

# FMS Agilent Presentation

Dioxin 2023

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# PFAS Methodology Overview

Current Standard & Consensus Methods Available:

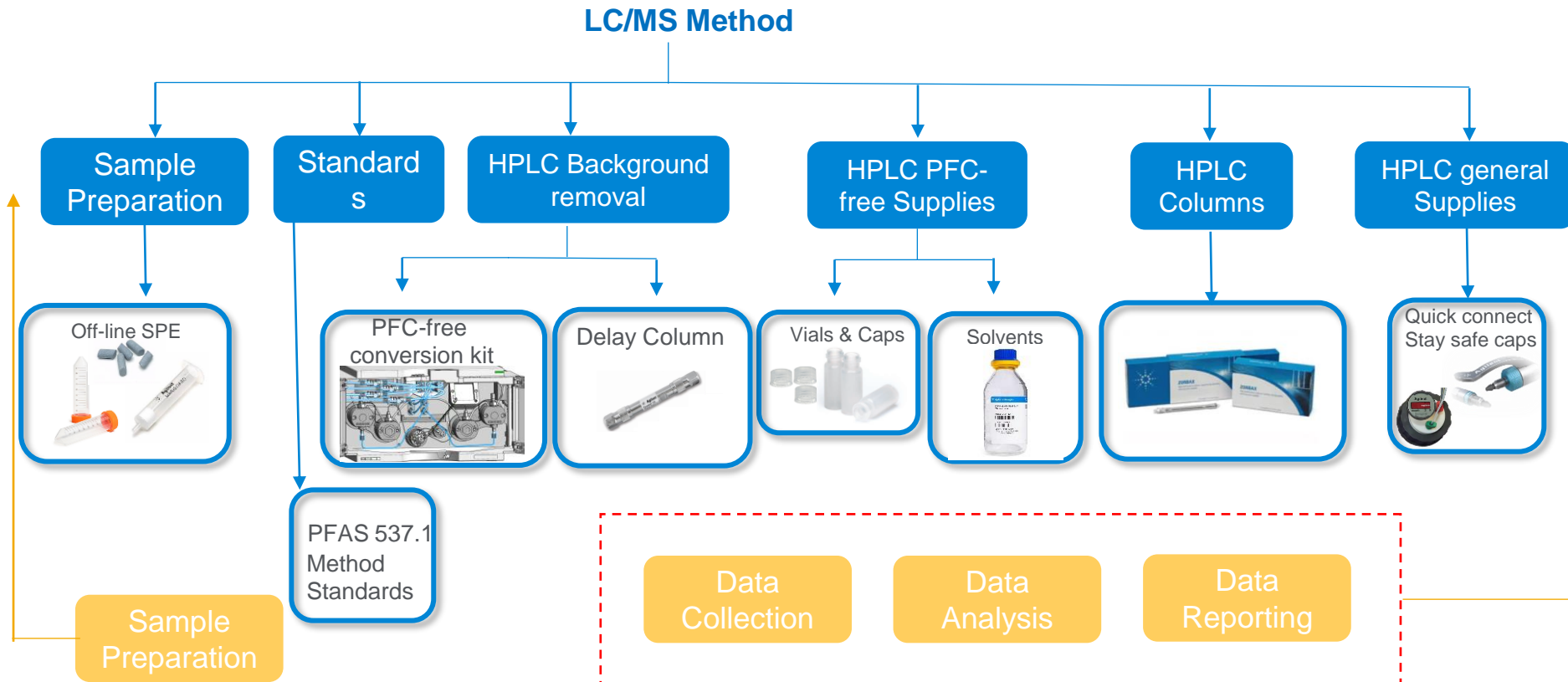
[PFAS Analytical Methods Development and Sampling Research | US EPA](#)

Method	Matrix Tested	# of Analytes	Sample prep procedure	Quantification Technique	Sample (mL)/ Injection (uL) volume	Year
USEPA 537	Drinking water	14	Solid phase extraction (SPE)	Internal Standard correction	250/10	2008
USEPA 537.1	Drinking water	18	SPE	Internal Standard	250/10	2018
USEPA 533	Drinking water	25	SPE	Isotope Dilution	250/10	2019
EPA 1633 (draft)	non-potable water, solids, tissues	40	SPE	Isotope Dilution		2022
ISO/DIS 21675:2019	Drinking water, Sea water, Fresh water, wastewater (<0.2% solids)	30	SPE	Internal standard correction	500/5	2019
USEPA 8327	Surface water, Ground water, Wastewater influent and effluent	24	Dilute & shoot	External calibration (isotope dilution also allowed)	5/30	2019
ASTM 7979	Surface water, Ground water, Wastewater influent and effluent	21	Filtration	External calibration (isotope dilution also allowed)	5/30	2017
ASTM 7968	Soil and solids	21	Filtration	External calibration	5g/30	2015

# PFAS Methodology Overview

Sample	Matrix Tested	Method	# Analytes	Sample prep procedure	Agilent Solution	Product image
Environmental	Drinking water	USEPA 537	14	Solid phase extraction (SPE)	Bond Elut PFAS WAX	
		USEPA 537.1	18			
		USEPA 533	25			
		DIN 38407-42				
		ISO/DIS21675:2019	30			
	Surface water / Ground water/ waste water	ISO/DIS21675:2019	30	Solid phase extraction (SPE)	Captiva Syringe Filter	
		USEPA 8327	24	Filtration		
		ASTM 7979	21			
		DIN 38407-42		Filtration or Solid phase extraction (SPE)	Bond Elut PFAS WAX	
	Soil/ Solids/sludge	EPA 1633 (Draft)	40	Solid phase extraction (SPE)		
		DIN 38414-14		Filtration or Solid phase extraction (SPE)		
		ASTM 7968	21	Filtration		
		CMA/3/D	30	Solid phase extraction (SPE) Matrix clean up with Caron S	Bond Elut Carbon S	
Food	Milk, Eggs, Meat, Seafood, Fruits, vegetables, baby foods	EU Directive	4	Filtration pass through	Captiva EMR-PFAS 1 Captiva EMR-PFAS 2	
				QuEChERS dSPE	Bond Elut dSPE	

# PFAS Sample Prep & Analysis: Basic Workflow Overview



# EPA 533 – a method to include “short-chain” PFAS

EPA 533	EPA 537.1
25 Analytes	18 Analytes
SPE with WAX	SPE with SDVB
28 days hold time	28 days hold time (14 days for Analysis)
Isotope Dilution	Internal Standard
Final extract in 80% MeOH	Final extract in 96% MeOH
2 ions required for reporting with LC/MS	Confirmation ion not necessary
Drinking water	Drinking water only
2019	2018/2020

Analyte	EPA 537.1	EPA 533	Analyte	EPA 537.1	EPA 533
PFBA		✓	PFBS	✓	✓
PFMPA		✓	PFPeS		✓
PFPeA		✓	PFHxS	✓	✓
PFMBA		✓	PFHpS		✓
PFEESA		✓	PFOS	✓	✓
NFDHA		✓	9Cl-PF3ONS	✓	✓
PFHxA	✓	✓	11Cl-PF3OUdS	✓	✓
HFPO-DA	✓	✓	NEtFOSAA	✓	
PFHpA	✓	✓	NMeFOSAA	✓	
ADONA	✓	✓	PFTeDA	✓	
PFOA	✓	✓	PFTrDA	✓	
PFNA	✓	✓	4:2 FTS		✓
PFDA	✓	✓	6:2 FTS		✓
PFUnA	✓	✓	8:2 FTS		✓
PFDaA	✓	✓			

# Product Recommendations

Sample Prep

Standards  
&  
Reagents

HPLC  
Background  
Removal

HPLC PFC-  
Free supplies  
& solvents

HPLC  
Columns

HPLC General  
supplies

## Solid Phase Extraction Methods:

### EPA 533 - Drinking water

- **Bond Elut PFAS WAX, 500 mg**, 6 mL, 30/pk
- Centrifuge tubes and caps, 15 mL, 50/pk
- Bond Elut Adapter cap for 1, 3, and 6 mL Bond Elut cartridges (15/pk)
- Bond Elut Empty SPE cartridge 60 mL (100/pk)
- Vac Elut 20 Manifold long valve stopcock, 20/pk
- Vac Elut SPS 24 manifold with collection rack for 10 x 75 mm test tubes
- Collection rack and funnel set for 12 or 15 mL conical tubes, for Vac Elut SPS 24 manifold
- 

[Application Note 5994-4960EN : Determination of Per and Polyfluoroalkyl Substances in Drinking Water Using Agilent Bond Elut PFAS WAX SPE and LC/MS/MS](#)

# Agilent Bond Elut PFAS WAX

Offers:

- ✓ Polymeric polystyrene divinylbenzene (PSDVB) sorbent
- ✓ WAX chemistry featuring a diamino functionality
- ✓  $pK_a > 8$
- ✓ Multiple bed mass options: 500 mg, 200 mg, 150 mg

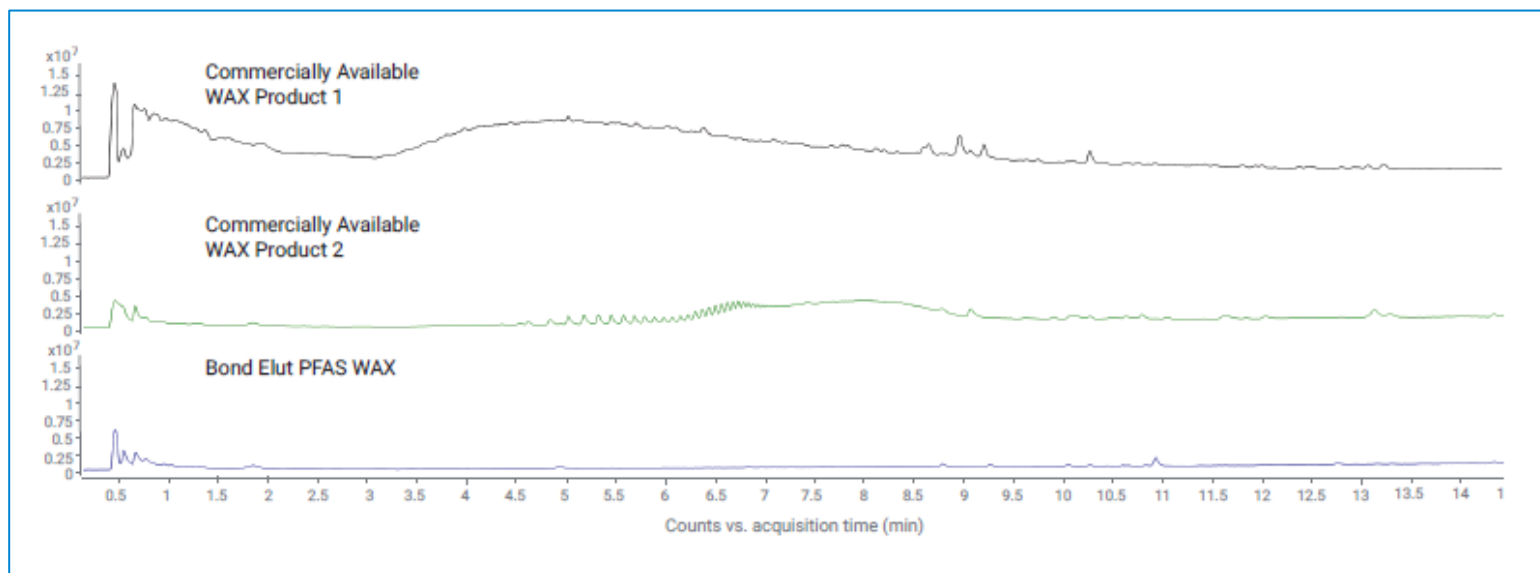
U.S. EPA 533 Method Requirements:

## 6.8.1 SPE Cartridges

SPE cartridges containing weak anion exchange, mixed-mode polymeric sorbent (polymeric backbone and a diamino ligand), particle size approximately 33  $\mu\text{m}$ . The SPE sorbent must have a  $pK_a$  above 8 so that it remains positively charged during extraction.



# Bond Elut PFAS WAX for your PFAS testing needs



Agilent Bond Elut SPE products are manufactured using state-of-the-art processes—and are subject to more than 25 tests to ensure consistent recovery, cleanliness, and flow.

*“Their judges evaluate products and programs based on environmental impact, contribution to sustainability goals, innovative application of technology, ease of use, efficiency, and reliability”*



Per- and polyfluoroalkyl substances (PFAS) have been used in industry and consumer products since the 1940s and have been widely detected in water, air, soil, fish, and other complex matrices. Greater scientific understanding of these chemicals has inspired a need for accurate PFAS monitoring and identification, which is one of many areas that Agilent is leading the way with sustainable technology and innovation.

Agilent Bond Elut PFAS WAX solid phase extraction (SPE) cartridges isolate PFAS from environmental matrices, such as drinking water, wastewater, soil and sludges while delivering the consistency, reliability and robustness customers need, whether analyzing a few samples per week or hundreds per day. The extended PFAS target list, performance and flexibility allows customers to address multiple needs with one solution instead of several, optimizing laboratory costs by reducing the cost per sample, complying with increasingly stringent regulatory requirements,


minimizing costly contamination and decreasing data processing times and required troubleshooting time. 🍌



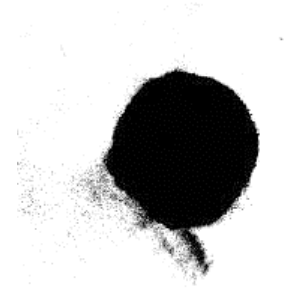
*“Illuminates the path towards environmental stewardship and energy efficiency”*

# Carbon S Sorbent – Matrix clean up

Agilent Carbon S sorbent is an *advanced hybrid carbon material* with **optimized carbon content and pore structure**



Significant improvement on sensitive pesticides recovery and reproducibility
Decent pigment removal, comparable to GCB sorbent and better than other sorbents for pigment removal
Improved sorbent selectivity and thus better balance



Carbon S  
sorbent

# Agilent Bond Elut Carbon S – Matrix clean up

## Offers:

- ✓ Carbon S is an advanced hybrid carbon material with optimized carbon content and pore structure
- ✓ Matrix clean up as opposed to enrichment which is offered by the Bond Elut WAX
- ✓ Compared to GCB, Carbon S provides equivalent or better pigment removal from sample matrices, while significantly improving recovery for some GCB-selective analytes (such as planar pesticides)
- ✓ Carbon S sorbent provides a better balance between analyte recovery and matrix removal efficiency than traditional GCB sorbent
- ✓ The Carbon S SPE cartridges can be used as a replacement for the GCB cartridges for applications where SPE methodology is used.

Methods: CMA/ 3/D and EPA 1633 (with Bond Elut PFAS WAX)

Application Note  
Environmental



Determination of Per and Polyfluoroalkyl Substances in Soils Using Carbon S SPE by LC/MS/MS

### Author

Matthew Giardina, Ph.D.  
Agilent Technologies, Inc.

### Abstract

This application note presents the development and evaluation of a multicomponent method for the analysis of per and polyfluoroalkyl substances (PFAS) in soil. The method incorporates a basic methanol extraction followed by a passthrough matrix removal step using an Agilent Bond Elut Carbon S solid phase extraction (SPE) cartridge and quantitative analysis by LC/MS/MS. For the 59 PFAS tested, the average recovery at the low spiking concentration (0.625 ng/g) was 99.9% with a relative standard deviation of 13.5%. Depending upon the soil matrix, the use of the Bond Elut Carbon S cartridges can improve chromatographic peak shape and retention for early eluting compounds such as PFBA.

# Formats

## Captiva EMR (Enhanced Matrix Removal) Passthrough Clean up

Captiva EMR-HCF1 with NH<sub>2</sub>

Captiva EMR-HCF2 with PSA

Captiva EMR-GPF

Captiva EMR-GPD

Captiva EMR-LPD

HC: High  
Chlorophyll  
GP: General  
Pigment  
LP: Low Pigment

F: Fresh  
(matrix)  
D: Dry (matrix)



## SPE

### Straight Syringe Barrels:

Bond Elut with Carbon S (variety of sizes)

Carbon S blended with PSA

Carbon S blended with NH<sub>2</sub>

### Bond Elut Jr.:

Carbon S

- 250mg
- 400mg



11 Different Sample Packs also (see P/N lists) of best sellers

# LC-TQ for PFAS



**Ultivo**  
*Core Routine*

 Ease of Use  Serviceability

 Throughput  Form-Factor



**6475**  
*Core Routine & Research*

 Rugged  Versatile

 Smart  Compliant

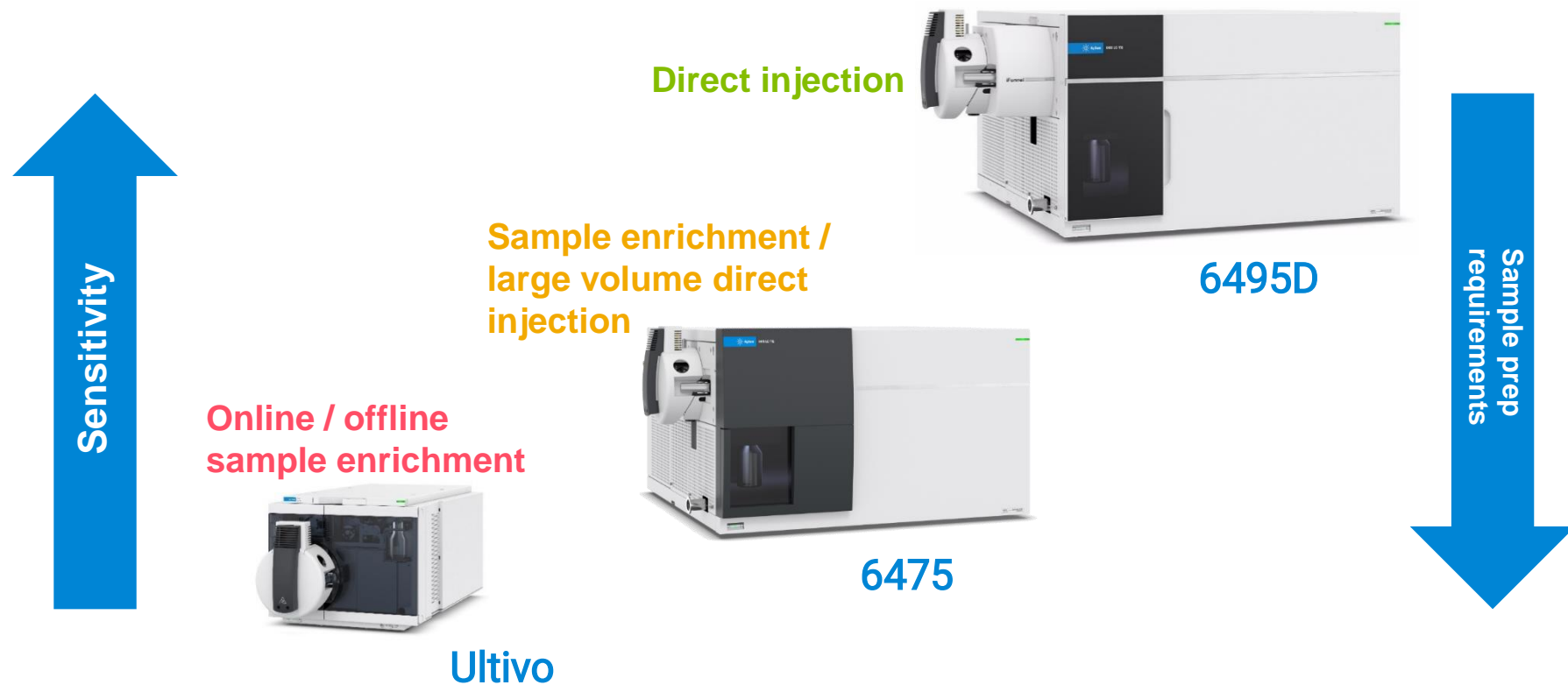


**6495D**  
*High Performance Routine & Research*

 High-end Performance  Easy to Maintain

 Made for Research  Production-Ready

# LC-TQ for PFAS



# Intelligent LC-TQs



## AI-based Tuning & Calibration

**SWARM Autotune** - find the best parameters, faster



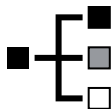
## Active System Monitoring

**Early Maintenance Feedback** – identify and anticipate maintenance events



## Maintenance and Method Automation

**Scheduled Tunes** – consistently keep the system in spec  
**Fully Automated MRM and Ion Source Optimizer** – streamline and speed up method development



## Reflexive Injection Logic

**Intelligent Reflex (iReflex)** - logic controls react to results to ensure samples are in spec or to accelerate throughput



# PFAS MRM Database

- Agilent offers a database of MRM transitions for > 100 PFAS compounds
- MRM transitions have been optimised for all three Agilent LC-TQs
- Transitions can be searched and then directly imported into a new or existing method, speeding up method set-up

The screenshot displays the PFAS MRM Database software interface. At the top, there are icons for Search/Filter, Import List, and a toolbar. Below this, the 'Filter Compounds' section includes checkboxes for 'Show All Records', 'Enable Filters', and 'Optimized Compounds'. It also features date and group name filters. The 'Search Compounds' section has a 'Search Text' field containing 'EPA 533', 'EPA 537.1', 'ASTM', and 'ISO', along with a 'Select Columns' dropdown menu. The 'Select Transitions' section allows users to choose between 'Select top 1 ranked transitions', 'Primary transitions', and 'Secondary transitions'. The 'Set primary and trigger flags' section includes a 'Set top 2 ranked transitions as primary' option. The 'Rank transitions by' section offers 'Abundance' and 'Response Factor' options. The main table lists MRM transitions with columns for Compound Name, IUPAC Name, CAS, Formula, ChemSpider, Polarity, Mass, Precursor, Product, and Frag. The table is currently displaying 20 rows of data. At the bottom, the 'Current Database' is identified as 'D:\MassHunter\Databases\PFAS MRM Database 6470', and there are buttons for 'Add to Import List', 'Import', and 'Close'.

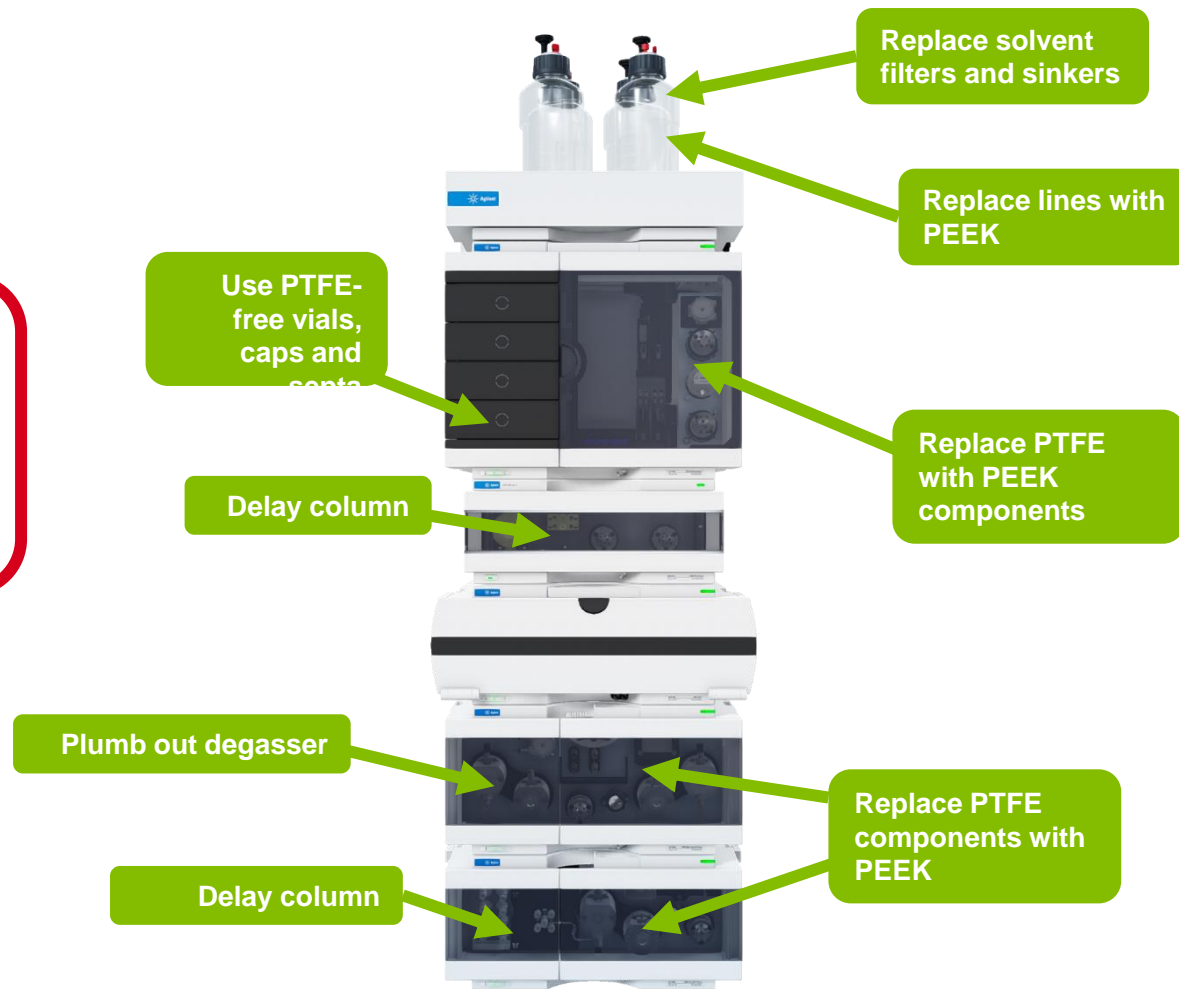
Compound Name	IUPAC Name	CAS	Formula	ChemSpider	Polarity	Mass	Precursor	Product	Frag
PFBA	Heptafluorobutanoic acid	375-22-4	C4HF7O2	9394	Negative	213.99	213	169	7
PFPeA	Nonafluoropentanoic acid	2706-90-3	C5HF9O2	68426	Negative	263.98	263	219	7
PFHxA	Undecafluorohexanoic acid	307-24-4	C6HF11O2	60864	Negative	313.98	313	269	7
PFHxA	Undecafluorohexanoic acid	307-24-4	C6HF11O2	60864	Negative	313.98	313	119	7
PFHpA	Tridecafluoroheptanoic acid	375-85-9	C7HF13O2	61135	Negative	363.98	363	319	7
PFHpA	Tridecafluoroheptanoic acid	375-85-9	C7HF13O2	61135	Negative	363.98	363	169	7
PFOA	Pentadecafluorooctanoic acid	335-67-1	C8HF15O2	9180	Negative	413.97	413	369	7
PFOA	Pentadecafluorooctanoic acid	335-67-1	C8HF15O2	9180	Negative	413.97	413	169	7
PFOA	Pentadecafluorooctanoic acid	335-67-1	C8HF15O2	9180	Negative	413.97	413	219	7
PFNA	Heptadecafluorononanoic acid	375-95-1	C9HF17O2	61138	Negative	463.97	463	419	7
PFNA	Heptadecafluorononanoic acid	375-95-1	C9HF17O2	61138	Negative	463.97	463	219	7
PFNA	Heptadecafluorononanoic acid	375-95-1	C9HF17O2	61138	Negative	463.97	463	169	7
PFUnDA	Henicosaflluoroundecanoic acid	2058-94-8	C11HF21O2	69649	Negative	563.96	563	519	10
PFUnDA	Henicosaflluoroundecanoic acid	2058-94-8	C11HF21O2	69649	Negative	563.96	563	269	10
PFUnDA	Henicosaflluoroundecanoic acid	2058-94-8	C11HF21O2	69649	Negative	563.96	563	319	10
PFDoDA	Tricosaflluorododecanoic acid	307-55-1	C12HF23O2	60867	Negative	613.96	613	569	10
PFDoDA	Tricosaflluorododecanoic acid	307-55-1	C12HF23O2	60867	Negative	613.96	613	319	10
PFDoDA	Tricosaflluorododecanoic acid	307-55-1	C12HF23O2	60867	Negative	613.96	613	269	10
PFDoDA	Tricosaflluorododecanoic acid	307-55-1	C12HF23O2	60867	Negative	613.96	613	169	10
PFTriDA	Pentacosaflluorotridecanoic acid	72629-94-8	C13HF25O2	2285907	Negative	663.96	663	619	10
PFTriDA	Pentacosaflluorotridecanoic acid	72629-94-8	C13HF25O2	2285907	Negative	663.96	663	319	10

# LC-MS for PFAS

## *Eliminating system contamination*

### Potential contamination sources

- Solvent filters
- PTFE-lined tubing
- PTFE-containing consumables
- Solvents
- PTFE materials in LC flow path



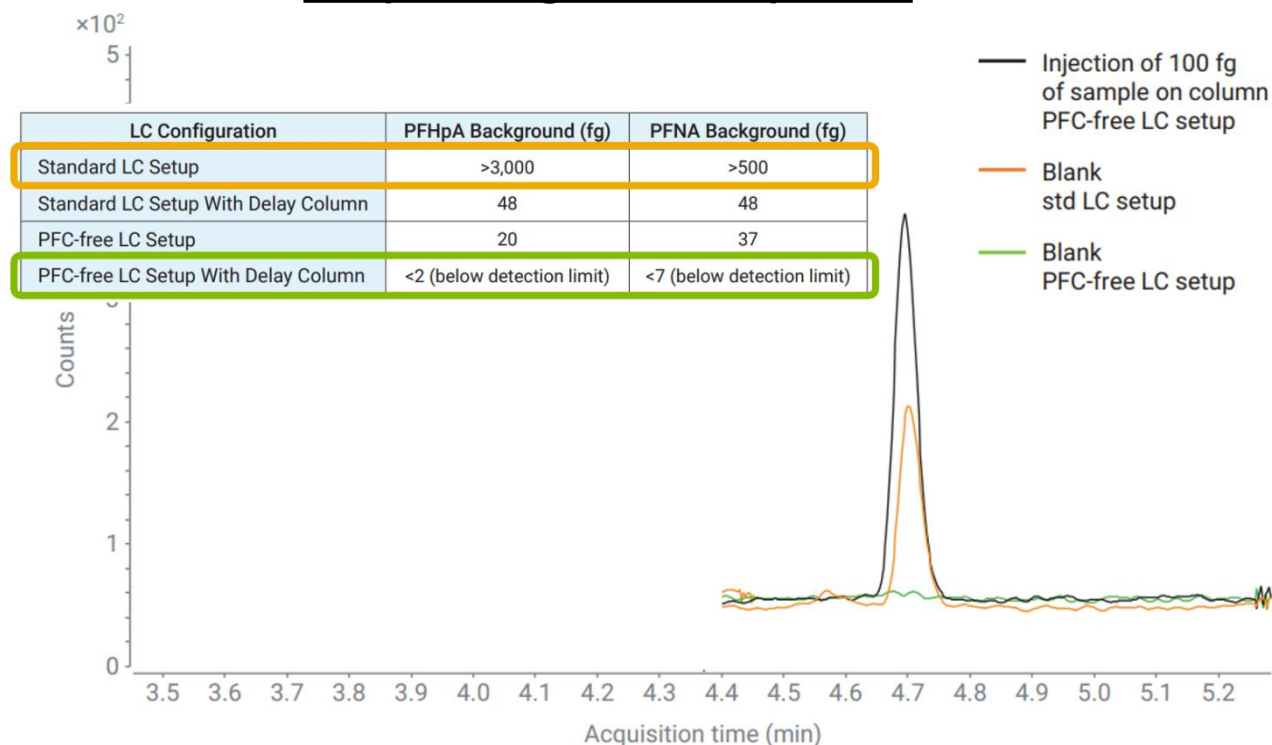
Agilent application note 5991-7863EN

# LC-MS for PFAS

## *Eliminating system contamination*

Agilent's **PFC-Free HPLC Conversion Kit** makes alterations to the HPLC to eliminate background PFAS signals

### PFHpA background comparison





# Agilent

Trusted Answers