

APPLICATIONS

Extraction and Analysis of GenX, a Novel PFOA-Replacement Compound, from Water by LC-MS/MS

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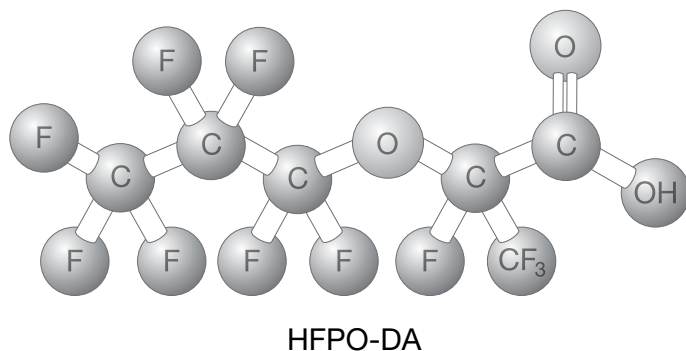
Background

GenX is an emerging per- and poly-fluorinated alkyl substance (PFAS) and the objective of this study was to include Gen-X to a multi-components PFAS method. GenX and several PFOS-replacement compounds (e.g. DONA) were optimized on the SCIEX Triple Quad™ 4500 MS. An MDL study showed that quantitation of GenX, along with other PFASs, can be achieved at approximately 50 ng/L in water samples.

Introduction

Per- and poly-fluorinated alkyl substances (PFAS) are widespread environmental contaminants found in soil, air, biota, and water. Over the past few years, there has been an increased interest in a small group of PFASs including perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) to a broad number of compounds representing multiple classes. Most recently, public attention has shifted towards several new classes of PFAS compounds found from drinking water in North Carolina. One of these commercial mixtures is known as GenX and the main component is the dimer acid of hexafluoropropyleneoxide (HFPO-DA). Because HFPO-DA exhibits similar chemical characteristics to other PFASs, the goal of this method was to analyze HFPO-DA in a multi-residue method along with 24 other common PFASs.

Figure 1.
Structure of HFPO-DA



Experimental Conditions

Sample Preparation:

All standards including HFPO-DA and its stable isotope labelled surrogate 13C3-HFPO-DA were purchased from Wellington Laboratories (Guelph, Ontario). 200 mL water samples were extracted using Strata®-XL-AW weak anion-exchange SPE cartridges following the conditions in the ISO Standard 25101. Samples were also passed through Strata GCB following the requirements of DOD QSM 5.1. The final volume of the SPE eluent was 8 mL. Final extracts consisted of 80 % methanol and 20 % water.

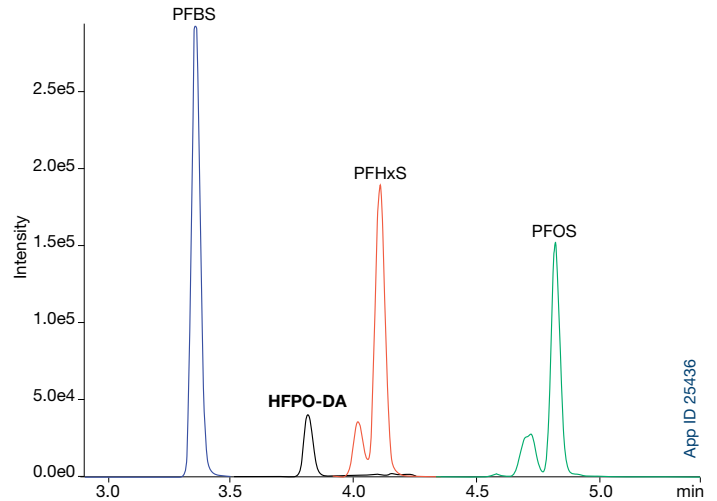
Solid Phase Extraction Protocol

Cartridge:	Strata XL-AW 500 mg/6 mL
Part No.:	8B-S051-HCH
Condition:	6 mL 0.3% Ammonium hydroxide in Methanol, followed by 6 mL Methanol
Equilibrate:	6 mL Water
Load:	Add sample at about 10 mL/min Note: use an adapter cap (Part No.: AH0-7191) and large volume sample reservoir (Part No.: AH0-7005)
Wash:	2x 6 mL Water
Elute:	10 mL 0.3% Ammonium hydroxide in Methanol (this 10 mL will elute PFAS/GenX analytes)
Load:	Pass SPE eluent through Strata GCB 250 mg/6 mL (8B-S528-FCH)
Evaporate:	to dryness and reconstitute to 1.0 mL with Methanol/ Water (96:4) (containing internal standards)

LC-MS/MS Parameters

Column:	Gemini [®] 3 μm C18	
Dimensions:	50 x 2.0 mm	
Part No.:	00B-4439-B0	
Mobile Phase:	A: 20 mM Ammonium acetate in Water B: Methanol	
Flow Rate:	0.6 mL/min	
Gradient:	Time (Min)	% B
	0	5
	0.1	55
	4.5	99
	8	99
	8.5	5
Delay column:	Luna [®] 5 μm C18(2) 30 x 3.0 mm (00A-4252-Y0) installed between the autosampler and mobile phase pump mixer	
Inj. Volume:	10 μL	

Figure 2.
Chromatography of HFPO-DA in a mixture of PFAS



Mass Spec Parameters

Mass Spec Detector: Sciex Triple Quad™ 4500

Ion Source Parameters: Samples were ionized using electrospray in negative ion-mode.

Parameter	Value
CAD	9
CUR	30
GS1	40
GS2	60
IS Voltage	-4500
TEM	450

MRM Transitions for HFPO-DA:

Compound	Q1	Q3	RT	DP	CE
HFPO-DA (Quant)	329	185	3.7	-30	-32
HFPO-DA (Qual)	329	169	3.7	-30	-18
¹³ C ₃ -HFPO-DA	332	185	3.7	-30	-32


Conclusion

GenX is an emerging contaminant, and analysis of GenX along with other PFASs is vital for proper risk assessment of contaminated water and soil and human exposure. These results show that GenX can be included in a method along with 24 other PFASs to be analyzed simultaneously in a single injection. The selectivity and sensitivity of the SCIEX 4500 Triple Quadrupole MS along with the chromatography provided by the Phenomenex Gemini C18 HPLC column allow this method to achieve sub-ppt reporting limits. Sample preparation on Strata XL-AW, a mixed-mode polymer reversed phase/weak anion exchange solid phase extraction sorbent with large pores and particles for quick flow through of large volumes, with conditions similar to ISO 25101:2009 effectively recovered the polar, short chained acids like PFBA, and further clean-up through Strata GCB, activated carbon, per DOD QSM 5.1 requirements enhances effectiveness for non-drinking water samples, like ground water and waste water.



Ordering Information

Sample Preparation

Strata® GCB Solid Phase Extraction (SPE)

Format	Sorbent Mass	Part Number	Unit
Tube			
	250 mg	8B-S528-FCH	6 mL (30/box)
	500 mg	8B-S528-HCH	6 mL (30/box)

Strata-XL-AW Solid Phase Extraction (SPE)

Format	Sorbent Mass	Part Number	Unit
Tube			
	30 mg	8B-S051-TAK	1 mL (100/box)
	60 mg	8B-S051-UBJ	3 mL (50/box)
	100 mg	8B-S051-EBJ	3 mL (50/box)
	100 mg	8B-S051-ECH	6 mL (30/box)
	200 mg	8B-S051-FBJ	3 mL (50/box)
	200 mg	8B-S051-FCH	6 mL (30/box)
	500 mg	8B-S051-HCH	6 mL (30/box)
Giga Tube			
	2 g	8B-S051-KEG	20 mL (20/box)

LC Columns

Gemini

3 µm Microbore, Minibore and MidBore™ Columns (mm)									SecurityGuard™ Cartridges (mm)	
Phases	50 x 1.0	20 x 2.0	30 x 2.0	50 x 2.0	100 x 2.0	150 x 2.0	50 x 3.0	100 x 3.0	150 x 3.0	4 x 2.0*
Gemini® C18	00B-4439-A0	00M-4439-B0	00A-4439-B0	00B-4439-B0	00D-4439-B0	00F-4439-B0	00B-4439-Y0	00D-4439-Y0	00F-4439-Y0	AJ0-7596
										for ID: 2.0-3.0 mm

Luna

5 µm Microbore and Minibore Columns (mm)								SecurityGuard™ Cartridges (mm)		
Phases	50 x 1.0	150 x 1.0	250 x 1.0	30 x 2.0	50 x 2.0	150 x 2.0	250 x 2.0	4 x 2.0*		
Luna® C18(2)	00B-4252-A0	00F-4252-A0	00G-4252-A0	00A-4252-B0	00B-4252-B0	00F-4252-B0	00G-4252-B0	AJ0-4286		
										for ID: 2.0-3.0 mm

5 µm MidBore and Analytical Columns (mm)								SecurityGuard™ Cartridges (mm)	
Phases	30 x 3.0	50 x 3.0	150 x 3.0	250 x 3.0	30 x 4.6	50 x 4.6	75 x 4.6	4 x 2.0*	4 x 3.0*
Luna C18(2)	00A-4252-Y0	00B-4252-Y0	00F-4252-Y0	00G-4252-Y0	00A-4252-E0	00B-4252-E0	00C-4252-E0	AJ0-4286	AJ0-4287
								for ID: 2.0-3.0 mm	3.2-8.0 mm

*SecurityGuard Analytical Cartridges require holder, Part No.: KJ0-4282

APPLICATIONS

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