APPLICATIONS

EPA Method 537.1: PFAS in Drinking Water Using Strata® SDB-L Solid Phase Extraction (SPE) and a Luna® Omega 1.6 µm PS C18 UHPLC Column

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Overview

This application demonstrates the suitability and advantage of using Strata SDB-L SPE along with a Luna Omega 1.6 µm PS C18 UHPLC column in the performance of EPA Method 537.1, the official SPE liquid chromatography/tandem mass spectrometry (LC-MS/MS) method for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in drinking water in the United States.

Introduction

EPA Method 537 Version 1.1, was first published in 2009 for use in the Third Unregulated Contaminant Rule (UCMR3) nationwide drinking water survey. This original PFAS method specified 14 target PFAS analytes, including Perfluorooctanesulfonic acid (PFOS) and perfluorooctanoic acid (PFOA), the two PFAS compounds of largest historic use. However, both the production and use of PFOS and PFOA were discontinued between 2000 and 2015 and were replaced by shorter chain PFAS compounds (informally referred to as the "GenX" compounds), which were thought to be less persistent in the environment and less toxic. Therefore, EPA Method 537.1 was introduced in 2018 as an update to EPA Method 537 Version 1.1. It included the original 14 PFAS analytes and added 4 of the shorter chain PFAS compounds for a total of 18 analytes. As originally published, EPA Method 537.1 specified that only SPE cartridges based upon SDVB (styrenedivinylbenzene) polymers could be used in the extraction procedure, owing to low recovery of the short chain PFAS compounds on non-SDVB polymers. This stipulation was continued in the most recent update: Method 537.1 V2, published in March, 2020.[1]

In this technical note we present analytical results for the analysis of drinking water by EPA Method 537.1 using Strata SDB-L SPE (based upon a SDVB polymer) and a Luna Omega 1.6 µm PS C18 UHPLC column. The data demonstrates excellent recovery for all 18 PFAS analytes on Strata SDB-L. Likewise, Luna Omega 1.6 µm PS C18 provides outstanding column efficiency and analyte resolution for greater method sensitivity and shorter run times.

Materials and Methods

Solid Phase Extraction Protocol

Following the procedures of EPA Method 537.1, V2, Sections 6.9 - 6.11 and 11.3 - 11.4

Cartridge: Strata SDB-L, 500 mg/6 mL
Part No.: 8B-S014-HCH
Load: 250 mL sample that has been fortified with surrogates
Elution: 2x 3 mL Methanol
Dry Down: With Nitrogen in a heated water bath
Reconstitute: Adjust final volume to 1 mL with 96:4 Water:Methanol (v/v) and add internal standards

HPLC Conditions

Following the procedures of EPA Method 537.1, V2, Sections 6.9 - 6.11 and 11.3 - 11.4

Column: Luna Omega 1.6 µm PS C18
Dimension: 100 x 2.1 mm
Part No.: 00D-4752-AN
Mobile Phase: A: 0.1% Acetic acid in Water
B: Methanol
Gradient:

Time (min)	%B
0	20
0.5	30
7	90
7.5	100
9	100

Flow Rate: 0.7 mL/min
Injection Volume: 4 µL
LC System: Agilent® 1260 Series HPLC
Detection: Agilent Ultivo™ Triple Quadrupole MS

Data and Results

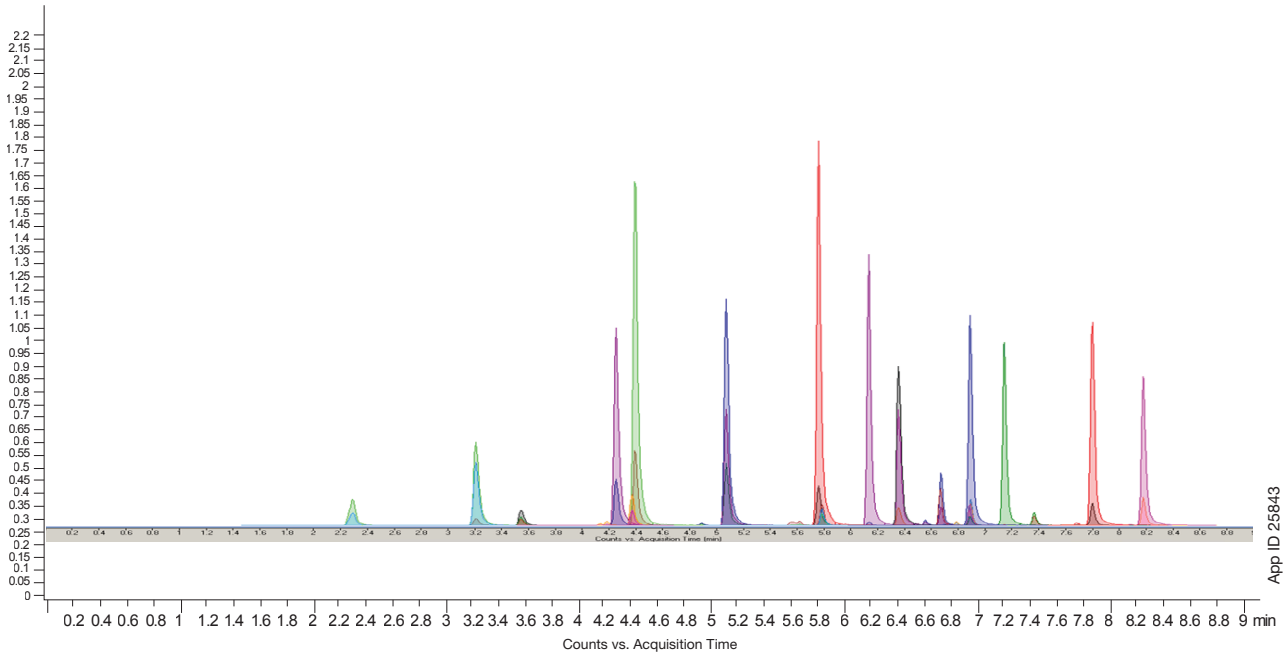
PFAS Target Analytes and UHPLC Retention Times

Analyte	RT (min)	Internal Standard
PFBS	2.29	13C4 -PFOS
PFHxA	3.20	13C2 -PFOA
HFPO-DA	3.55	13C2 -PFOA
PFHpA	4.24	13C2 -PFOA
PFHxS	4.39	13C4 -PFOS
ADONA	4.41	13C2 -PFOA
PFOA	5.08	13C2 -PFOA
PFOS	5.72	13C4 -PFOS
PFNA	5.77	13C2 -PFOA
9Cl-PF3ONS	6.15	13C4 -PFOS
PFDA	6.35	13C2 -PFOA
NMeFOSAA	6.70	d3 -NMeFOSAA
PFUnA	6.83	13C2 -PFOA
NEtFOSAA	6.88	d3 -NMeFOSAA
11Cl-PF3OUdS	7.17	13C4 -PFOS
PFDoA	7.37	13C2 -PFOA
PFTrDA	7.80	13C2 -PFOA
PFTA	8.18	13C2 -PFOA

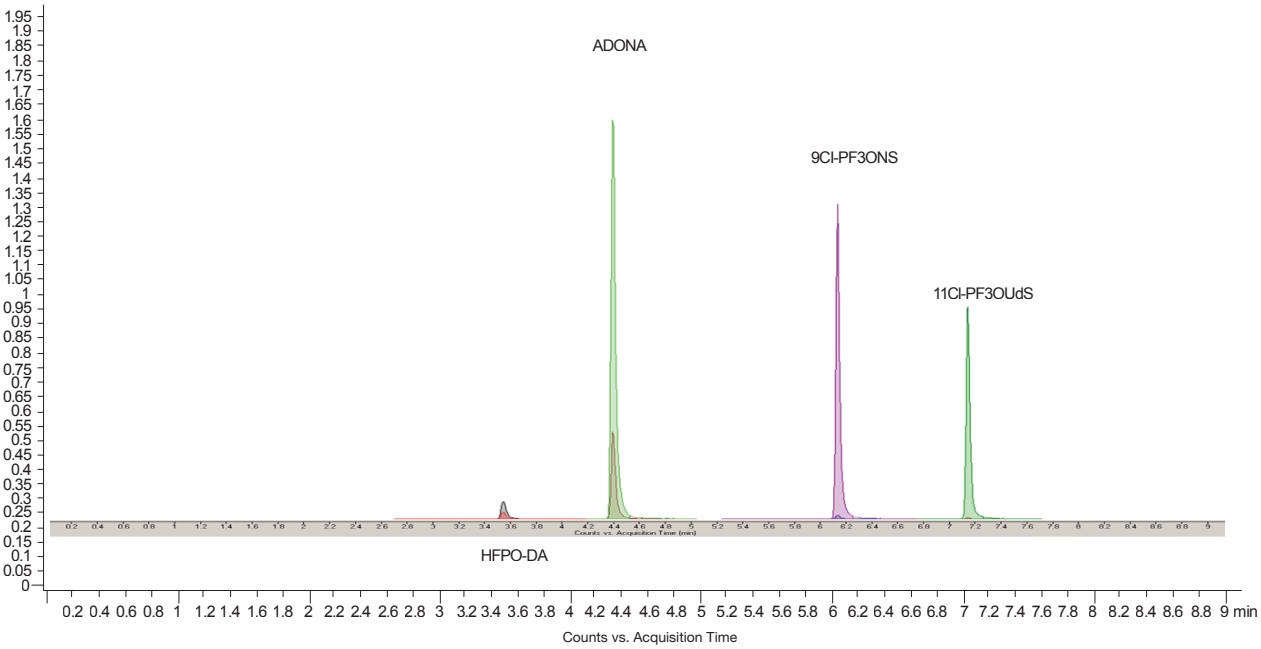
APPLICATIONS

Full PFAS Target Analytes

PFAS Replacement Compounds



Short Chain ("Gen X") PFAS Analytes



Method Precision and Accuracy from the Analysis of 50 Consecutive Laboratory Control Samples (LCS)

	Mean % Recovery	Standard Deviation
13C2-PFDA	106	15.8
13C2-PFHxA	108	18.2
d5-EtFOSAA	104	19.8
13C2-HFPO-DA	104	17.1
11Cl-PF3OUdS	105	10.9
9Cl-PF3OUNS	104	11.9
ADONA	103	13.6
Et-FOSAA	111	13.8
HFPO-DA	104	15.6
Me-FOSAA	113	18.5
PFBS	104	14.7
PFDA	106	12
PFDoA	105	17.4
PFHpA	111	14.8
PFHxA	109	14.1
PFHxS	108	15.1
PFNA	109	12.7
PFOA	109	12.5
PFOS	111	13
PFTeDA	103	14.6
PFTrDA	104	13.7
PFUnA	107	13.7

Discussion

These results fully demonstrate the suitability of the combination of Strata® SDB-L 500 mg/6 mL and Luna® Omega 1.6 µm PS C18 for use in EPA Method 537.1. Luna Omega 1.6 µm PS C18 provides excellent separation of all analytes, including the shorter chain "Gen X" compounds. The accuracy and precision of the data, as demonstrated by the analysis of 50 consecutive LCS samples, are well within the requirements of the method. However, beyond meeting method requirements, the additional advantage of the Strata/Luna combination is its contribution to environmental laboratory productivity. In the published version of EPA Method 537.1, the run time for the 25 analytes (18 target analytes and 7 internal standards) was 25 minutes. In the data presented here, the equivalent run time is 8.5 minutes. Which represents a nearly 3-fold productivity increase in the chromatographic step compared to the method as originally published. This illustration of EPA Method 537.1 suitability and productivity demonstrates why the combination of Strata SDB-L and Luna Omega 1.6 µm PS C18 has become the environmental testing industry's go-to approach for PFAS drinking water analysis, regardless of the instrumentation platform used.

Conclusion

EPA Method 537.1 is the official US regulatory method to be used by environmental laboratories to quantitate PFAS in drinking water. Health advisory drinking water limits have been established for PFOS and PFOA have been established at 70 µg/L and it is widely expected that official drinking water limits will shortly be promulgated for these two compounds, and for additional PFAS in the near future. As drinking water utilities and water resource agencies opt (or are required) to routinely test for PFAS in drinking water or water supplies, EPA 537.1 is destined to transition from its prior status as an exotic analytical method to a common environmental laboratory test. The Strata/Luna combination is already widely used in EPA Method 537.1 testing in the United States owing to the combination of high accuracy and precision and reduced analysis time. These properties will allow for higher sample throughput at lower detection levels as PFAS water testing becomes ever more prevalent.

Acknowledgements

We would like to acknowledge the invaluable assistance of Weck Laboratories in demonstrating the suitability of Strata SDB-L and Luna Omega 1.6 µm PS C18 for this essential environmental application.

References

1. https://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=348508&Lab=CESER&simpleSearch=0&showCriteria=2&searchAll=537.1&TIMSType=&dateBeginPublishedPresented=03%2F24%2F2018

Strata® SDB-L SPE Ordering Information

Format	Sorbent Mass	Part Number	Unit
Tube			
	100 mg	8B-S014-EAK	1 mL (100/box)
	200 mg	8B-S014-FBJ	3 mL (50/box)
	200 mg	8B-S014-FCH	6 mL (30/box)
	500 mg	8B-S014-HBJ	3 mL (50/box)
	500 mg	8B-S014-HCH	6 mL (30/box)
	1 g	8B-S014-JCH	6 mL (30/box)
Giga™ Tube			
	10 g	8B-S014-MFF	60 mL (16/box)

Luna® Omega PS C18 Ordering Information

1.6 µm Microbore Columns (mm)		
Phases	**50 x 1.0**	**100 x 1.0**
PS C18	00B-4752-A0	00D-4752-A0

1.6 µm Minibore Columns (mm)					SecurityGuard™ ULTRA Cartridges‡
Phases	**30 x 2.1**	**50 x 2.1**	**100 x 2.1**	**150 x 2.1**	**3/pk**
PS C18	00A-4752-AN	00B-4752-AN	00D-4752-AN	00F-4752-AN	AJ0-9508
					for 2.1 mm ID

3 µm Capillary Columns (mm)							Trap Column
Phases	**50 x 0.30**	**100 x 0.30**	**150 x 0.30**	**50 x 0.50**	**100 x 0.50**	**150 x 0.50**	**20 x 0.30**
PS C18	00B-4758-AC	00D-4758-AC	00F-4758-AC	00B-4758-AF	00D-4758-AF	00F-4758-AF	05M-4758-AC

3 µm Minibore and MidBore™ Columns (mm)								SecurityGuard Cartridges (mm)
Phases	**30 x 2.1**	**50 x 2.1**	**100 x 2.1**	**150 x 2.1**	**50 x 3.0**	**100 x 3.0**	**150 x 3.0**	**4 x 2.0***
								/10 pk
PS C18	00A-4758-AN	00B-4758-AN	00D-4758-AN	00F-4758-AN	00B-4758-Y0	00D-4758-Y0	00F-4758-Y0	AJ0-7605
								for ID: 2.0-3.0 mm

3 µm Analytical Columns (mm)					SecurityGuard Cartridges (mm)
Phases	**50 x 4.6**	**100 x 4.6**	**150 x 4.6**	**250 x 4.6**	**4 x 3.0***
					/10 pk
PS C18	00B-4758-E0	00D-4758-E0	00F-4758-E0	00G-4758-E0	AJ0-7606
					for ID: 3.2-8.0 mm

5 µm Minibore and MidBore™ Columns (mm)							SecurityGuard Cartridges (mm)
Phases	**50 x 2.1**	**100 x 2.1**	**150 x 2.1**	**50 x 3.0**	**100 x 3.0**	**150 x 3.0**	**4 x 2.0***
							/10 pk
PS C18	00B-4753-AN	00D-4753-AN	00F-4753-AN	00B-4753-Y0	00D-4753-Y0	00F-4753-Y0	AJ0-7605
							for ID: 2.0 - 3.0 mm

5 µm Analytical Columns (mm)					SecurityGuard Cartridges (mm)
Phases	**50 x 4.6**	**100 x 4.6**	**150 x 4.6**	**250 x 4.6**	**4 x 3.0***
					/10 pk
PS C18	00B-4753-E0	00D-4753-E0	00F-4753-E0	00G-4753-E0	AJ0-7606
					for ID: 3.2-8.0 mm

5 µm Semi-Preparative Columns (mm)		SecurityGuard Cartridges (mm)
Phases	**250 x 10**	**10 x 10****
		/3 pk
PS C18	00G-4753-N0	AJ0-9520
		for ID: 9-16 mm

5 µm Axia™ Packed Preparative Columns (mm)						SecurityGuard Cartridges (mm)	
Phases	**150 x 21.2**	**250 x 21.2**	**150 x 30**	**250 x 30**	**250 x 50**	**15 x 21.2****	**15 x 30.0♦**
						/ea	/ea
PS C18	00F-4753-P0-AX	00G-4753-P0-AX	00F-4753-U0-AX	00G-4753-U0-AX	00G-4753-V0-AX	AJ0-7608	AJ0-7609
						for ID: 18-29 mm	for ID: 30-49 mm

‡ SecurityGuard ULTRA Cartridges require holder, Part No.: AJ0-9000
* SecurityGuard Analytical Cartridges require holder, Part No.: KJ0-4282
***SemiPREP SecurityGuard Cartridges require holder, Part No.: AJ0-9281
**PREP SecurityGuard Cartridges require holder, Part No.: AJ0-8223
♦ PREP SecurityGuard Cartridges require holder, Part No.: AJ0-8277

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