Supelco®
Analytical Products

Application Note

Ascentis® Express PFAS HPLC Columns

LC-MS Analysis of 33 PFAS Compounds in 5 minutes

Introduction

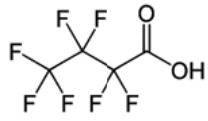
PFAS (Per- and poly-fluoroalkyl substances) are persistent, man-made organic compounds, widely found in the environment. Recent awareness has brought attention to the toxicity of these substances. The U.S. Food and Drug Administration (FDA) and the U.S. Environmental Protection Agency (EPA) have initiated actions against PFAS. For determination of PFAS, liquid chromatography–mass spectrometry (LC-MS) is a commonly used technique.

The EPA has developed, validated, and published three methods to support the analysis of 29 PFAS in drinking water, Method 533, 537 and 537.1. EPA 8327 covers the analysis of selected PFAS compounds in prepared extracts of various matrices (e.g., waters and solids) by liquid chromatography/ tandem mass spectrometry (LC/MS/MS) analysis.

As technological advancements continue to progress, mass spectrometers will continue to be improved regarding their level of sensitivity, mass resolution, and scanning speed. This will 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. However, an increase in the speed of analysis will lead to a decrease in the resolution therefore causing coelutions. The rapid separation of 33 PFAS compounds found in EPA 537.1, EPA 533, and EPA 8327 was completed in 5 minutes in this application note.

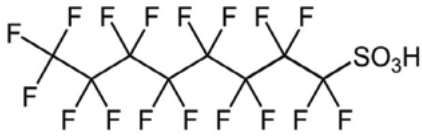
The HPLC column of choice for PFAS analysis by LC-MS/(MS) is a C18 column based on fully porous silica particles (FPP) or on superficially porous silica particles (SPP). In contrast to ordinary C18 columns, Ascentis® Express PFAS columns are tested using a PFAS compound mixture. This ensures the full suitability of the column for PFAS analysis.

The contamination of PFAS compounds from the HPLC system and materials used in analytics is a concern. Therefore, it is recommended to use a delay column, which is placed before injection in the system set-up.

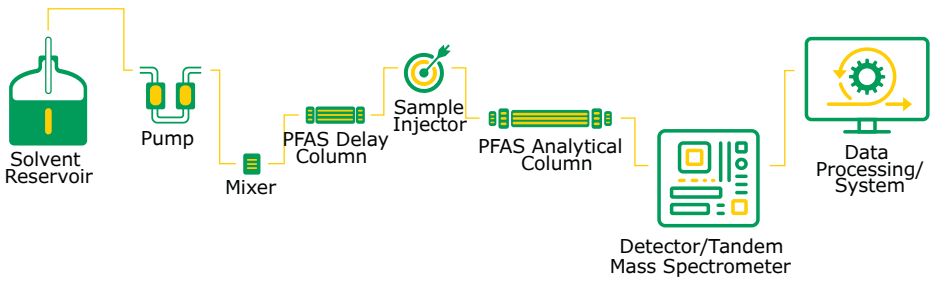


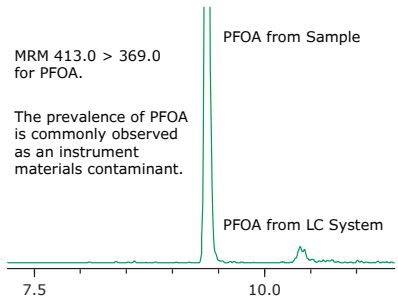
PFBA

PFOA



PFOS





The highly retentive endcapped silane of the Ascentis® Express PFAS Delay column provides high retention of PFAS compounds across various mobile phase conditions and is used to delay background instrument PFAS contamination from interference with analyzed samples. For this reason, the Ascentis® Express PFAS Delay column is placed upstream of the sample injector and after the mixer.

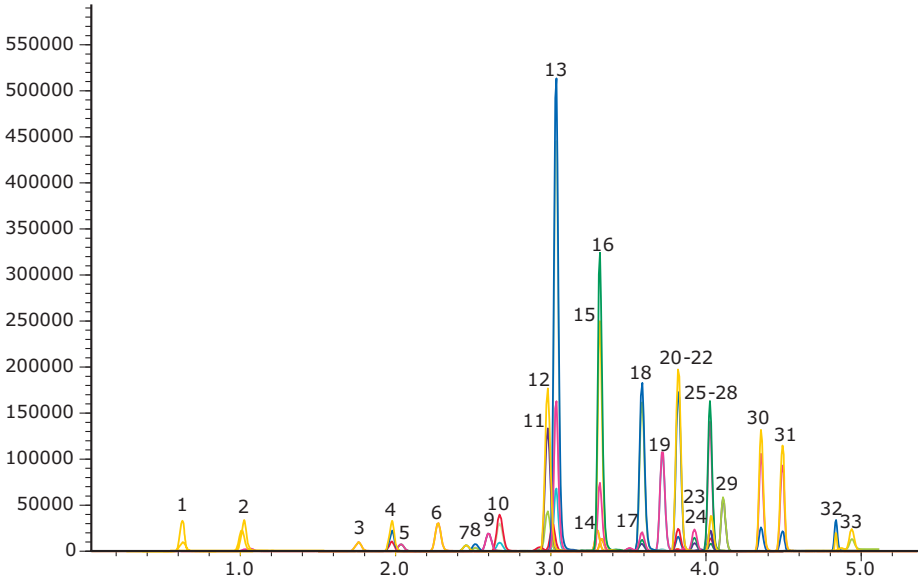
LC Conditions:	
Analytical Column:	Ascentis® Express PFAS, 2.7 µm, 10 cm x 2.1 mm, 90 Å (53559-U)
Delay Column:	Ascentis® Express PFAS Delay, 2.7 µm, 5 cm x 3 mm (53572-U)
Gradient:	

Time	%B
0.0	33.0
4.0	98.0
4.1	100.0
6.0	100.0
6.1	33.0
7.5	End

Mobile Phase A:	10 mM Ammonium Acetate
Mobile Phase B:	Methanol
Flow Rate:	0.4 mL/min
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 temp:	250 °C
Heat Block:	400 °C

Analysis of 33 PFAS Compounds in Under 5 Minutes



Peak #	Compound	Transition	tR (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
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

Conclusion

The new Ascentis® Express PFAS HPLC column allows the highly efficient separation of 33 PFAS compounds in 5 minutes, and it is equally adept at delaying PFAS contamination originating from the instrument by using the Ascentis® Express PFAS Delay column.

This application note demonstrates that the Fused-Core® technology of Ascentis® Express PFAS HPLC columns benefits PFAS analysis for fast, efficient, and rugged separations which are paramount to environmental analysis.

Product list	Cat. No
Ascentis® Express PFAS, 2.7 µm, 10 cm x 2.1 mm, 90 Å	**53559-U**
Ascentis® Express PFAS Delay, 2.7 µm, 5 cm x 3 mm	**53572-U**
Methanol for chromatography (LC-MS grade) LiChrosolv®	**1.06035**
Water for chromatography (LC-MS grade) LiChrosolv® or ultrapure water from a Milli-Q® IQ 7 series water purification system	**1.15333 or ZIQ7005T0**
Ammonium acetate suitable for mass spectrometry (MS), LiChropur™, eluent additive for LC-MS	**73594**

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