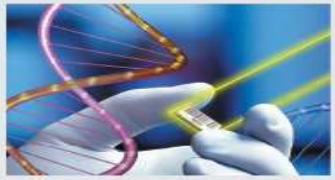
matriks

KJEMISK TEKNOLOGI


PFAS-Analyseløsninger som sikrer suksess

Ørjan Espeseth
Matriks AS

Today's Agenda

- PFAS Overview
- Regulation
- Targeted workflows
- Non-targeted workflows
- Automated Sample prep

Rainwear

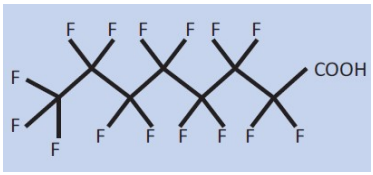
Non-stick coating

Cosmetics

Medical equipment

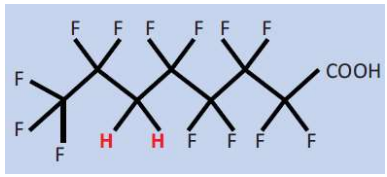
What are PFAS?

PFAS = Per and Polyfluoro Alkyl Substances



ALL H atoms linked to C in alkyl chain substituted with F

e.g. PFOA, PFOS, PFBA, PFHxS, PFDA, GenX



SOME *(but not all)* H atoms linked to C in alkyl chain are substituted with F

e.g. x:2 FTS, x:2 FTA, x:2 FTUCA, diPAP

- PFAS considered forever chemicals
- > 14,000 PFAS structures
- PFAS migration from food contact materials into food likely a main route of human exposure to PFAS
- PFAS substitutes degrade into PFAS
  - 8:2 FTOH & 10:2 FTOH converts to PFOA & PFDA
  - N-MeFOSE & N-EtFOSE converts to PFOS

ATSDR, NCEH Fact Sheet. (cdc.gov)
CompTox Chemicals Dashboard (epa.gov)
Li D et al., LCGC Europe-12-01-2020, Volume 33, Issue 12

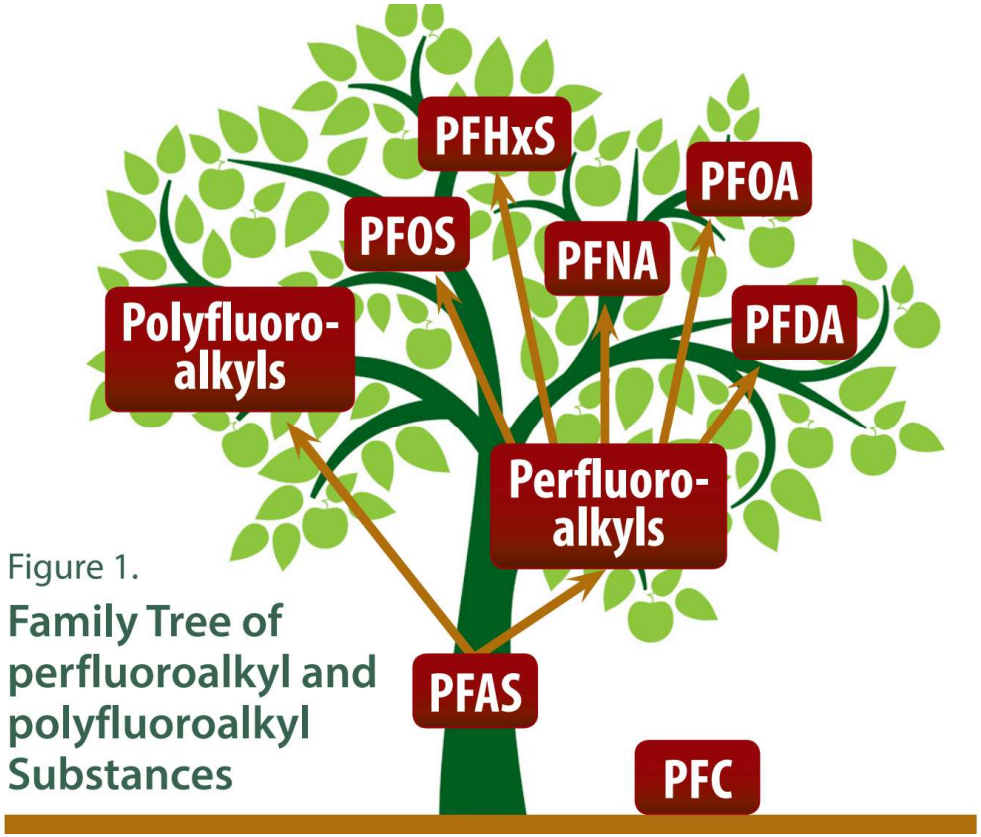


Figure 1.
Family Tree of perfluoroalkyl and polyfluoroalkyl Substances

Abbreviation	Chemical name
PFOS	Perfluorooctane sulfonic acid
PFOA (aka C8)	Perfluorooctanoic acid
PFNA	Perfluorononanoic acid
PFDA	Perfluorodecanoic acid
PFOSA (aka FOSA)	Perfluorooctane sulfonaminde
MeFOSAA (aka Me-PFOSA-AcOH)	2-(N-Methyl-perfluorooctane sulfonamido) acetic acid
Et-FOSAA (aka Et-PFOSA-AcOH)	2-(N-Ethyl-perfluorooctane sulfonamido acetic acid
PFHxS	Perfluorohexane sulfonic acid

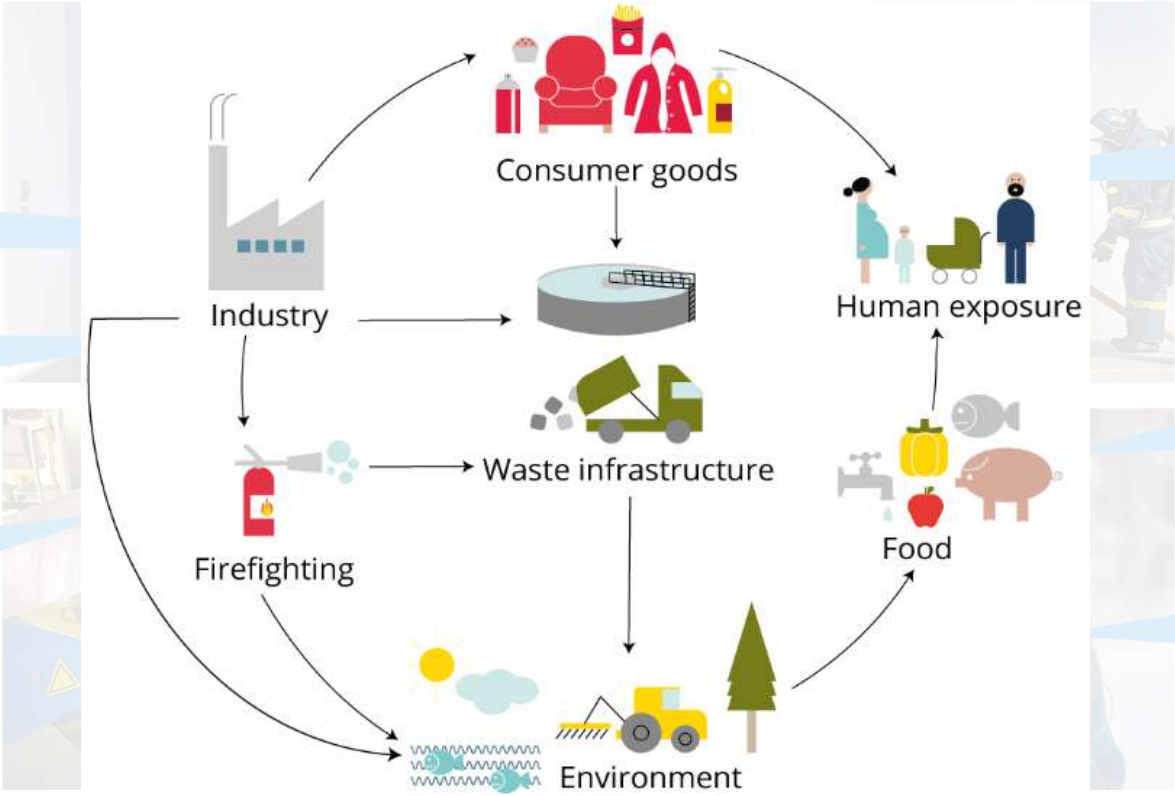
PFAS related products

Common household products and industrial uses

Non-stick surfaces

Grease-proof food packaging

Surfactants and lubricants



Fire-fighting foams

Stain guards

Water repellents

Diagram from www.eea.europa.eu

PFAS Analysis in Different Media

Ubiquitous presence requires testing in multiple matrices

Drinking Water

WasteWater

Soil

Food & Beverage

Ambient & Indoor Air

Materials & Textiles

Let’s talk regulations…

Proposed regulations

- EU have proposed via ECHA/REACH to ban all production, use and commercialization of 10000 PFAS products
  - 25 ppb for single PFAS, 250 ppb for total PFAS, 50 ppm F
- Norway have proposed to ban all PFAS products
  - Introduction over 18 months, not clear when it will be introduced
  - List of exceptions for specific use/applications with 5/10 years time

- EU Drinking Water directive lists 20 PFAS compounds to be monitored
  - Sum of 20 PFAS at 100 ng/L in drinking water, 500 ng/L total PFAS (no method yet)
- New proposal for 24 PFAS compounds
- Norway has currently no regulation for PFAS in drinking water, but will likely adopt the EU regulation (or similar)

- Norway – Classification of water and sediment
  - Only PFOS is listed

- More and more PFAS compounds are added so how to know the total level if only a few are monitored?

Drinking Water Inspectorate (United Kingdom) PFAS

No	Abbreviation	CAS Reg No	PFAS Category
1	PFBA	375-22-4	PFCA
2	PFPeA	2706-90-3	PFCA
3	PFHxA	307-24-4	PFCA
4	PFHpA	375-85-9	PFCA
5	PFOA	335-67-1	PFCA
6	PFNA	375-95-1	PFCA
7	PFDA	335-76-2	PFCA
8	PFUnA; PFUdA	2058-94-8	PFCA
9	PFDoA	307-55-1	PFCA
10	PFTrDA; PFTriA	72629-94-8	PFCA
11	PFTeA	376-06-7	PFCA
12	PFHxDA	67905-19-5	PFCA
13	PFODA	16517-11-6	PFCA
14	PFBS	375-73-5	PFSA
15	PFPeS	2706-91-4	PFSA
16	PFHxS	355-46-4	PFSA
17	PFHpS	375-92-8	PFSA
18	PFOS	1763-23-1	PFSA
19	PFNS	68259-12-1	PFSA
20	PFDS	335-77-3	PFSA
21	PFUnDS	749786-16-1	
22	PFDoS	79780-39-5	PFSA
23	HFPO-DA (Gen X)	13252-13-6	PFECA
24	HFPO-TA	13252-14-7	PFECA
25	DONA; ADONA	919005-14-4	PFECA
26	PFMOPrA	377-73-1	PFECA

No	Abbreviation	CAS Reg No	PFAS Category
27	NFDHA	151772-58-6	PFECA
28	PFMOBA	863090-89-5	PFECA
29	PFecHS *CAS No needs checking	133201-07-7	
30	3:3 FTCA	356-02-5	n:3 FTCA
31	5:3 FTCA	914637-49-3	n:3 FTCA
32	7:3 FTCA	812-70-4	n:3 FTCA
33	PFEESA	113507-82-7	PFESA
34	6:2 Cl-PFESA; 9Cl-PF3ONS	756426-58-1	Cl-PFESA
35	8:2 Cl-PFESA; 11Cl-PF3OUdS	763051-92-9	Cl-PFESA
36	4:2 FTSA; 4:2 FTS	757124-72-4	FTSA
37	6:2 FTSA; 6:2 FTS	27619-97-2	FTSA
38	8:2 FTSA; 8:2 FTS	39108-34-4	FTSA
39	FBSA	30334-69-1	FASA
40	FHxSA	41997-13-1	FASA
41	FOSA	754-91-6	FASA
42	MeFOSA; N-MeFOSA	31506-32-8	FASA
43	EtFOSA; N-EtFOSA	4151-50-2	FASA
44	MeFOSE	24448-09-7	FASE
45	EtFOSE	1691-99-2	FASE
46	NMeFOSAA; MeFOSAA	2355-31-9	FASAA
47	NEtFOSAA; EtFOSAA	2991-50-6	FASAA

	Proposed Detection Limit
PFAS (individual)	0.1ug/L
PFAS (total)	0.5ug/L

EU Lists of PFAS Compounds

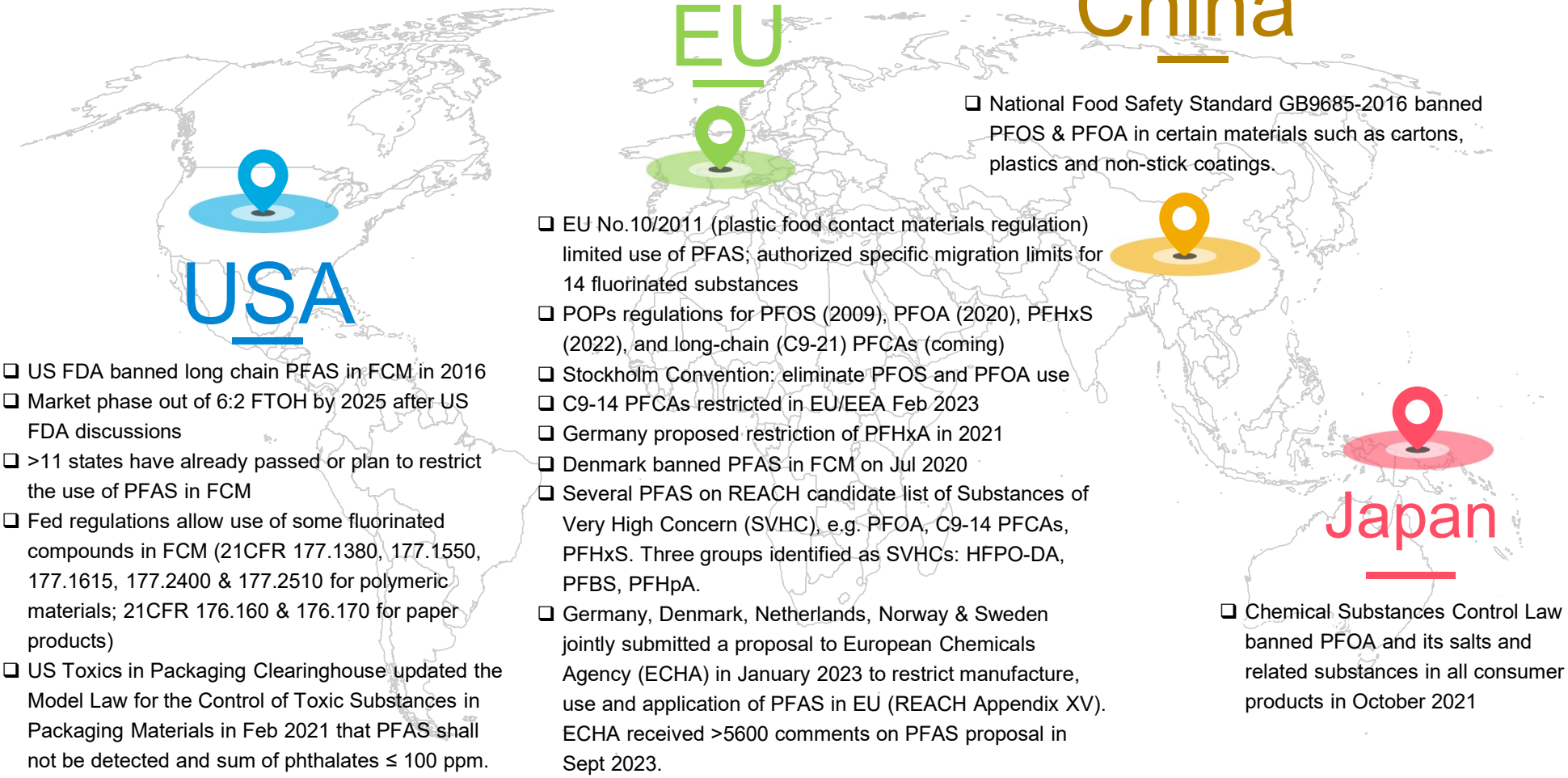
	Old EC Drinking Water Directive		
	Analyte	**Acronym**	**CAS No**
1	Perfluorobutanoic acid	PFBA	375-22-4
2	Perfluoropentanoic acid	PFPA	2706-90-3
3	Perfluorohexanoic acid	PFHxA	307-24-4
4	Perfluoroheptanoic acid	PFHpA	375-85-9
5	Perfluorooctanoic acid	PFOA	335-67-1
6	Perfluorononanoic acid	PFNA	375-95-1
7	Perfluorodecanoic acid	PFDA	335-76-2
8	Perfluoroundecanoic acid	PFUnDA	2058-94-8
9	Perfluorododecanoic acid	PFDoDA,	307-55-1
10	Perfluorotridecanoic acid	PFTrDA	72629-94-8
11	Perfluorobutane sulfonate	PFBS	375-73-5
12	Perfluoropentanesulfonic acid	PFPS	2706-91-4
13	Perfluorohexane sulfonate	PFHxS	355-46-4
14	Perfluoroheptane sulfonate	PFHpS	375-92-8
15	Perfluorooctane sulfonic acid	PFOS	2795-39-3
16	Perfluorononanesulfonic acid	PFNS	68259-12-1
17	Perfluorodecane sulfonate	PFDS	335-77-3
18	**Perfluoroundecane sulfonic acid**	**PFUnDS**	**749786-16-1**
19	**Perfluorododecane sulfonic acid**	**PFDoDS**	**79780-39-5**
20	**Perfluorotridecane sulfonic acid**	**PFTrDS**	**791563-89-8**

In the EU, a proposed revision to the EC Directives 2000/60/EC, 2006/118/EC, and 2008/105/EC attempts to include an environmental quality standard for the sum of 24 PFAS at 4.4 ng/L in surface and ground waters

	New Proposed EC Drinking Water Directive		
	Analyte	**Acronym**	**CAS No**
1	Perfluorobutanoic acid	PFBA	375- 22-4
2	Perfluoropentanoic acid	PFPeA	2706-90-3
3	Perfluorohexanoic acid	PFHxA	307-24-4
4	Perfluoroheptanoic acid	PFHpA	375-85-9
5	Perfluorooctanoic acid	PFOA	335-67-1
6	Perfluorononanoic acid	PFNA	375-95-1
7	Perfluorodecanoic acid	PFDA	335-76-2
8	Perfluoroundecanoic acid	PFUnDA	2058-94-8
9	Perfluorododecanoic acid	PFDoDA,	307-55-1
10	Perfluorotridecanoic acid	PFTrDA	72629-94-8
11	Perfluorobutane sulfonate	PFBS	375-73-5
12	Perfluoropentanesulfonic acid	PFPeS	2706-91-4
13	Perfluorohexane sulfonate	PFHxS	355-46-4
14	Perfluoroheptane sulfonate	PFHpS	375-92-8
15	Perfluorooctane sulfonic acid	PFOS	1763-23-1
16	Perfluorodecane sulfonate	PFDS	335-77-3
17	Perfluorotetradecanoic acid	PFTeDA	376-06-7
18	Perfluorohexadecanoic acid	PFHxDA	67905-19-5
19	Perfluorooctadecanoic acid	PFODA	16517-11-6
20	Ammonium perfluoro (2-methyl-3-oxahexanoate)	HFPO-DA (Gen X)	62037-80-3
21	Ammonium 2,2,3-trifluoro-3-(1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propoxy)propanoate	ADONA	958445-44-8
22	2- (Perfluorohexyl)ethyl alcohol	6:2 FTOH	647-42-7
23	2-(Perfluorooctyl)ethanol	8:2 FTOH	678-39-7
24	**Acetic acid / 2,2-difluoro-2-((2,2,4,5-tetrafluoro-5-(trifluoromethoxy)-1,3-dioxolan-4- yl)oxy)-**	**C6O4**	**1190931-41-9**

Link: [Proposed revisions to EC directive 2000/60/EC]

Global Regulatory Overview of PFAS in Food Contact Materials (FCM)



USA

- US FDA banned long chain PFAS in FCM in 2016
- Market phase out of 6:2 FTOH by 2025 after US FDA discussions
- >11 states have already passed or plan to restrict the use of PFAS in FCM
- Fed regulations allow use of some fluorinated compounds in FCM (21CFR 177.1380, 177.1550, 177.1615, 177.2400 & 177.2510 for polymeric materials; 21CFR 176.160 & 176.170 for paper products)
- US Toxics in Packaging Clearinghouse updated the Model Law for the Control of Toxic Substances in Packaging Materials in Feb 2021 that PFAS shall not be detected and sum of phthalates ≤ 100 ppm.

EU

- EU No.10/2011 (plastic food contact materials regulation) limited use of PFAS; authorized specific migration limits for 14 fluorinated substances
- POPs regulations for PFOS (2009), PFOA (2020), PFHxS (2022), and long-chain (C9-21) PFCAs (coming)
- Stockholm Convention: eliminate PFOS and PFOA use
- C9-14 PFCAs restricted in EU/EEA Feb 2023
- Germany proposed restriction of PFHxA in 2021
- Denmark banned PFAS in FCM on Jul 2020
- Several PFAS on REACH candidate list of Substances of Very High Concern (SVHC), e.g. PFOA, C9-14 PFCAs, PFHxS. Three groups identified as SVHCs: HFPO-DA, PFBS, PFHpA.
- Germany, Denmark, Netherlands, Norway & Sweden jointly submitted a proposal to European Chemicals Agency (ECHA) in January 2023 to restrict manufacture, use and application of PFAS in EU (REACH Appendix XV). ECHA received >5600 comments on PFAS proposal in Sept 2023.

China

- National Food Safety Standard GB9685-2016 banned PFOS & PFOA in certain materials such as cartons, plastics and non-stick coatings.

Japan

- Chemical Substances Control Law banned PFOA and its salts and related substances in all consumer products in October 2021

PFAS Analysis: Agilent Workflow Overview

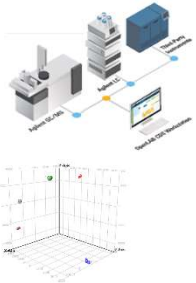
Adding value through integrating technologies and services together

Development | Monitoring | Data Analysis and Integrity

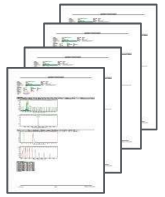
Sample Preparation

Separation

Detection



Data Analysis



Reporting

Services and Consulting with expertise

Consumables and Reagents

Agilent InfinityLab LC Series UHPLC for Every Lab

1220 Infinity II LC

Affordable Efficiency

The Agilent 1220 Infinity II LC is an affordable, high-quality integrated LC system, putting you on the fast track to efficiency.

1260 Infinity II LC

Everyday Efficiency in Every Way

The Agilent 1260 Infinity II LC is the trusted platform with the broadest instrument choice, taking you to the next level of efficiency.

1290 Infinity II LC

The Benchmark in Efficiency

The Agilent 1290 Infinity II LC embodies the next generation of LC, giving you the ultra-high performance to achieve maximum efficiency.

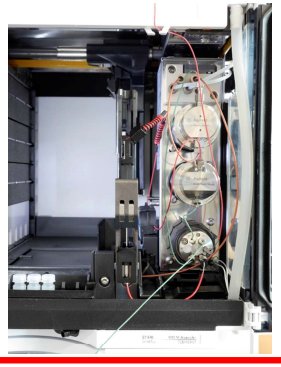
InfinityLab Accessories

Eliminate Background Contamination with PFC-Free Conversion Kit (PN 5004-0006)

Potential Contamination Sources

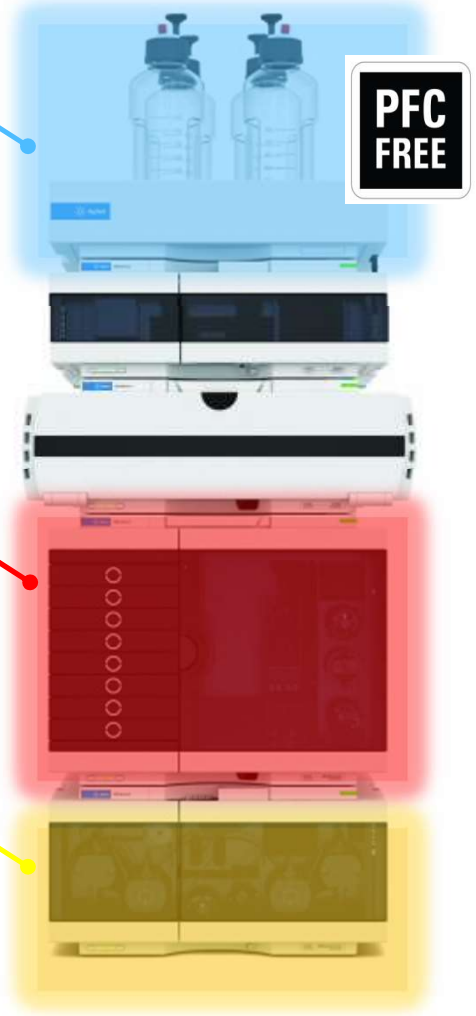
- Solvents
- Filtration apparatus
- Teflon lined tubing

Stay Safe PP bottlehead assemblies with PP tubing and stainless-steel solvent filters PN 5004-0004



- Stay PFC free Multiwash tubing kit PN 5004-0003
- 2 mL screw style clear polypropylene vial (100pk) PN 5191-8150
- 9 mm screw style clear polypropylene cap with thin membrane polypropylene / silicone bilayer septum (100pk) PN 5191-8151

- Bypass degasser & solvent selection valve
- Remove PTFE filter
- Add Inline Filter
- Add InfinityLab PFC Delay Column PN 8062-5100
- Connect Bottlehead assemblies PN 5004-0004



Application Note: Reduce PFAS Background with the Agilent PFC-free HPLC Conversion Kit (5994-2291EN)

Agilent's Triple Quadrupole LC/MS Portfolio

Focused on reliability, robustness, and routine analysis applications

Ultivo LC/TQ

Core Routine

- Ease of Use
- Serviceability
- Throughput
- Form-Factor

6475 LC/TQ

Core Routine & Research

- Trusted
- Rugged
- Versatile
- Numerous Apps.

NEW 6495 LC/TQ

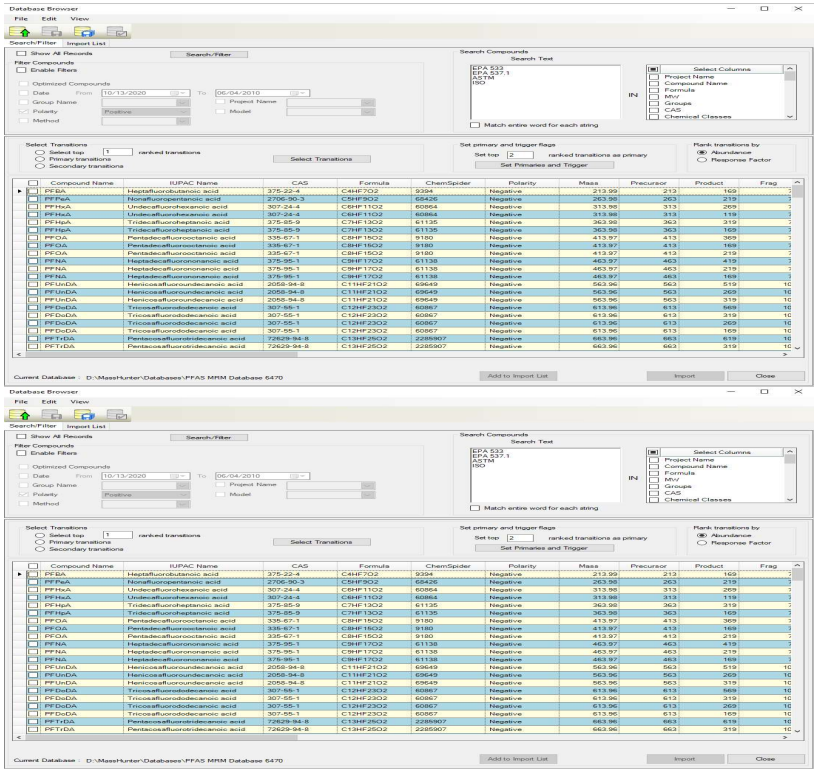
High Perf. Routine & Research

- High-end performance
- Easy to maintain
- Research Suited
- Production-Ready Robustness

Performance

PFAS MRM Database for Faster Method Development

Quickly build acquisition methods for targeted screening and quantitation using the Agilent PFAS MRM database



Key Features

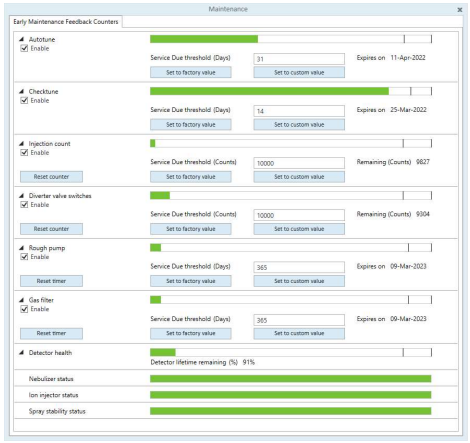
- Curated database of ~100 native and isotopically labelled PFAS compounds including MS acquisition parameters plus meta data
- Encompasses EPA , ASTM, ISO methods and EU DWD plus emerging PFAS classes
- Suitable for drinking water, non-potable water, and soil applications
- Seamlessly integrates into MassHunter Data Acquisition software
- Compatible with all current models of Agilent 6400 Series Triple Quadrupole LC/MS

Let the System Work for You with Guided Maintenance and Automated System Monitoring

Intelligence that Inspires

Start building your lab of the future today

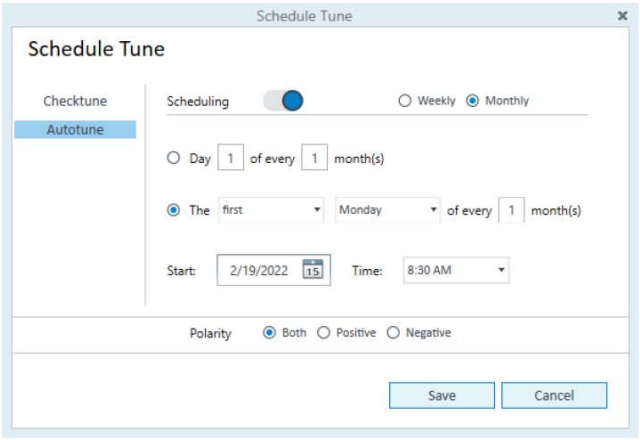
Guided Maintenance



Event timers read like a battery bar to help anticipate maintenance events

Event flags detects and pinpoints when an adverse event has occurred

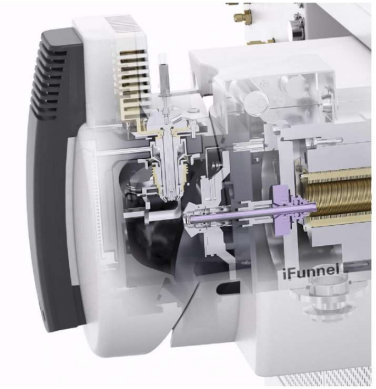
Scheduled Tuning



5AM → Instrument tunes before anyone arrives

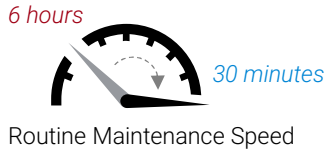
Scientist arrives → Instrument is ready to go

Easy access cleaning



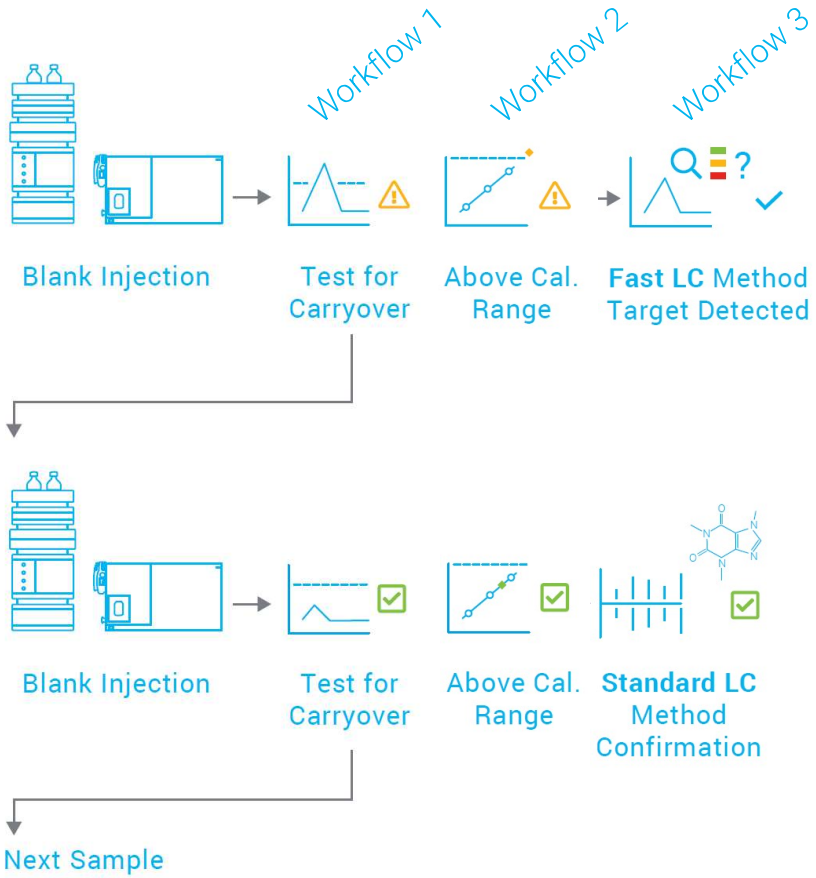
Reduce maintenance time by 92% with Agilent VacShield

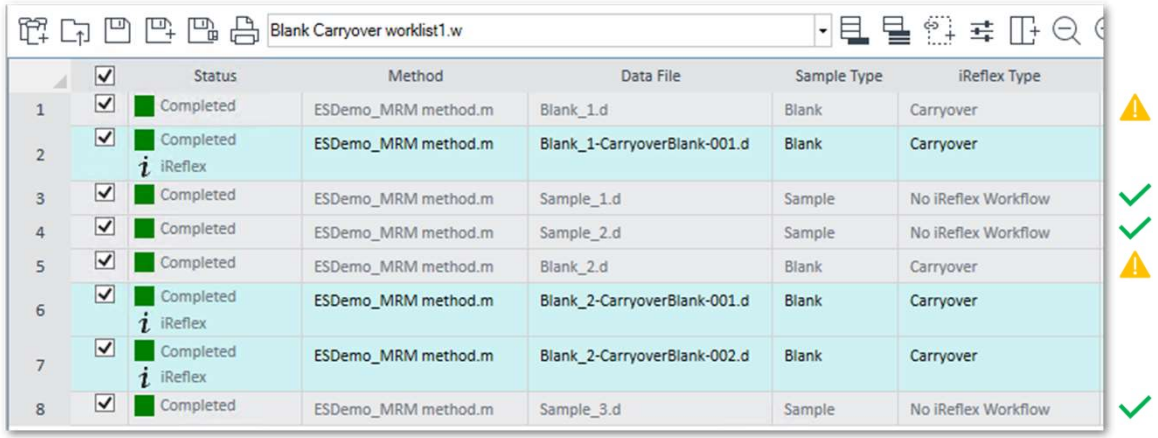
No venting required: perform maintenance in 30 minutes



Reflexive Injection Logic

Intelligent Reflex helps assure samples are in spec or to increase throughput





	Status	Method	Data File	Sample Type	iReflex Type
1	Completed	ESDemo_MRM method.m	Blank_1.d	Blank	Carryover
2	Completed iReflex	ESDemo_MRM method.m	Blank_1-CarryoverBlank-001.d	Blank	Carryover
3	Completed	ESDemo_MRM method.m	Sample_1.d	Sample	No iReflex Workflow
4	Completed	ESDemo_MRM method.m	Sample_2.d	Sample	No iReflex Workflow
5	Completed	ESDemo_MRM method.m	Blank_2.d	Blank	Carryover
6	Completed iReflex	ESDemo_MRM method.m	Blank_2-CarryoverBlank-001.d	Blank	Carryover
7	Completed iReflex	ESDemo_MRM method.m	Blank_2-CarryoverBlank-002.d	Blank	Carryover
8	Completed	ESDemo_MRM method.m	Sample_3.d	Sample	No iReflex Workflow

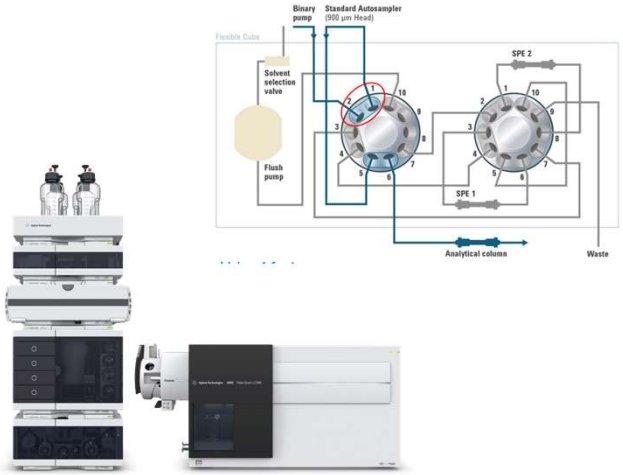
Additional blank injections inserted if carryover was detected

Target Strategies to achieve low level detection limits

LC/MS after
sample enrichment

Ultivo
6475
6495

Online-SPE LC/MS



Ultivo
6475
6495

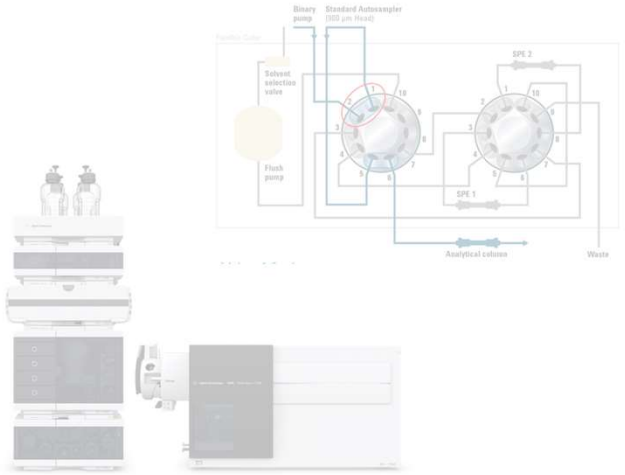
LC/MS with
direct injection

6475
6495

Strategies to achieve low level detection limits

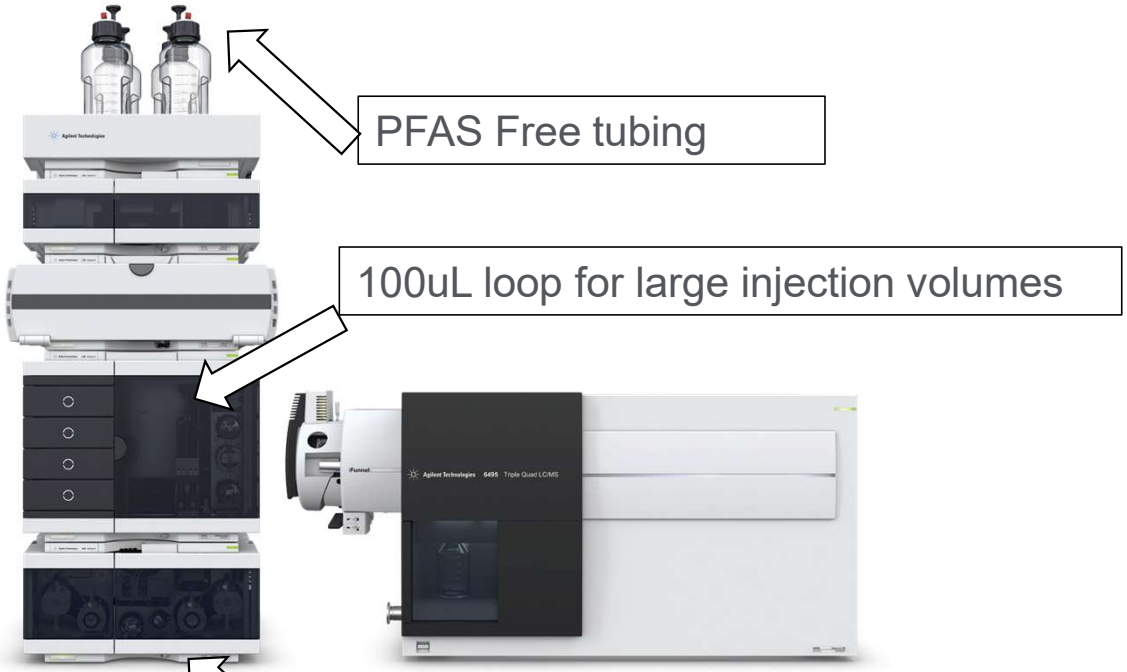
LC/MS after
sample enrichment

Online-SPE LC/MS



LC/MS with
direct injection

Hardware Setup



Configuration:

1290 Infinity II Multisampler (G7167B), 100 µL Head installed

1290 Infinity II Multi Column Compartment (G7116B)

100uL Injection Loop

PFAS Free kit installation (5004-0006)

6475 or 6495 Triple Quadrupole LC/MS

PFAS Free consumables

LC Conditions

LC System: Agilent 1290 Infinity II

LC Conditions		
Delay Column	Infinity Lab PFC Free Delay Column 4.6 x 30 mm	
Analytical column	Zorbax Eclipse Plus C18 2.1 x 100 mm, 1.8 um	
Column Temperature	40 ºC	
Injection Volume	100uL	
Standard Wash	IPA:MeOH, 10 sec flush, multi-wash optional	
Mobile Phase	A: 5 mM Ammonium Acetate in Water	B: Methanol
Flow Rate	0.4 mL/min	
Gradient program	Time (min)	B (%)
	0	10
	1.0	10
	1.5	55
	5.5	70
	7.0	80
	10.2	93
	10.21	100
	12.7	100
	12.71	10
Stop Time	14.5 min	

Agilent 1290 Infinity II LC

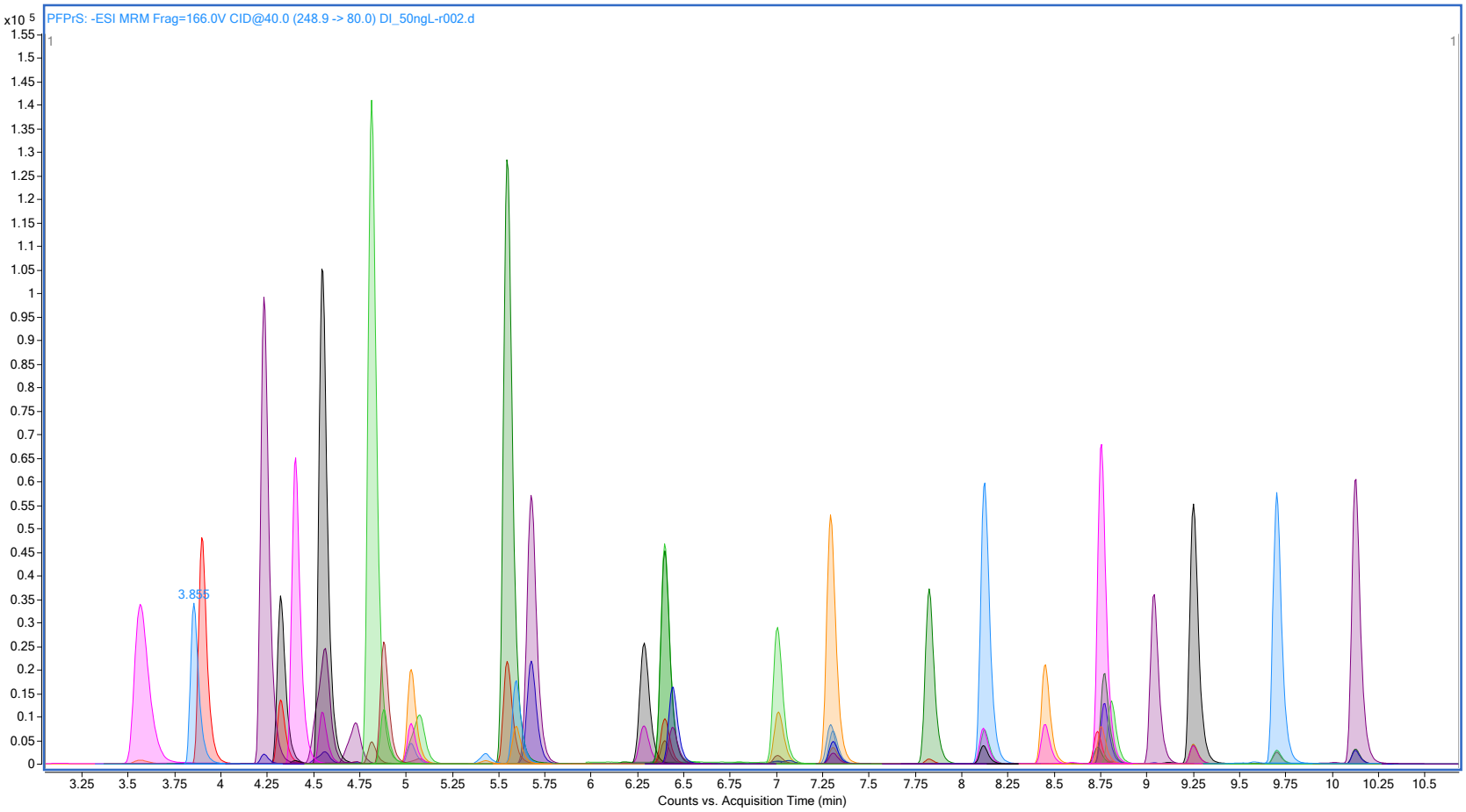
MS Parameters

MS Parameters	
Mass Spectrometer	Agilent 6495 with JetStream ESI using MRM mode
Ionization Mode	Negative
Gas Temperature	250 ºC
Gas Flow	11 L/min
Nebulizer Pressure	25 psi
Sheath Gas Temperature	375 ºC
Sheath Gas Flow	11 L/min
Capillary Voltage	2500 V
High Pressure iFunnel RF	90 V
Low Pressure iFunnel RF	60 V

Agilent 6495 C with JetStream ESI

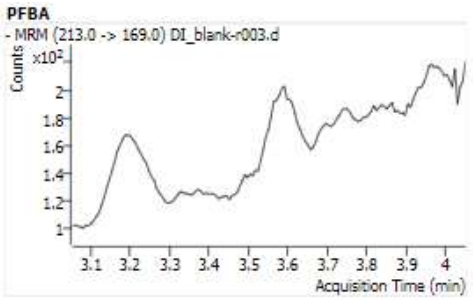
Chromatogram

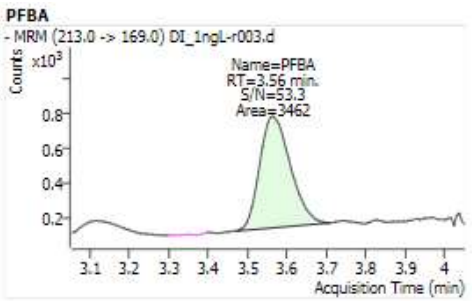
- Data from Direct injections 50ng/L

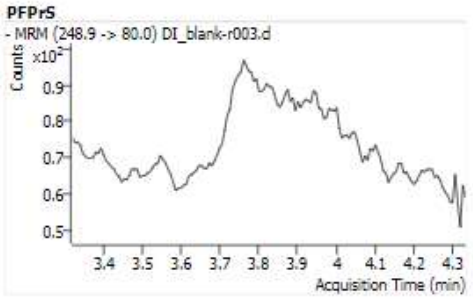


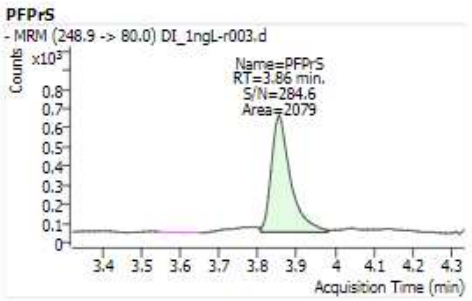
Advantages of Direct Injection

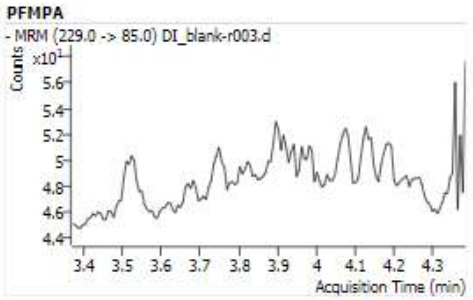
Blank Values vs 1ng/L – 100uL 100% Aqueous Injection

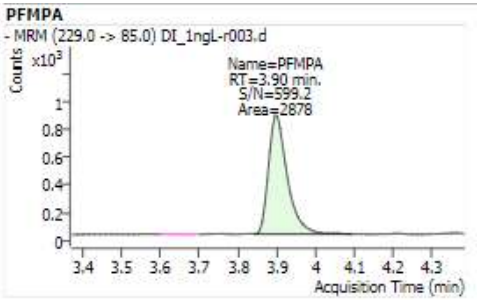


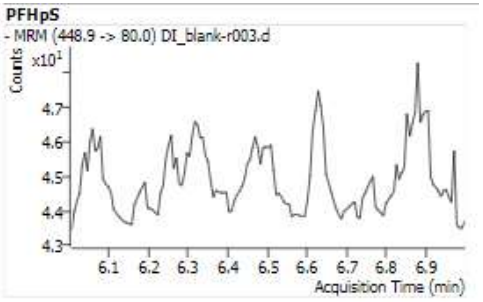


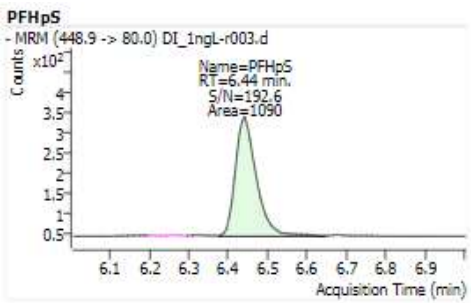


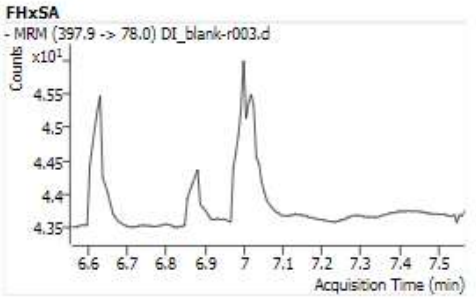


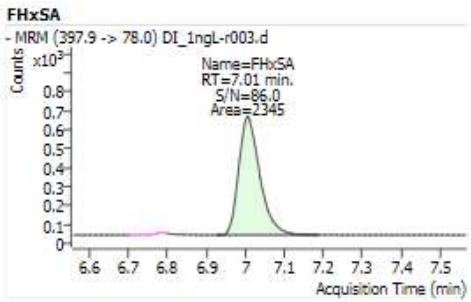


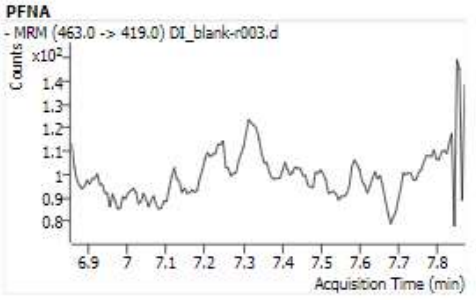


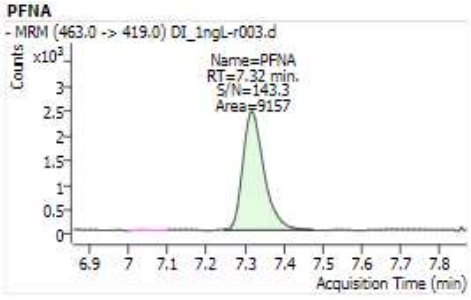






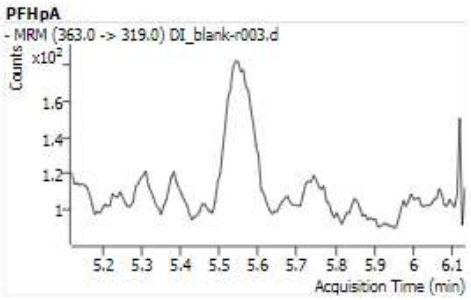


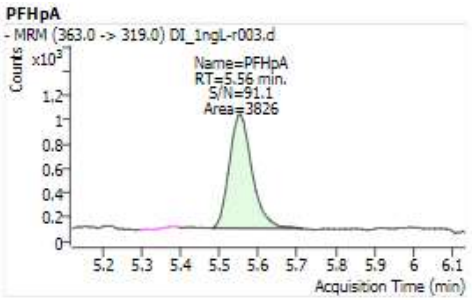


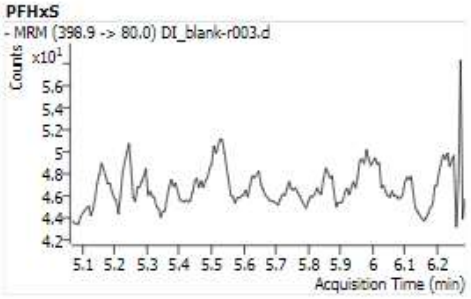


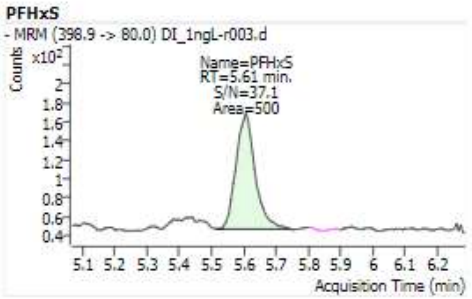
Advantages of Direct Injection

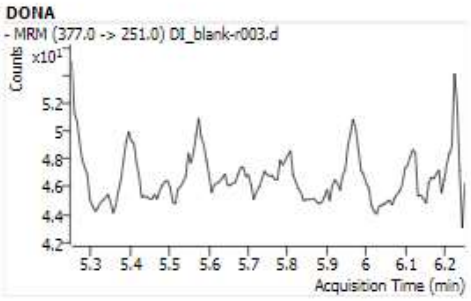
Blank Values vs 1ng/L – 100uL 50:50 Water:MeOH

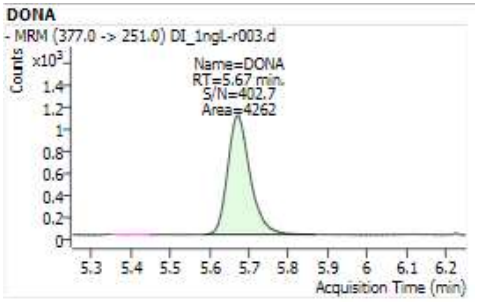


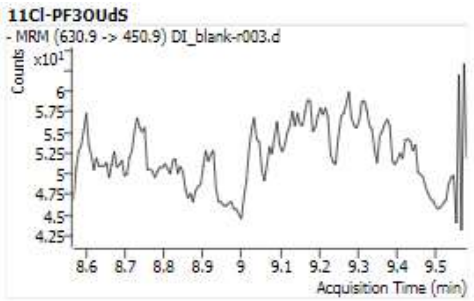


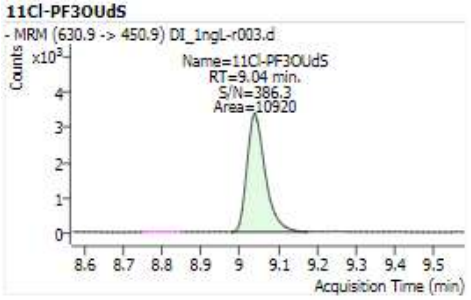


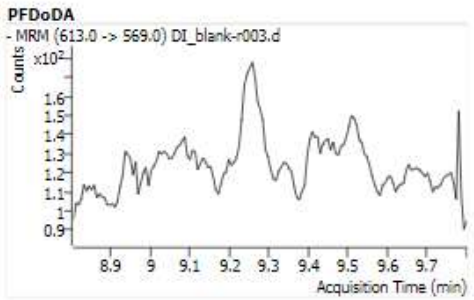


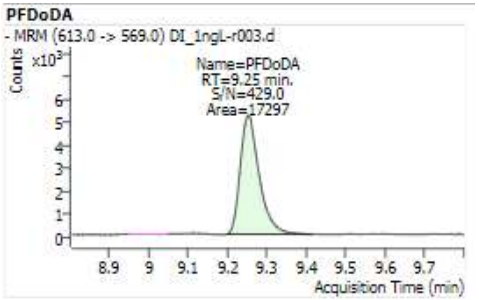


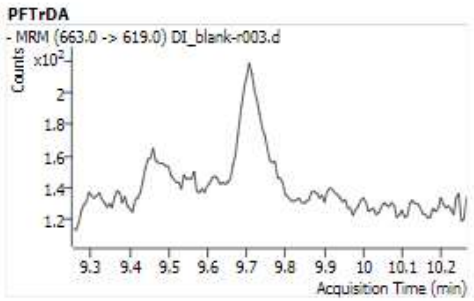


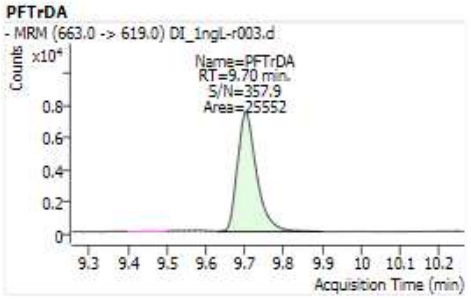












Direct Injection Applicatio

Home › Solutions › Environmental Testing › Water Testing › PFAS in Water › Direct Injection Methods for PFAS in Waters

Fast Analysis of PFAS in Water by Direct Injection

Certain regulatory methods, including US EPA Method 8327 and ASTM D7979, allow for rapid analysis of PFAS by direct injection. Quantitation of PFAS by direct injection eliminates the need for costly and time-consuming sample preparation techniques. In addition to rapid quantification of PFAS, direct injection methods can be more reproducible because there are no extraction variables that can cause contamination. Another advantage is a reduction in PFAS background contamination introduced through extraction consumables. The sensitivity of Agilent triple quadrupole LC/MS systems achieves the low detection levels needed for direct injection methods.

Syringes are a critical element in the direct injection workflow to remove particulates. Use of disposable syringes provides significant savings in time, solvent cost, and solvent waste disposal. Choice of the appropriate syringe material is critical to accurate and precise quantitation of PFAS. Agilent Captiva disposable syringes are ideally suited for this analysis due to their low sorption of PFAS compounds and low background.

Contact us

Application Note

Environmental

Agilent
Trusted Answers

A Method for PFAS Analysis in Drinking and Nonpotable Waters Using the Agilent 6495 LC/MS System

Direct aqueous injection method for the determination of 47 PFAS in water

Authors

David Powell and
Marcus Chadha
Agilent Technologies, Inc.

Laura Pinkney,
Beverley Kerrigan, and
Jenny Grimshaw
United Utilities PLC,
Warrington, UK

Abstract

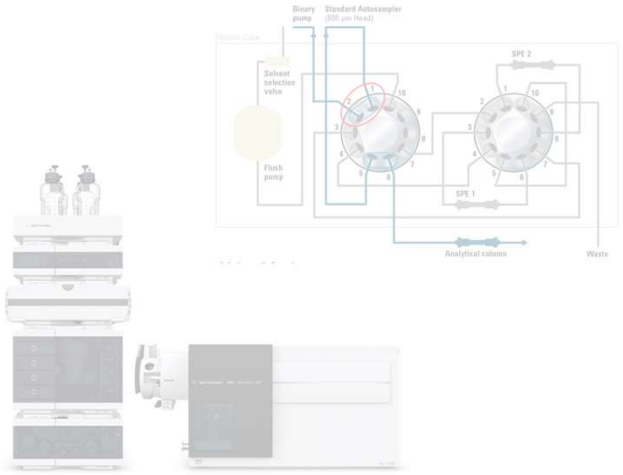
A comprehensive liquid chromatography/triple quadrupole mass spectrometry (LC/MS/MS) method was developed and validated for the quantitation of 47 per- and polyfluoroalkyl substances (PFAS) with the intention to accelerate and simplify routine laboratory water testing. Compound transitions and optimized parameters were applied to the analytical method. The method suitability was demonstrated using an Agilent 1290 Infinity II LC system coupled to an Agilent 6495 triple quadrupole LC/MS on water, surface water, and ground water, using direct injection of samples.

Agilent

Strategies to achieve low level detection limits

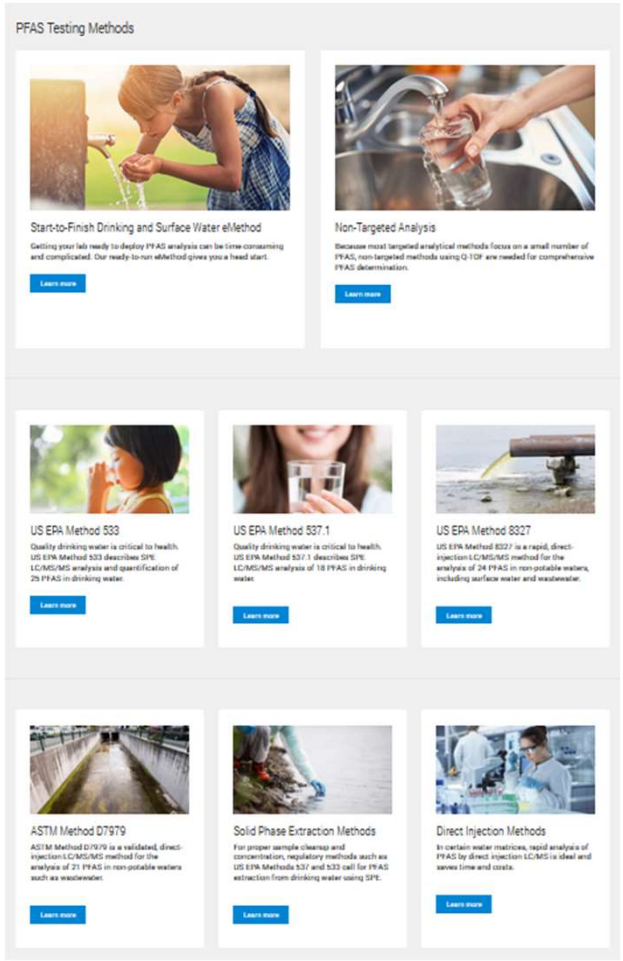
LC/MS after
sample enrichment

Online-SPE LC/MS



LC/MS with
direct injection

LC/MS after enrichment. US EPA methods on Agilent.com



Application Note

Environmental

Agilent

Trusted Answers

Analysis of Per/Polyfluoroalkyl Substances (PFAS) in Drinking Water by EPA 537.1 and EPA 533 Using the Agilent Ultivo Triple Quadrupole LC/MS

Authors

Agustin Pierri
Weck Laboratories Inc.,
Industry, CA, USA

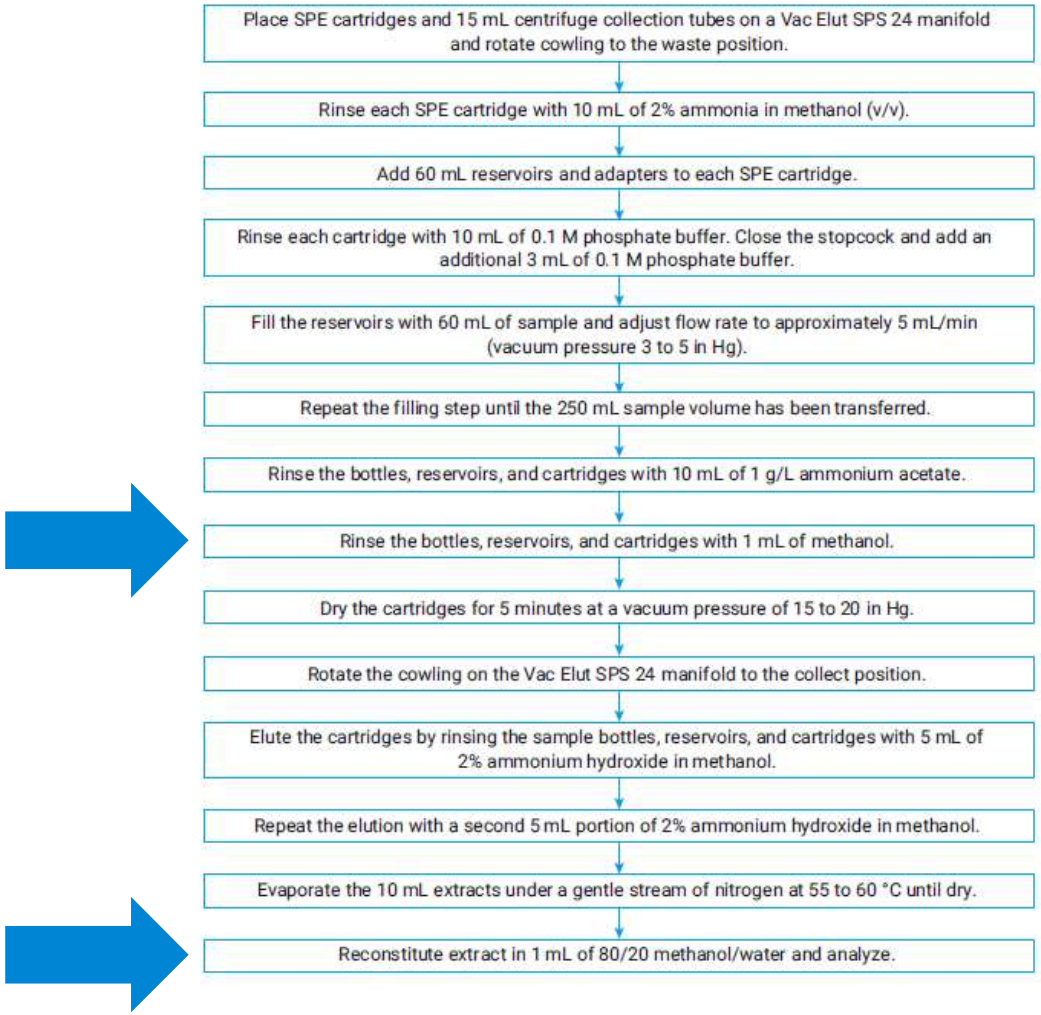
Tarun Anumol and Emily Parry
Agilent Technologies, Inc.
Wilmington, DE, USA

Abstract

This application note highlights the validation of EPA methods 537.1 and 533 for per/polyfluoroalkyl substances (PFAS) in drinking water in a commercial lab setting. The analytes were all separated using Agilent Poroshell EC-120 columns in both methods. Peak asymmetry factors were within EPA guidelines while achieving a run time of less than 10 minutes, allowing significant throughput gains over the EPA method run times. Recovery of all analytes following the solid-phase extraction

Compound	EPA 533 MDL (ng/L)	EPA 537.1 MDL (ng/L)
PFBA	0.44	–
PFMPA	0.28	–
PFPeA	0.24	–
PFBS	0.26	0.67
PFMBA	0.33	–
PFEESA	0.13	–
NFDHA	0.42	–
4:2FTS	0.36	–
PFHxA	0.39	0.67
PFPeS	0.41	–
HFPO-DA	0.44	0.81
PFHpA	0.27	
PFHxS	0.4	0.79
ADONA	0.27	–
6:2FTS	0.67	–
PFOA	0.29	0.39
PFHpS	0.44	
PFNA	0.41	0.62
PFOS	0.44	0.55
9Cl-PF3ONS	0.48	0.82
8:2FTS	0.49	–
PFDA	0.27	0.68
PFUnA	0.41	–
11Cl-PF3OUdS	0.16	0.63
PFDoA	0.59	0.45
Me-FOSAA	–	0.82
Et-FOSAA	–	0.91
PFHpA	–	0.54
PFTeDA	–	0.62
PFTrDA	–	0.41
PFUnA	–	0.56
ADONA	–	0.46

LC/MS after enrichment



Application Note
Environmental

Agilent
Trusted Answers

Analysis of Per- and Polyfluoroalkyl Substances in Drinking Water using SampliQ Weak Anion Exchange Solid Phase Extraction 150 mg Cartridge

Authors

Matthew Giardina, PhD and
New Lee Sun
Agilent Technologies, Inc.

Abstract

Optimizing the sorbent mass-to-sample volume ratio is an important consideration when developing a solid phase extraction method. For strongly retained analytes, the sorbent mass-to-sample volume ratio may be relatively small compared to weakly retained analytes, which require a larger bed mass. Using more sorbent mass than required has some downsides including increased cost, larger elution volume, and the potential increase in co-extraction of matrix interferences. In this paper, the

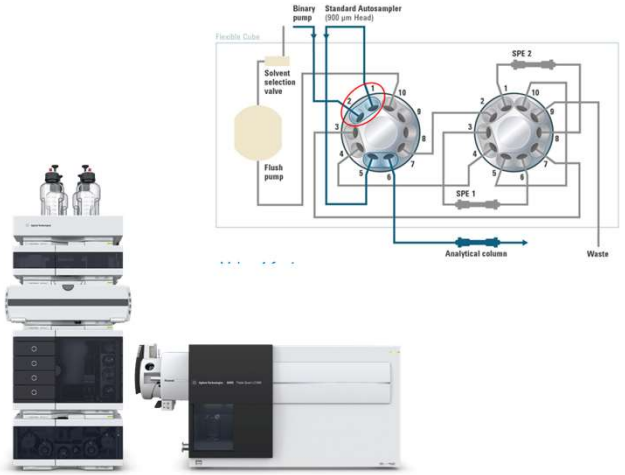
https://www.agilent.com/cs/library/applications/an-pfas-drinking-water-sampliq-wax-1290-infinity-lc-5994-3616en-agilent.pdf

Enrichment factor: 250

Strategies to achieve low level detection limits

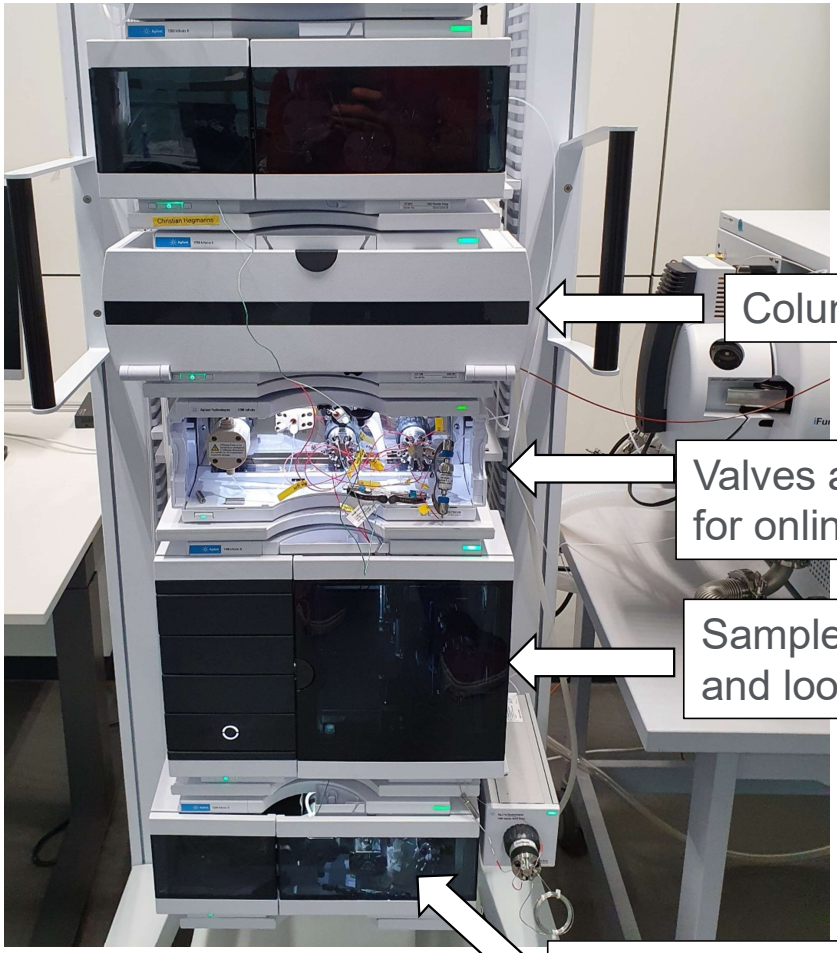
LC/MS after sample enrichment

Online-SPE LC/MS



LC/MS with direct injection

Hardware Setup, recommendation for online-SPE use only



Configuration:

1260 Infinity II Flexible Pump (G7104C), Analytical Pump

1260 Infinity II Flexible Pump (G7104C), Loading Pump

1290 Infinity II Multisampler (G7167B), **900 µL Head** installed

1290 Infinity II Multi Column Compartment (G7116B)

1290 Infinity Flexible Cube with online SPE starter kit and online SPE direct injection kit

6475 or 6495 Triple Quadrupole LC/MS

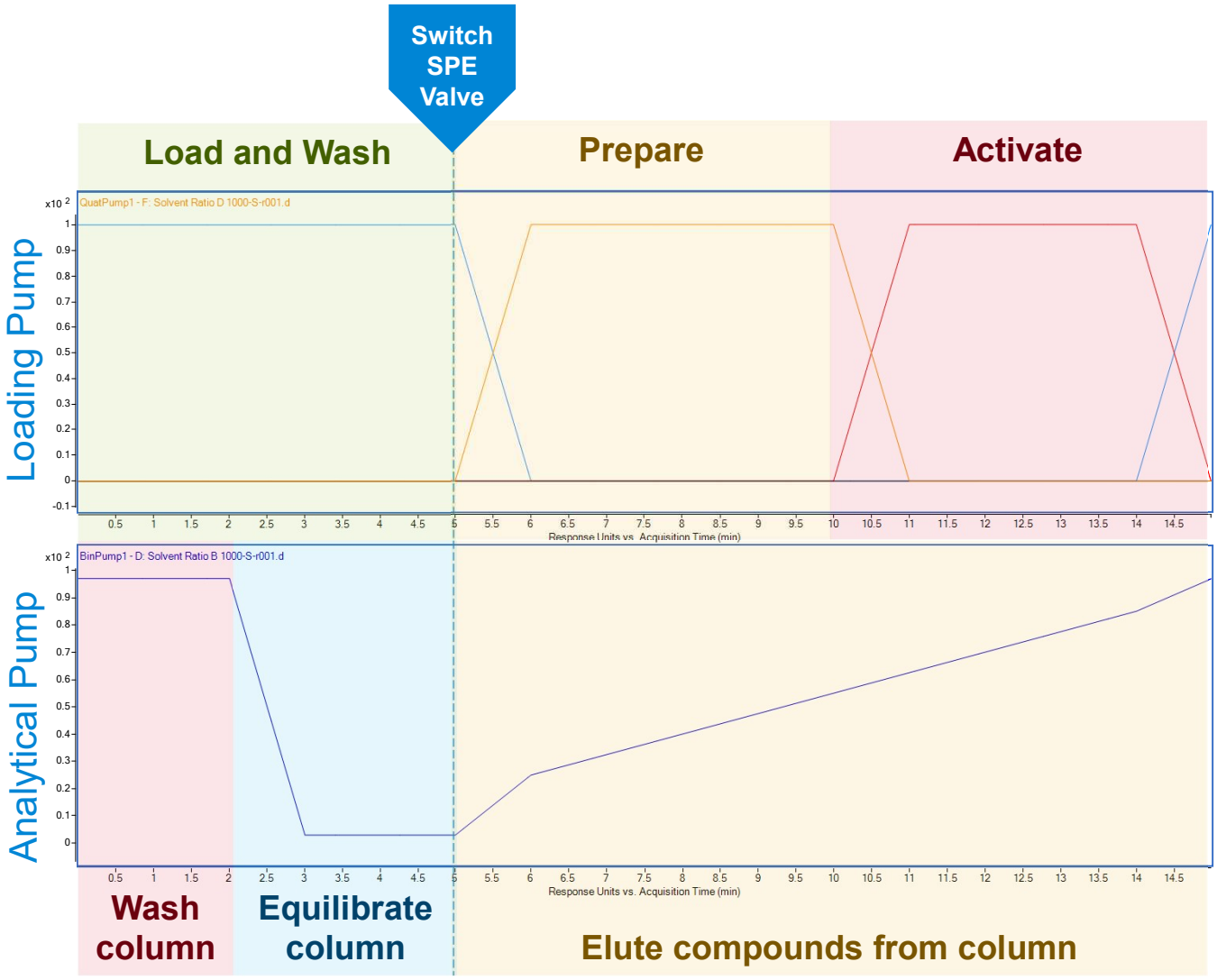
Programing Online SPE

During the first 5 minutes:

- The SPE pump loads and washes the sample on the SPE sorbent
- The Gradient pump washes and equilibrates the column.

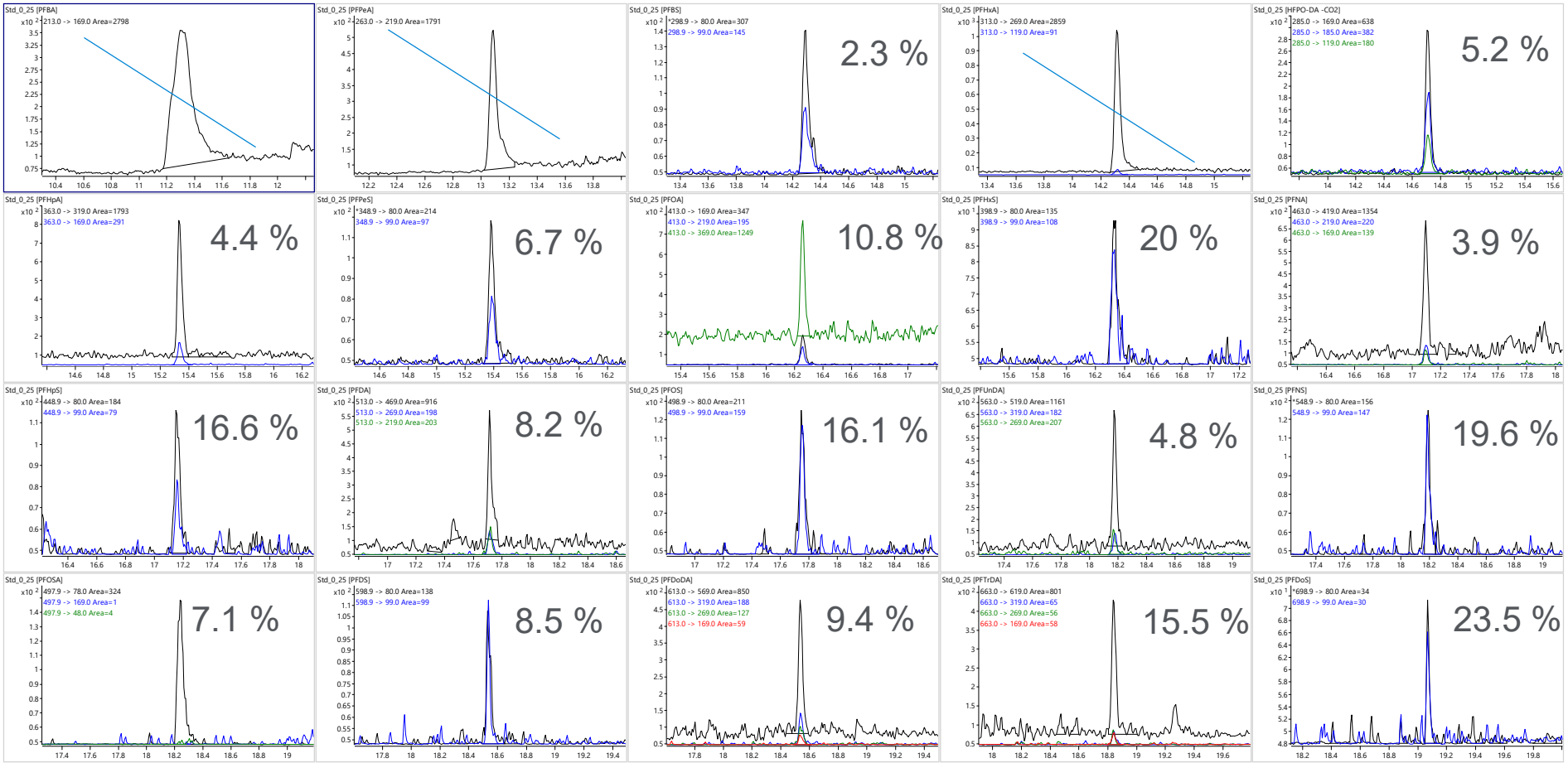
The next 10 minutes:

- The SPE pump prepares the sample loop and SPE sorbent, then activates the SPE, then loads the sample into the sample loop.
- The Gradient pump Elutes the compounds from the column.



Online SPE Sensitivity

% RSD @ 0.25 ng/L n=6



Agilent's Triple Quadrupole GC/MS Portfolio

Focused on reliability, robustness, and routine analysis applications

7000E GC/TQ

Inert Plus EI Source | CI Source | HydroInert Source

8890 GC

Intuvo 9000 GC | Intuvo 9000 GC

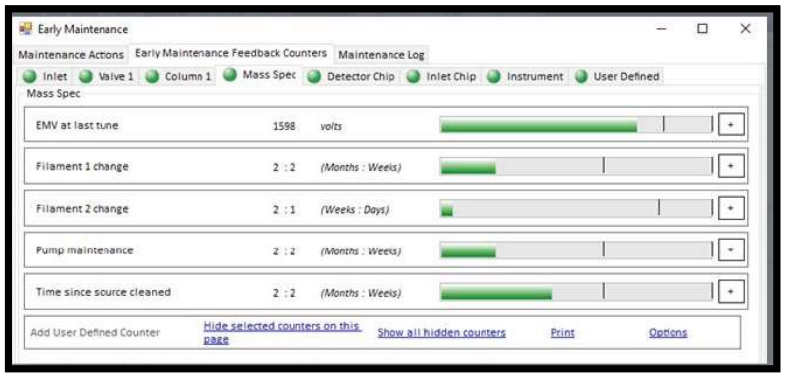
7010C GC/TQ

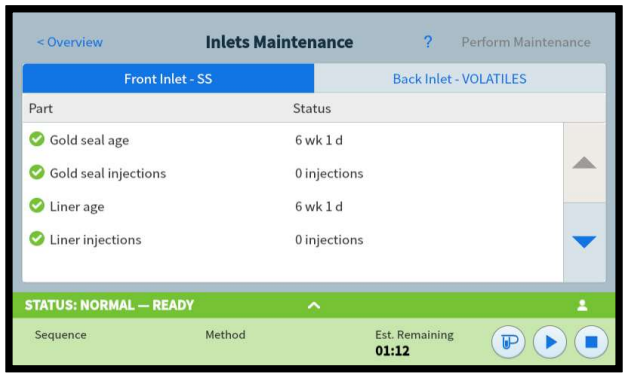
HES EI Source | CI Source

8890 GC

Intuvo 9000 GC

Next-Generation GC and Mass Spec Intelligence





Self-Aware Features

Diagnostic & maintenance options to prevent common GC and MS problems

Remote Connectivity

GC: Browser interface enables remote connectivity and access to instrument features

MS: detailed system report sent to service engineer for speedy diagnostic efforts

Performance Evaluation

Minimize downtime from contamination with automated chromatography checks

On-Board Help

Accessible via the touchscreen or browser interface

Reduce GC/MS Downtime

JetClean Self cleaning ion source

With innovative JetClean technology, your lab can:

- Increase instrument uptime. Fewer manual cleanings maximize productivity.
- Maintain data quality. A clean ion source ensures run-to-run reproducibility.
- Enhance operator convenience. Automated cleaning requires virtually no user intervention.

Without JetClean self-cleaning ion source

January X2	February X2	March X2
April X2	May X2	June X2
July X2	August X2	September X2
October X2	November X2	December X2

Twenty-four cleanings per year

With JetClean self-cleaning ion source*

January	February	March
April	May	June
July	August	September X1
October	November	December

One cleaning per year

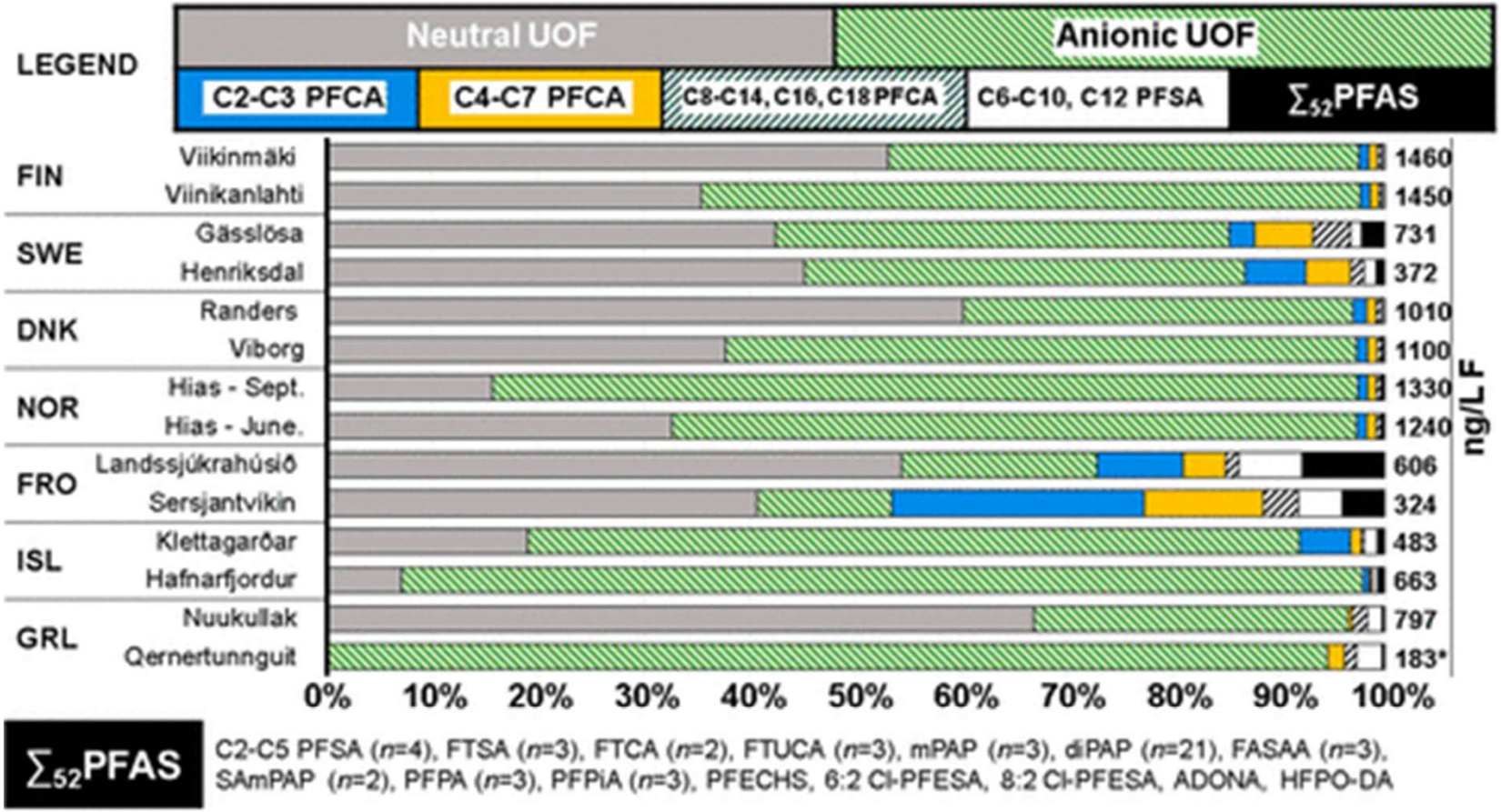
Cleaning frequency reduced by **up to 90%**

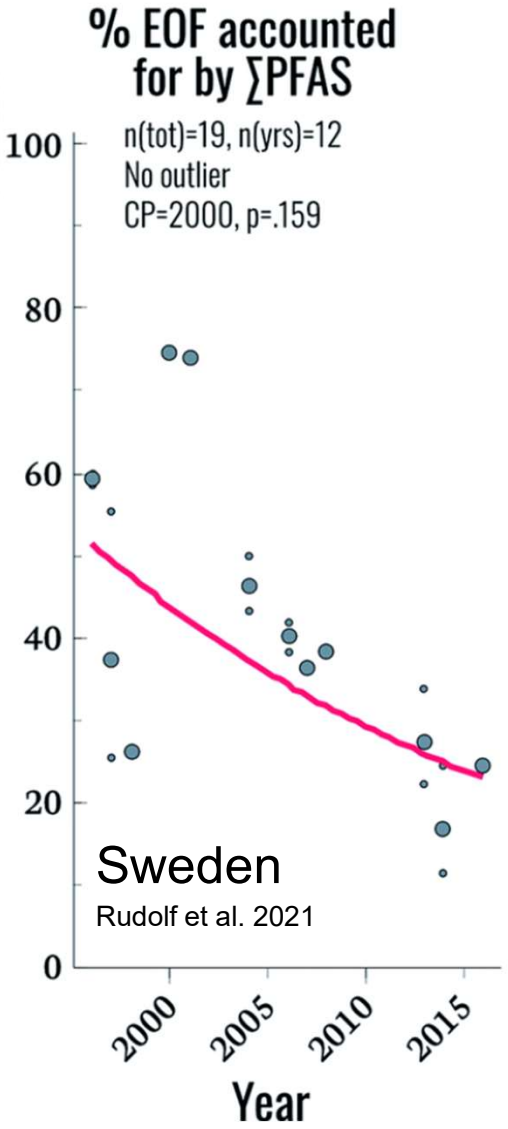
Non-Targeted PFAS Analysis

Agilent
Trusted Answers

Wastewater and Sewage is Mostly Unknown PFAS

Miaz et al. 2020





Rudolf et al. 2021, DOI: 10.1021/acsestwater.1c00168 *ACS EST Water* 2021, 1, 9, 2087–2096
Miaz et al. 2020, DOI: 10.1039/C9EM00502A *Environ. Sci.: Processes Impacts*, 2020, 22, 1071-1083

Why Non-Targeted Analysis of PFAS?

Challenges

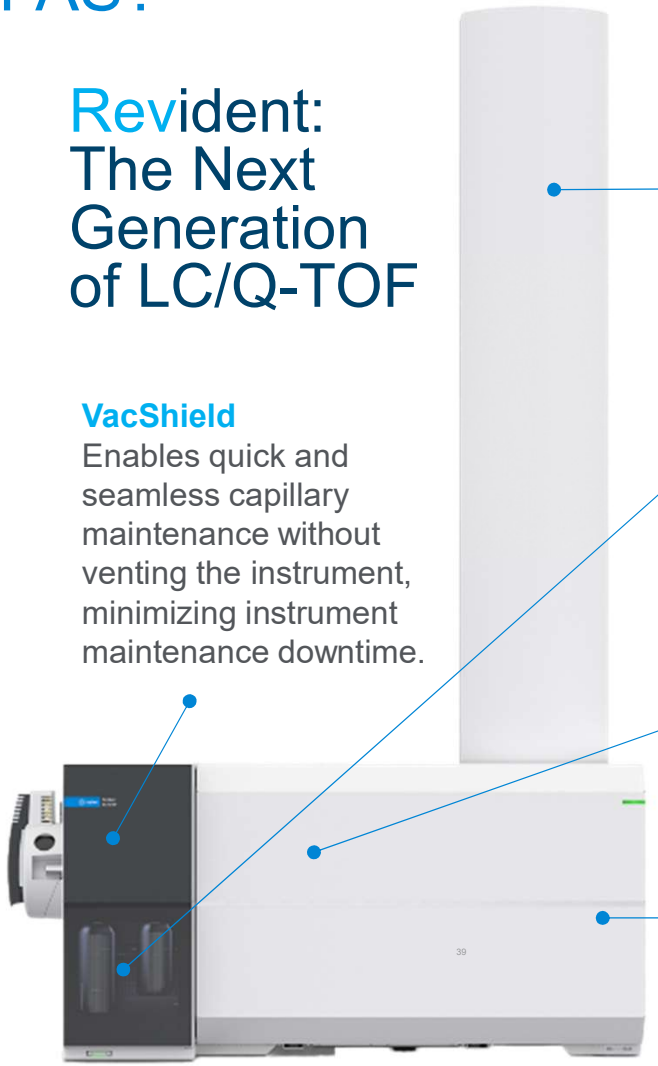
- More than 14,000 PFAS structures in EPA CompTox Dashboard
- However, there are very few standards, and are prohibitively expensive.
- Both targeted and non-targeted solutions are required, depending on the study goals.
- Non-target tools needed to aid in putative identification of novel PFAS

Agilent Non-Targeted PFAS Solution

- High resolution accurate mass LC-QTOF MS with end-to-end intelligence
- FluoroMatch PFAS annotation software

[CompTox Chemicals Dashboard (epa.gov)]

Revident: The Next Generation of LC/Q-TOF



VacShield
Enables quick and seamless capillary maintenance without venting the instrument, minimizing instrument maintenance downtime.

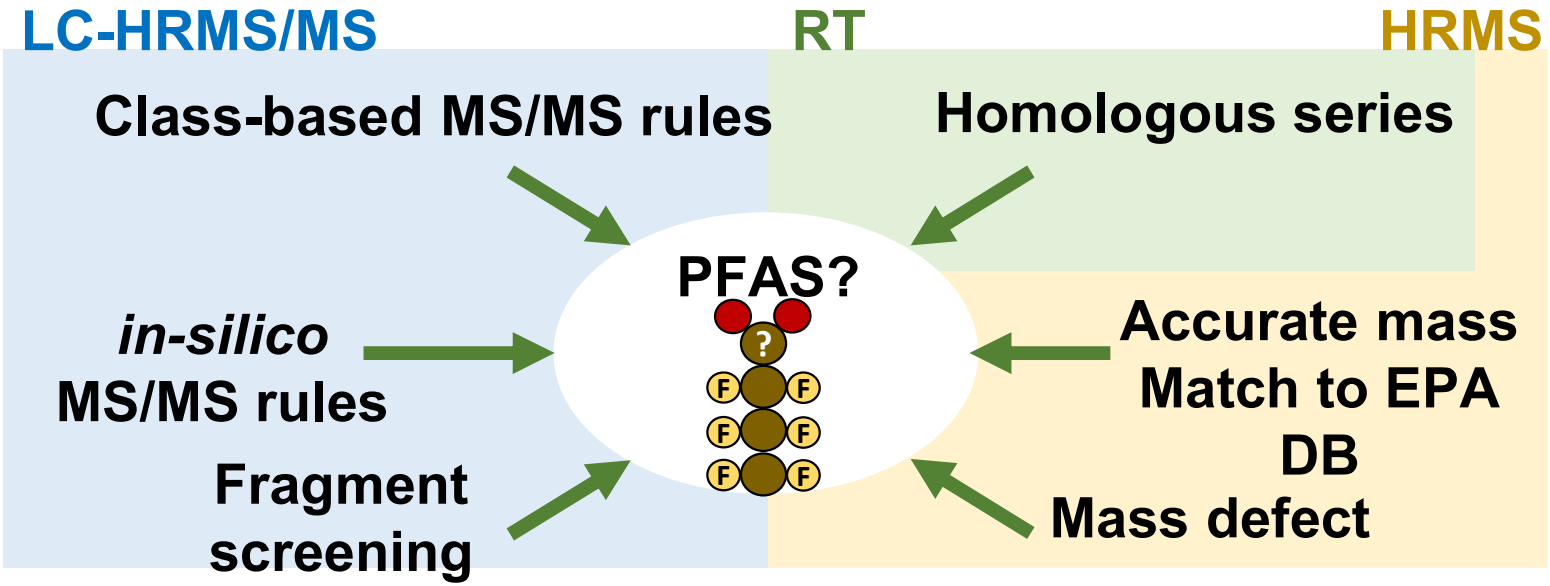
Temperature-stabilized flight tube
Temperature sensors along the flight tube and heater controls allow for maximum temperature stability and mass accuracy.

Improved CDS
A new pump-based calibrant delivery system (CDS) permits constant and reliable flow rates for scheduled autotunes and calibration.

New Ion Optics
Robustness proven ion optics from Agilent's Triple Quads ensure robustness for the analyses.

New Detector
A completely new ADC Detector delivers increased mass resolution at low *m/z*, better dynamic range and improved isotopic fidelity.

FluoroMatch Annotation – Incorporating RT, MS, and MS/MS



- Fluoromatch PFAS is an open source library/workflow from University of Yale
- Workflow fully supported in MassHunter
- Includes around 7000 PFAS compound.

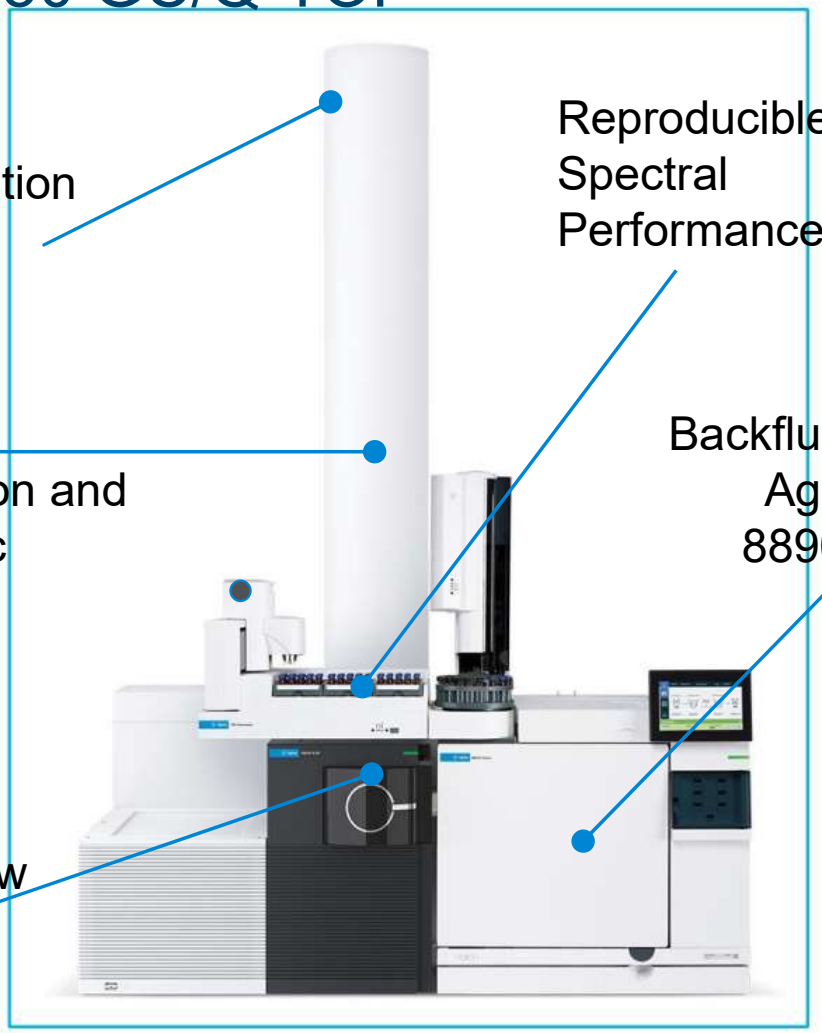
To download FluoroMatch software: http://innovativeomics.com/software/fluoromatch-flow-covers-entire-pfas-workflow/

Non-target GC/MS Q-TOF

Challenges

- GC/MS is typically used for detecting volatile and non-polar PFAS compounds.
- GC/Q-TOF system to take advantage of high resolution for detecting compounds
- For specific and sensitive PFAS detection in soil and drinking water, we have created an accurate mass GC/MS library for PFAS.
- Will also allow identification of other contaminants in drinking water such as disinfection byproducts, industrial chemicals originated from personal care products, drugs, and pesticide residues.

Agilent 7250 GC/Q-TOF



Non-target GC/MS Q-TOF PCDL library

Accurate Mass Library for PFAS for more than 100 PFAS

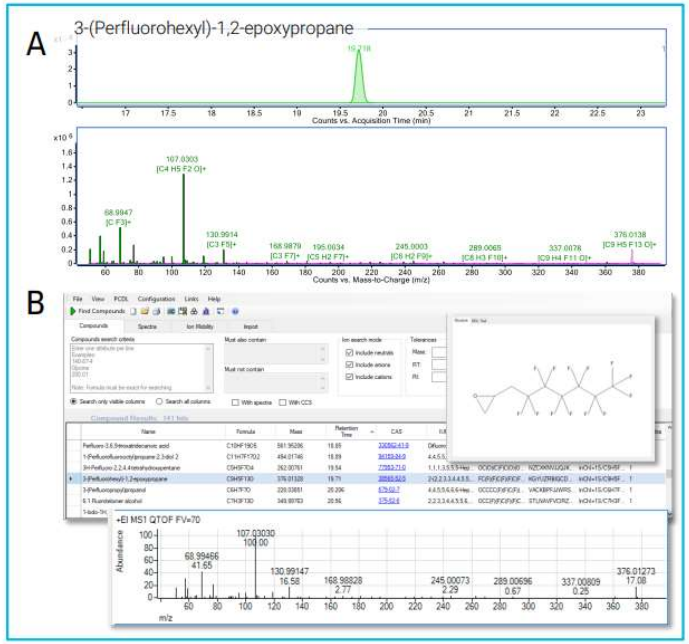


Figure 2. (A) EIC of the molecular ion and fragment formula annotation of spectrum for one of the PFAS compounds in MassHunter Qualitative Analysis software. (B) The PFAS PCDL contains EI spectra as well as the metadata including molecular structure and database identifiers.

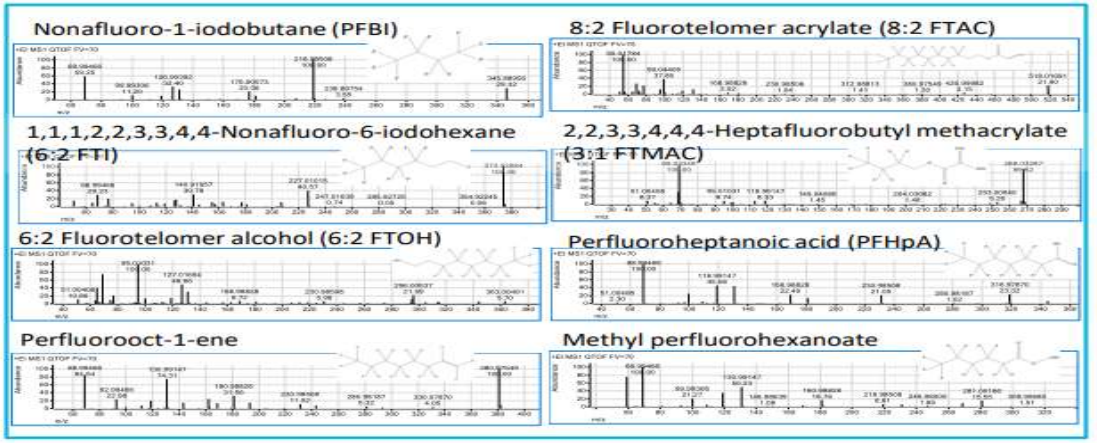


Figure 3. Examples of spectra in PFAS PCDL from different PFAS compound classes.

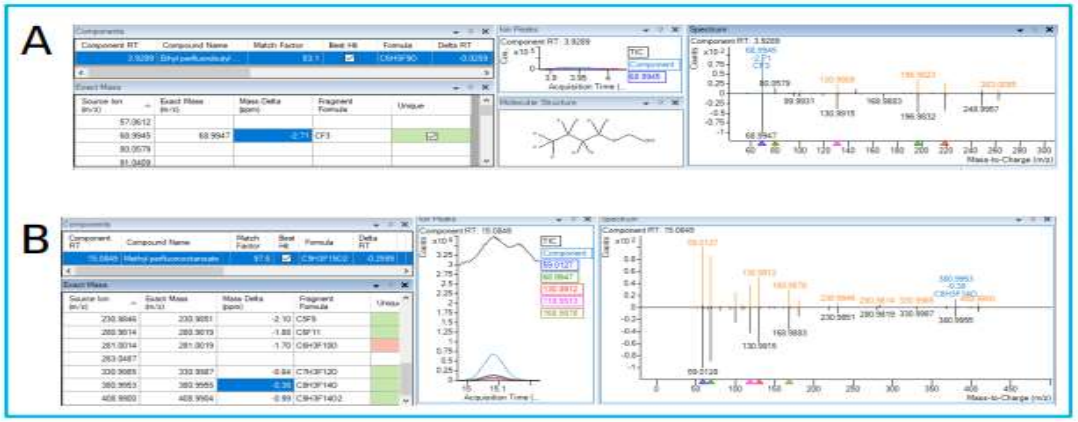


Figure 4. PFAS identified in soil Field 1 (A, ethyl perfluorobutyl ether) and drinking water Irvine (B, Methyl perfluorooctanoate) samples.

Automated Sample Prep of water samples - LCTech

- Automated SPE – Walk away operation
- modular with SPE, GPC and evaporation
- From small sample tubes to flasks of 10 l
- Up to 30 x 250 ml samples
- Up to 4 l sample (10 l possible)
- Up to 6 samples simultaneously
- No need for fume hood

Automated Sample Prep of solid samples - LCTech

From extraction of samples to extract ready for injection

X-TRACTION PFAS

For extraction

D-EVA

For the sensor-controlled concentration of extracts

FREESTYLE SPE PFAS

For clean-up (e.g. Dual-SPE). Ideal with the EluCLEAN SPE columns.

D-EVA

For sensor-controlled concentration to a few µL

Automated Sampling of trace PFAS in air - Markes

- Thermal desorption
- Can be combined with GC/MS SQ, TQ or QTOF for automated sampling and analysis of PFAS in air

PFAS i skiwax - FTIR

Summary

- Agilent's integrated targeted and non-targeted workflows for PFAS analysis have the advantage of measuring PFAS from various matrices with great sensitivity, accuracy, and reliability. Agilent offers complete workflows from sample prep, separation, detection, data analysis, and reporting for PFAS analysis.
- Both GC-MS and LC-MS systems have the smart features to increase uptime and simplify usage, reducing the economic footprint of the lab. The Intelligent Reflex of the LC-MS systems automatically reruns the same vial when the sample is out of calibration range or if the blank is contaminated.
- Agilent/Matriks can help you with implementing a PFAS workflow in your laboratory with our expert in-house application services.