mm
Part Number: 92113-415
Mobile Phase A: 20 mM Ammonium Acetete
B: Methanol

Gradient:

Time	%B
0.0	20
6	90
8	90
8.10	20
10.00	End

Flow Rate: 0.4 mL/min
Pressure: 505 bar
Temperature: 44 °C
Detection: -ESI MRM
Injection Volume: 2.0 µL
Sample Solvent: Methanol (96%) Water (4%)
LC System: Agilent Triple Quadrupole LC/MS 6400

MS Conditions:

Gas Temp: 130 °C
Nebulizer: 25 psi
Gas Flow: 11 L/min
Sheath Gas Heater: 250 °C
Capillary: 3500 V
Data courtesy of STRIDE Center for PFAS Solutions

Advanced Materials Technology offers both HALO® PFAS delay and analytical columns to further mitigate the effects of PFAS contamination from instrumentation, and provide a more accurate analysis. Here we show the functionality of the delay column by showing PFAS species PFOA, separated from the PFOA originating from the instrument components.

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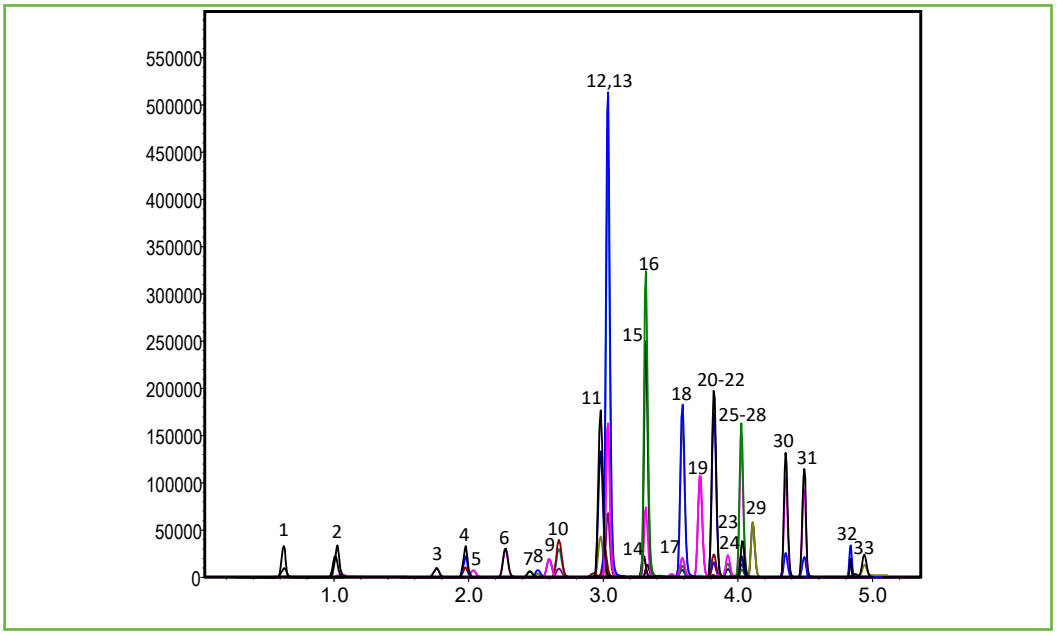
Rapid Analysis of 33 PFAS Compounds in Under 5 Minutes

248-PF



Peak #	Compound	Transition	t_R (min)
1	PFBA	213.0000>169.0000	0.755
2	4:2FTS	229.0000>85.0000	1.031
3	PFPeA	263.0000>219.0000	1.762
4	PFBS	299.0000>80.0000	1.979
5	PFHpS	279.0000>85.0000	2.035
6	PFPeS	315.0000>135.0000	2.273
7	PFMPA	327.0000>307.0000	2.454
8	PFHxA	313.0000>269.0000	2.514
9	PFEESA	349.0000>80.0000	2.599
10	HFPO-DA	285.0000>169.0000	2.670
11	PFHxS	399.0000>80.0000	3.013
12	NaDONA	377.0000>251.0000	3.033
13	ADONA	377.0000>250.9000	3.034
14	FOSA	427.0000>407.0000	3.299
15	PFOA	413.0000>369.0000	3.316
16	PFMBA	449.0000>80.0000	3.328
17	PFHpA	363.0000>319.0000	3.388

Peak #	Compound	Transition	t_R (min)
18	PFOS	499.0000>80.0000	3.588
19	9Cl-PF3ONS	530.9000>351.0000	3.719
20	8:2FTS	549.0000>80.0000	3.816
21	PFNS	527.0000>507.0000	3.820
22	PFDA	513.0000>469.0000	3.822
23	N-MeFOSAA	570.0000>419.0000	3.925
24	PFNA	463.0000>419.0000	3.942
25	NFDHA	599.0000>80.0000	4.015
26	PFUnA	563.0000>519.0000	4.025
27	N-EtFOSAA	584.0000>419.0000	4.029
28	6:2FTS	498.0000>78.0000	4.033
29	11Cl-PF3OUdS	630.7000>451.0000	4.110
30	PFTrDA	663.0000>619.0000	4.355
31	PFDoA	613.0000>569.0000	4.496
32	PFTeDA	713.0000>669.0000	4.745
33	PFDS	295.0000>201.0000	4.921

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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 Acetete
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: 479 bar
Temperature: 35 °C
Injection Volume: 2.0 µL
Sample Solvent: Methanol (96%) Water (4%)

MS Conditions:

Detection: -ESI MS/MS
LC System: Shimadzu Nexera X2
ESI LCMS System: Shimadzu LCMS-8040
Spray Voltage: -2.0 kV
Nebulizing Gas: 2 L/min
Drying Gas: 15 L/min
DL Temperature: 250 ˚C
Heat Block: 400 ˚C

As technological advancements continue to progress, mass spectrometers will continue to be improved in regards to the level of sensitivity, mass resolution, and scanning speed. This will undoubtedly impact future developments in PFAS analysis, and column performance must be able to handle these advancements. With this in mind, we developed a method for separation at maximum speed to test the suitability of the columns for use in these advanced conditions. The higher scanning speed of the MS instruments will lead to faster analysis time and higher flow rates, but a deleterious effect however, is often times an increase in the speed of analysis will lead to a decrease in the resolution therefore causing coelutions. Here we present this high resolution separation on the HALO® PFAS delay column and the HALO® PFAS analytical column for the separation of 33 PFAS species found in EPA 537.1, EPA 533, and EPA 8327, completed in under 5 minutes.

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