Advances in automated sample preparation and analysis of polyfluoroalkyl substances in the environmental water samples

Tarun Anumol[1], James Pyke[1],
[1] Agilent Technologies Inc., USA

PFAS related products

Common household products and industrial uses

PFAS related products

Common laboratory materials

Solvent caps, filters and tubing

Caps on samples vials

Pump seals, frits, degasser materials

Air conditioning filters

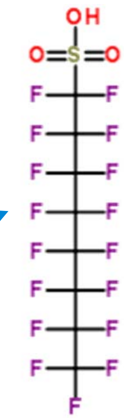
Gloves/Coats

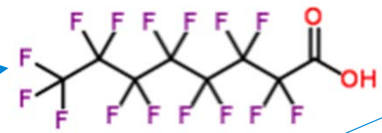
Sample preparation consumables

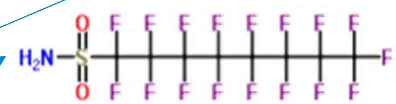
Regulated PFAS monitoring

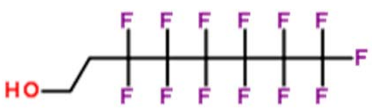
Common target compounds

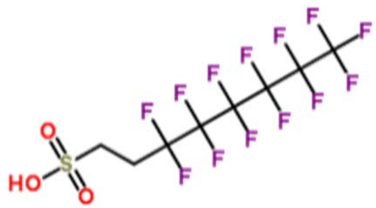
Compound	Formula	Abbreviation
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluro-n-butanoic acid	$C_4HF_7O_2$	PFBA
Perfluoro-n-pentanoic acid	$C_5HF_9O_2$	PFPeA
Perfluoro-n-hexanoic acid	$C_6HF_{11}O_2$	PFHxA
Perfluoro-n-heptanoic acid	$C_7HF_{13}O_2$	PFHpA
Perfluoro-n-octanoic acid	$C_8HF_{15}O_2$	PFOA
Perfluoro-n-nonanoic acid	$C_9HF_{17}O_2$	PFNA
Perfluoro-n-decanoic acid	$C_{10}HF_{19}O_2$	PFDA
Perfluoro-n-undecanoic acid	$C_{11}HF_{21}O_2$	PFUdA
Perfluoro-n-dodecanoic acid	$C_{12}HF_{23}O_2$	PFDoA
Perfluoro-n-tridecanoic acid	$C_{13}HF_{25}O_2$	PFTrDA
Perfluoro-n-tetradecanoic acid	$C_{14}HF_{27}O_2$	PFTeDA
Perfluoro-n-pentadecanoic acid	$C_{15}HF_{29}O_2$	PFPeDA
Perfluorinated sulfonates (PFSAs)		
Perfluoro-1-butanesulfonate	$C_4F_9SO_3^-$	PFBS
Perfluoro-1-hexanesulfonate	$C_6F_{13}SO_3^-$	PFHxS
Perfluoro-1-octanesulfonate	$C_8F_{17}SO_3^-$	PFOS
Perfluoro-1-decanesulfonate	$C_{10}F_{21}SO_3^-$	PFDS
Perfluorinated sulfonamides (FOSA)		
Perfluoro-1-octansulfonamide	$C_8H_2F_{17}NO_2S$	FOSA
N-Methylperfluoro-1-octanesulfonamide	$C_9H_4F_{17}NO_2S$	N-MeFOSA
N-Ethylperfluoro-1-octanesulfonamide	$C_{10}H_6F_{17}NO_2S$	N-EtFOSA
Perfluorinated sulfonamidoethanols (FOSE)		
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	$C_{11}H_8F_{17}NO_3S$	N-MeFOSE
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	$C_{12}H_{10}F_{17}NO_3S$	N-EtFOSE
Fluorinated Telomer Sulfonates (FTS)		
1H,1H,2H,2H-Perfluorohexanesulfonic acid	$C_6H_5F_9SO_3$	4:2 FTS
1H,1H,2H,2H-perfluorooctane sulfonate	$C_8H_5F_{13}SO_3$	6:2 FTS
1H,1H,2H,2H-Perfluorodecanesulfonic acid	$C_{10}H_5F_{17}SO_3$	8:2 FTS
Perflourinated sulfonamidoacetic acids (FOSAA)		
Perfluorooctane sulfonamidoacetic acid	$C_{10}H_4F_{17}NO_4S$	FOSAA
N-methylperfluorooctane sulfonamidoacetic acid	$C_{11}H_6F_{17}NO_4S$	N-MeFOSAA
N-ethylperfluorooctane sulfonamidoacetic acid	$C_{12}H_8F_{17}NO_4S$	N-EtFOSAA

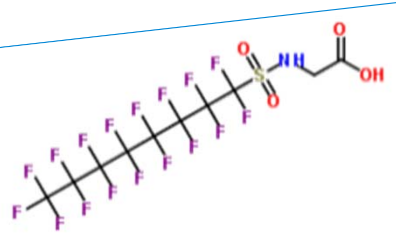












PFAS Analysis – LC Instrument Setup

Eliminate Background Contamination

Recommended Plumbing Configurations for the Reduction of PFAS background

5991-7863EN

Recommended Plumbing Configurations for Reduction in Per/Polyfluoroalkyl Substance Background with Agilent 1260/1290 Infinity (II) LC Systems

Application Note

Environmental

Authors

Tarun Anumol
Agilent Technologies Inc.
2850 Centerville Rd,
Wilmington, DE, USA

Sheher Bano Mohsin, and
Michael Woodman
Agilent Technologies Inc.
Wooddale, IL, USA

Peter Abrahamsson
Agilent Technologies Ltd.
Sweden

Introduction

Per/Polyfluoroalkyl substances (PFASs), also known as perfluorinated compounds (PFCs), are substances that have a few or all of the hydrogen atoms in an alkyl chain replaced by fluorine. These C-F bonds give them uniquely desirable properties for use in industry and commerce. However, PFASs are persistent and bioaccumulative in the environment and wildlife, making them contaminants of concern. Their wide ranging use leads to emission into the environment. PFASs such as perfluorooctanoic acid (PFOA) and perfluorooctane sulfanoic acid (PFOS) are ubiquitous, and have been detected in water, soil, sludge, and biota in the parts per billion (ppb) or parts per trillion (ppt) range. As a result, The USEPA has recently issued a combined drinking water health advisory for PFOA and PFOS at 70 ng/L. Several states in the US have drinking water advisories that range from 20–400 ng/L for some of these PFASs.

Since PFASs are routinely used in manufacturing processes, it is possible to have system contamination that may be caused by solvents, tubing, fittings, filters, and other parts used in the manufacture and routine operation of a liquid chromatograph. Polytetrafluoroethylene (PTFE) is a fluoropolymer used in all major (U)HPLC systems, and can be a potential source of PFAS contamination during analysis. This application note describes the various potential sources of PFAS contamination in an Agilent 1260 Infinity II and an Agilent 1290 Infinity II system, while providing a solution to significantly reduce PFAS background interference to allow sensitive quantification of these analytes with Agilent LC/MS systems. This application note is intended for users with dedicated LC systems to perform ultra-low trace level analysis of PFASs with minimal background. For users performing multiple analyses on the same LC, the use of a delay column is suggested to reduce PFAS background.

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PFAS Analysis – LC Instrument Setup

Eliminate Background Contamination

Potential Contamination Sources

- Solvent Filtration apparatus
- PTFE lined tubing
- PTFE containing caps

Replace Inlet Lines with Peek

Replace Bottle Caps with PP Caps (Hole Drilled)

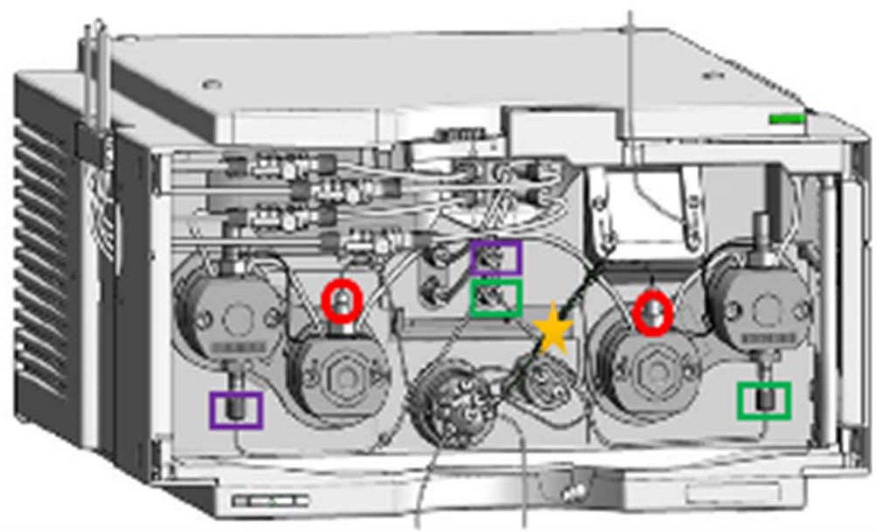
Replace Glass Sinkers with Stainless Steel Sinkers

PFAS Analysis – LC Instrument Setup

Eliminate Background Contamination

Potential Contamination Sources

- Solvents
- PTFE materials in LC flow path



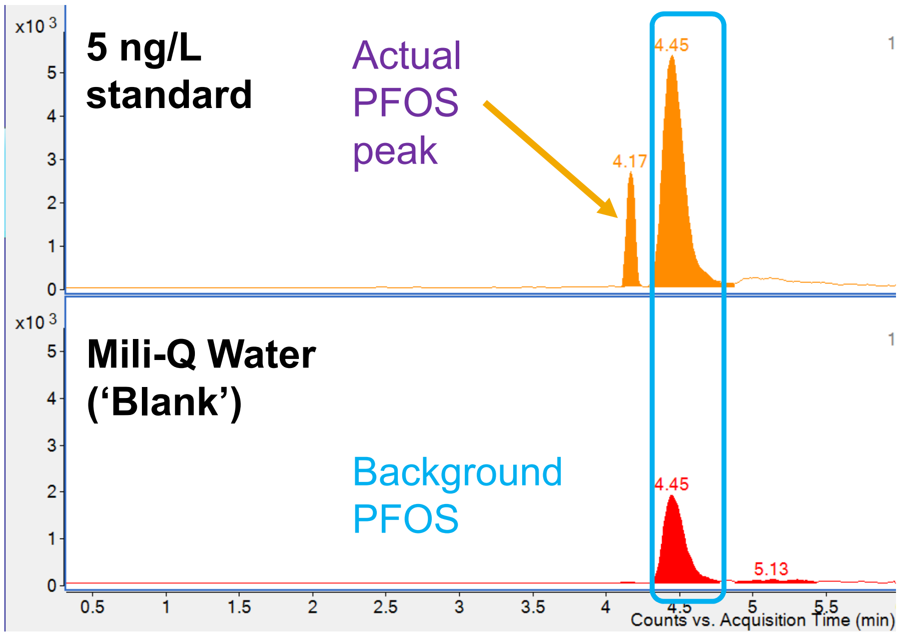
Remove PTFE filters

Add delay column (Zorbax Eclipse Plus C-18, 4.6x50 mm)

Bypass degasser using 1/8" PEEK tubing from solvent bottles directly to inlet check valve

PFAS Analysis – LC Instrument Setup

Eliminate Background Contamination



PFAS Analysis – LC Instrument Setup
Eliminate Background Contamination

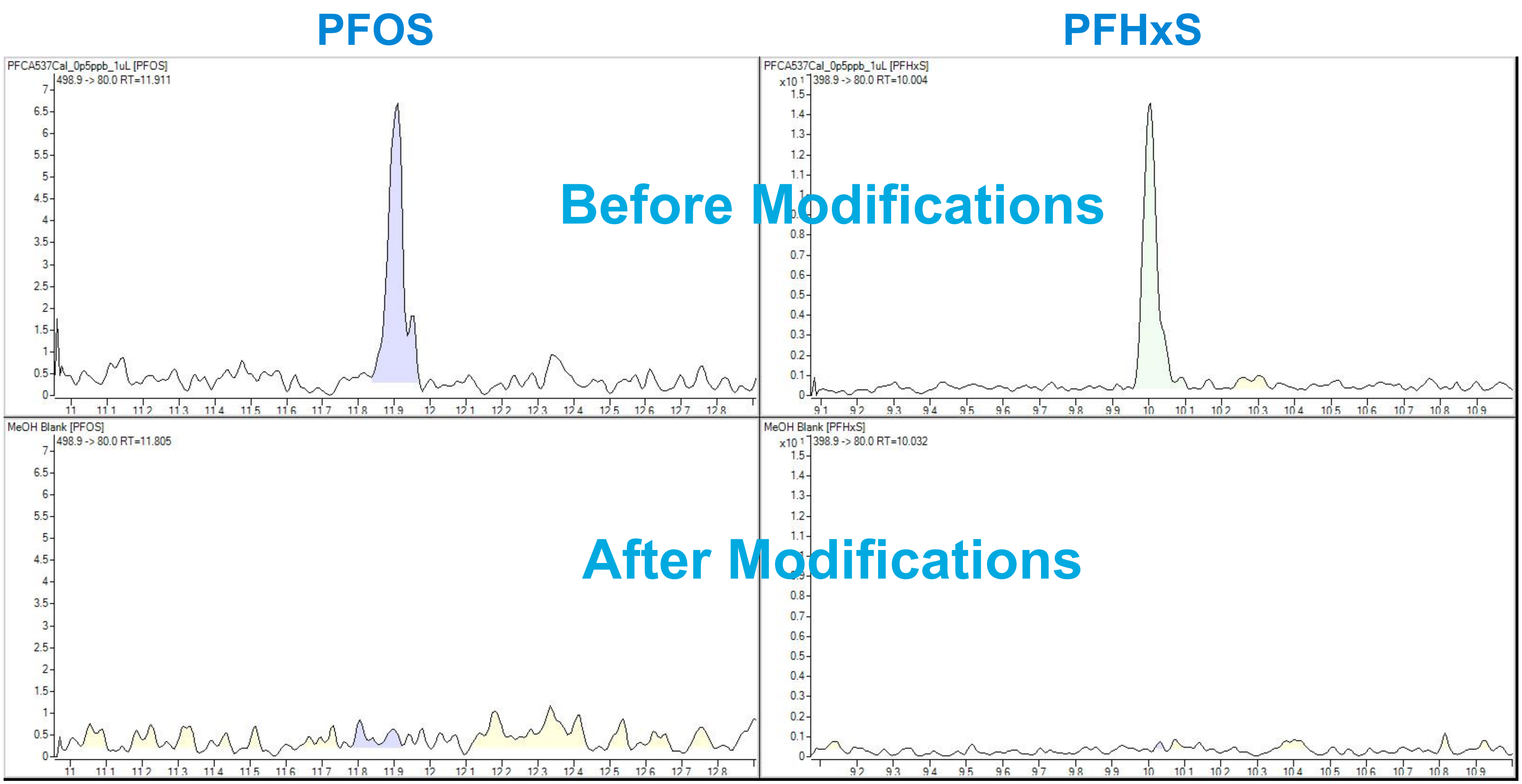
Potential Contamination Sources

- Solvents
- PTFE materials in LC flow path

Use snap top polyethylene membrane caps (Septa of regular caps has PTFE)

Replace rotor seal with VESPEL

PFAS Analysis – LC Instrument Setup
Eliminate Background Contamination



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Regulations

Global trend to monitor broader range of PFAS at lower levels

News Releases from Headquarters > Water (OW)

EPA Acting Administrator Announces First-Ever Comprehensive Nationwide PFAS Action Plan

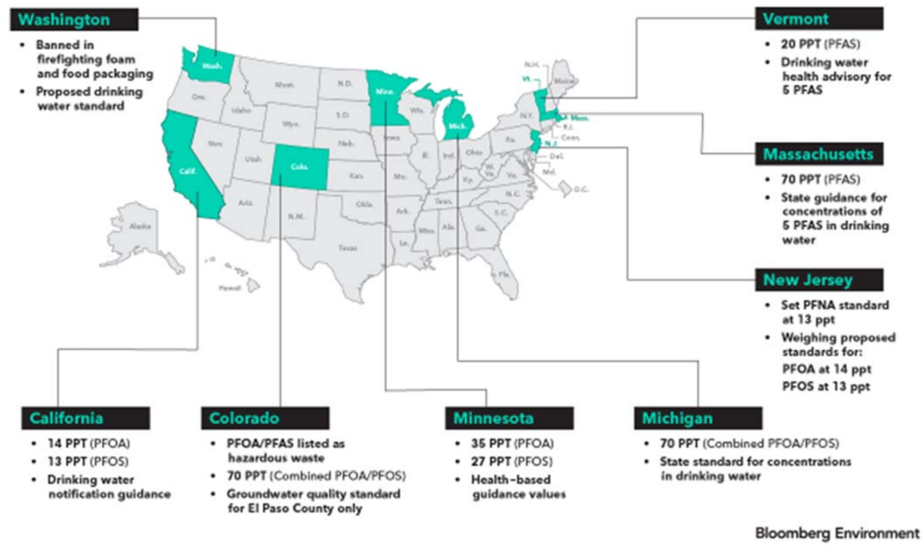
Historic plan outlines concrete steps the agency is taking to address PFAS and to protect public health

02/14/2019

https://www.epa.gov/newsreleases/epa-acting-administrator-announces-first-ever-comprehensive-nationwide-pfas-action-plan

The proposal is to regulate the group of PFASs, as defined by the OECD[60], and to suggest values of **0.1 µg/l for individual PFAS and 0.5 µg/l for PFASs in total**, as is done for pesticides. As these values are higher than those referred to in Sweden or the United States, it should be feasible to meet them.

http://ec.europa.eu/environment/water/water-drink/pdf/revised_drinking_water_directive.pdf



https://pfasproject.com/2018/10/02/analysis-of-state-by-state-differences-in-pfas-regulation/

Our aims…

1. To develop **efficient** analytical techniques by:
   - Reducing sample preparation time
   - Reducing data acquisition time
2. To **mitigate risk** involved in PFC analysis by:
   - Simplifying and automating sample preparation steps
   - Prove results through proficiency testing at NMI
3. To be **fast** and **effective** with the data analysis workflow by:
   - Simplifying the data analysis interface
   - Exploit the ‘Review-by-exception’ workflow
   - Include customer calculations in the interface and report
4. To configure a system that is **flexible** and offers **redundancy**
   - Accommodate multiple, related environmental applications
5. To increase **ROI** by
   - Allow skilled operators grow business
   - Exploit small footprint technology

Range of analytical efficiency…

Faster, cheaper, quicker

1D-LC

- Most published
- Simple LC configuration

Automatic Column Regeneration

- Reduce analysis time, using traditional 1D-LC techniques
- Requires additional LC pump and valve

Online SPE

- Reduce and automate sample preparation time
- Requires additional LC pump and valve

Rapid LC

- Reduce detector time
- Increase ROI
- Requires dual needle autosampler and a High Speed LC pump

Traditional 1D-LC

- LC configuration:
  - Binary pump, Autosampler, Column compartment
- Typically 10-20 mins injection-to-injection, depending on the matrix type

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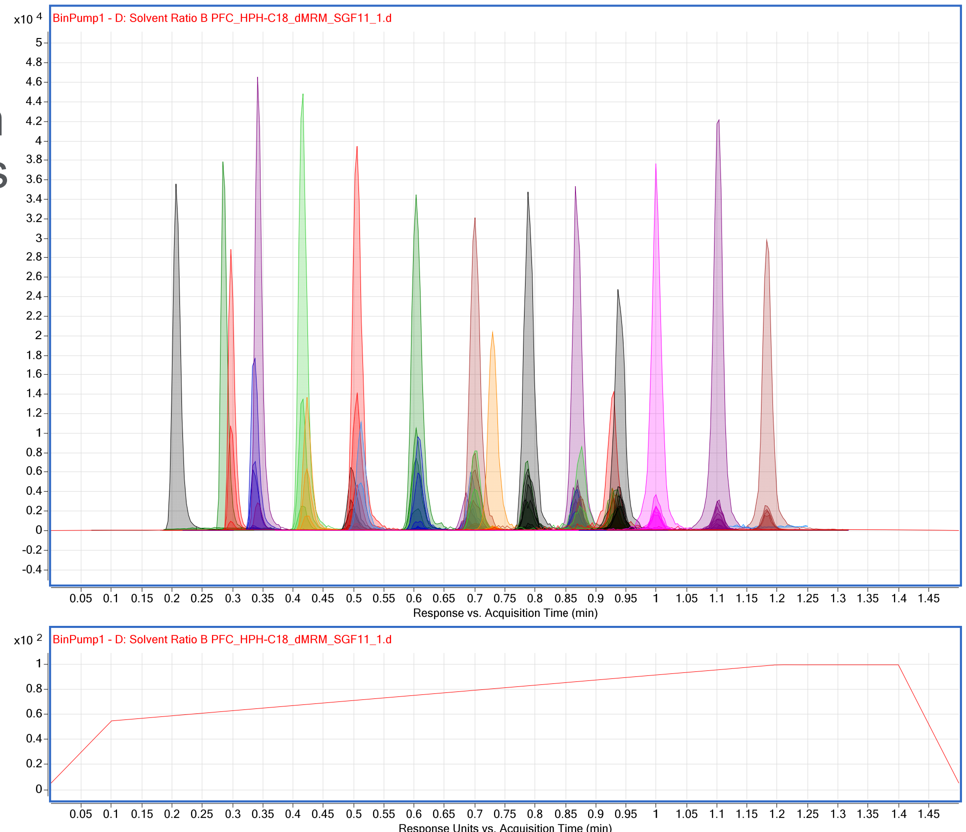
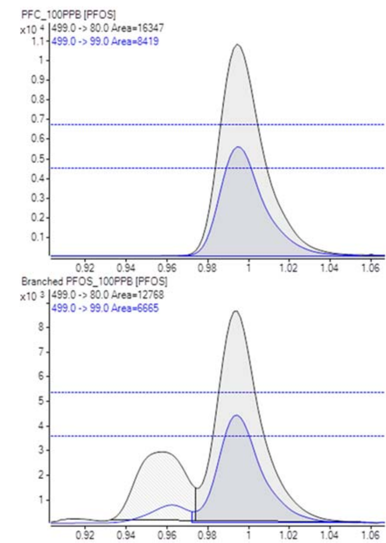
Rapid LC with dual needle 1290 Infinity II Multisampler

1.5 minute injection-to-injection cycle time for analysis of PFC's

Chromatographic resolution of branched PFOS shown below.



90 seconds !



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Rapid LC with dual needle 1290 Infinity II Multisampler

Testing Robustness with soil and water samples

294 injections

11h 36m total run time

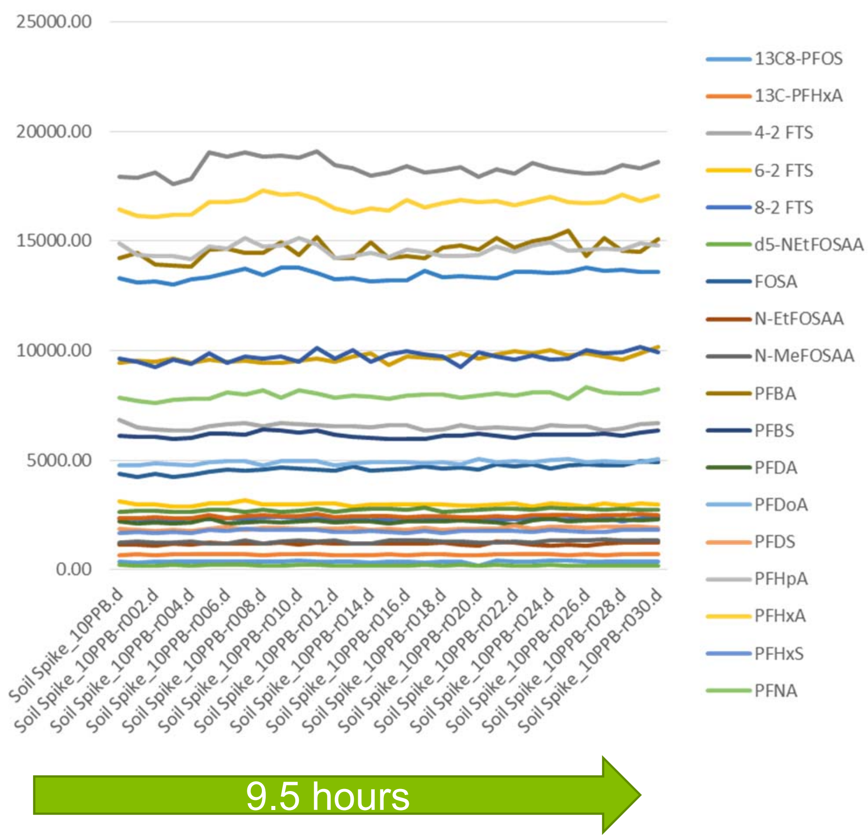
2:36 mins injection-to-injection (without dual needle or ACR)

Sample	Position	Method	File name	Sample Type	Level
DB	No Injection	Rapid_PFCs-EclipsePlusC18_dMRM.m	DB-r001.d	DoubleBlank	
PFC_0.1PPB	P7-D1	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_0.1PPB.d	Calibration	0.1
PFC_1PPB	P7-D2	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_1PPB.d	Calibration	1
PFC_5PPB	P7-D3	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_5PPB.d	Calibration	5
PFC_10PPB	P7-D4	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_10PPB.d	Calibration	10
PFC_25PPB	P7-D5	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_25PPB.d	Calibration	25
PFC_50PPB	P7-D6	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_50PPB.d	Calibration	50
PFC_100PPB	P7-D7	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_100PPB.d	Calibration	100
ICC_50PPB	P7-D8	Rapid_PFCs-EclipsePlusC18_dMRM.m	ICC_50PPB.d	CC	50
DB	No Injection	Rapid_PFCs-EclipsePlusC18_dMRM.m	DB-r001-r001.d	DoubleBlank	
ICC_50PPB	P7-D8	Rapid_PFCs-EclipsePlusC18_dMRM.m	ICC_50PPB-r001.d	CC	50
Branched PFOS_100PPB	P7-D9	Rapid_PFCs-EclipsePlusC18_dMRM.m	Branched PFOS_100PPB-r001.d	Sample	100
Water Blank_0PPB	P7-E1	Rapid_PFCs-EclipsePlusC18_dMRM.m	Water Blank_0PPB-r001.d	Blank	
Water Spike_25PPB	P7-E2	Rapid_PFCs-EclipsePlusC18_dMRM.m	Water Spike_25PPB-r001.d	QC	25
Soil Blank_0PPB	P7-E3	Rapid_PFCs-EclipsePlusC18_dMRM.m	Soil Blank_0PPB-r001.d	Blank	
Soil Spike_10PPB	P7-E4	Rapid_PFCs-EclipsePlusC18_dMRM.m	Soil Spike_10PPB-r001.d	QC	10
PFOS Soil Sample_2PPB	P7-E5	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFOS Soil Sample_2PPB-r001.d	Sample	
PFOS Soil Spike_12PPB	P7-E6	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFOS Soil Spike_12PPB-r001.d	MatrixSpike	12
DB	No Injection	Rapid_PFCs-EclipsePlusC18_dMRM.m	DB-E.d	DoubleBlank	
PFC_0.1PPB	P7-D1	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_0.1PPB-E.d	Calibration	0.1
PFC_1PPB	P7-D2	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_1PPB-E.d	Calibration	1
PFC_5PPB	P7-D3	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_5PPB-E.d	Calibration	5
PFC_10PPB	P7-D4	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_10PPB-E.d	Calibration	10
PFC_25PPB	P7-D5	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_25PPB-E.d	Calibration	25
PFC_50PPB	P7-D6	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_50PPB-E.d	Calibration	50
PFC_100PPB	P7-D7	Rapid_PFCs-EclipsePlusC18_dMRM.m	PFC_100PPB-E.d	Calibration	100

Repeated 30x

Rapid LC with dual needle 1290 Infinity II Multisampler

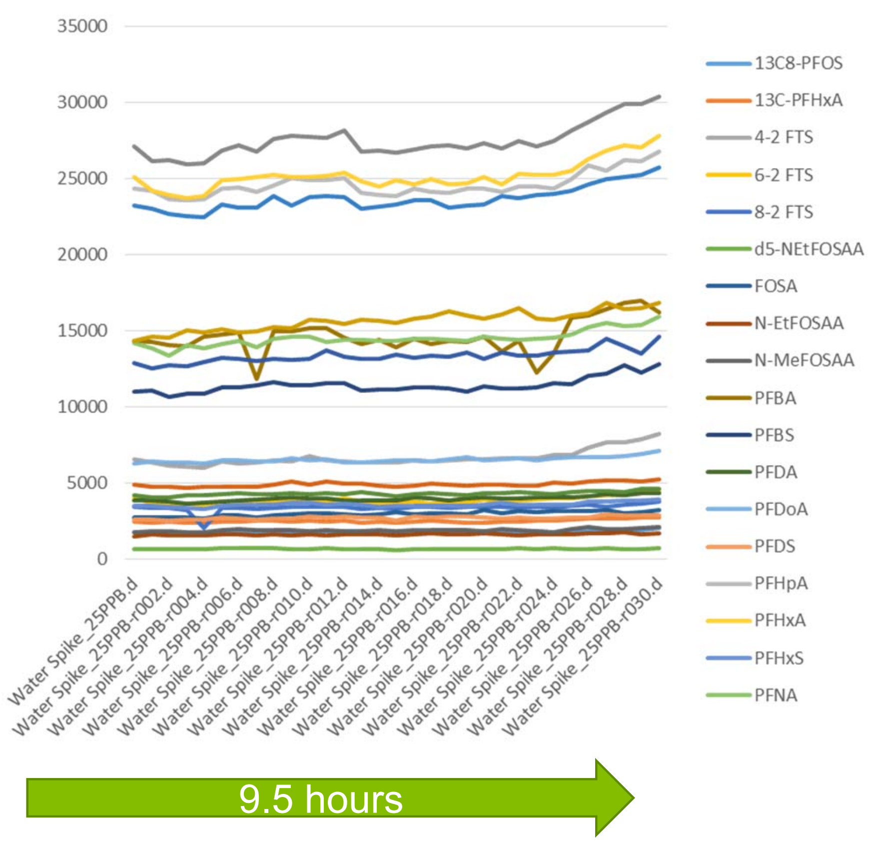
Repeatability of 30 soil extracts



Compound	Area %CV	RT %CV
4-2 FTS	1.8%	0.14%
6-2 FTS	2.2%	0.17%
8-2 FTS	1.9%	0.17%
FOSA	3.9%	0.23%
N-EtFOSAA	3.9%	0.17%
N-MeFOSAA	3.9%	0.17%
PFBA	2.8%	0.15%
PFBS	1.9%	0.17%
PFDA	3.3%	0.15%
PFDoA	1.7%	0.17%
PFDS	3.4%	0.20%
PFHpA	1.8%	0.10%
PFHxA	1.9%	0.10%
PFHxS	2.7%	0.10%
PFNA	2.0%	0.10%
PFOA	1.6%	0.07%
PFOS	2.4%	0.08%
PFPeA	2.2%	0.08%
PFTeDA	2.1%	0.06%
PFTrDA	2.4%	0.09%
PFUnA	2.2%	0.06%

Rapid LC with dual needle 1290 Infinity II Multisampler

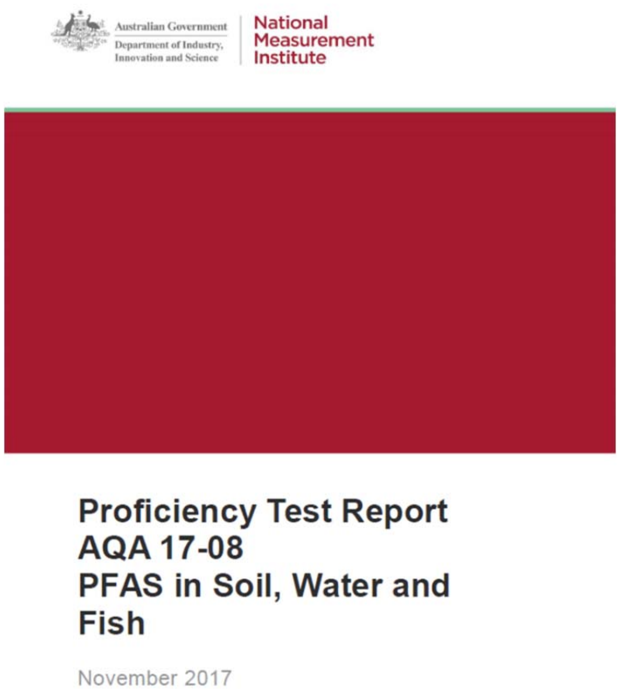
Repeatability of 30 water extracts



Compound	Area %CV	RT %CV
4-2 FTS	7.9%	0.19%
6-2 FTS	5.4%	0.18%
8-2 FTS	8.1%	0.18%
FOSA	5.3%	0.25%
N-EtFOSAA	3.5%	0.19%
N-MeFOSAA	4.8%	0.16%
PFBA	7.8%	0.16%
PFBS	4.4%	0.14%
PFDA	4.2%	0.31%
PFDoA	2.8%	0.16%
PFDS	3.8%	0.16%
PFHpA	3.2%	0.13%
PFHxA	3.8%	0.13%
PFHxS	4.0%	0.11%
PFNA	3.6%	0.12%
PFOA	3.3%	0.08%
PFOS	3.2%	0.08%
PFPeA	4.1%	0.07%
PFTeDA	4.2%	0.06%
PFTrDA	3.4%	0.07%
PFUnA	3.0%	0.06%

AQA 17-08 NMI Proficiency test results

Analytical method details

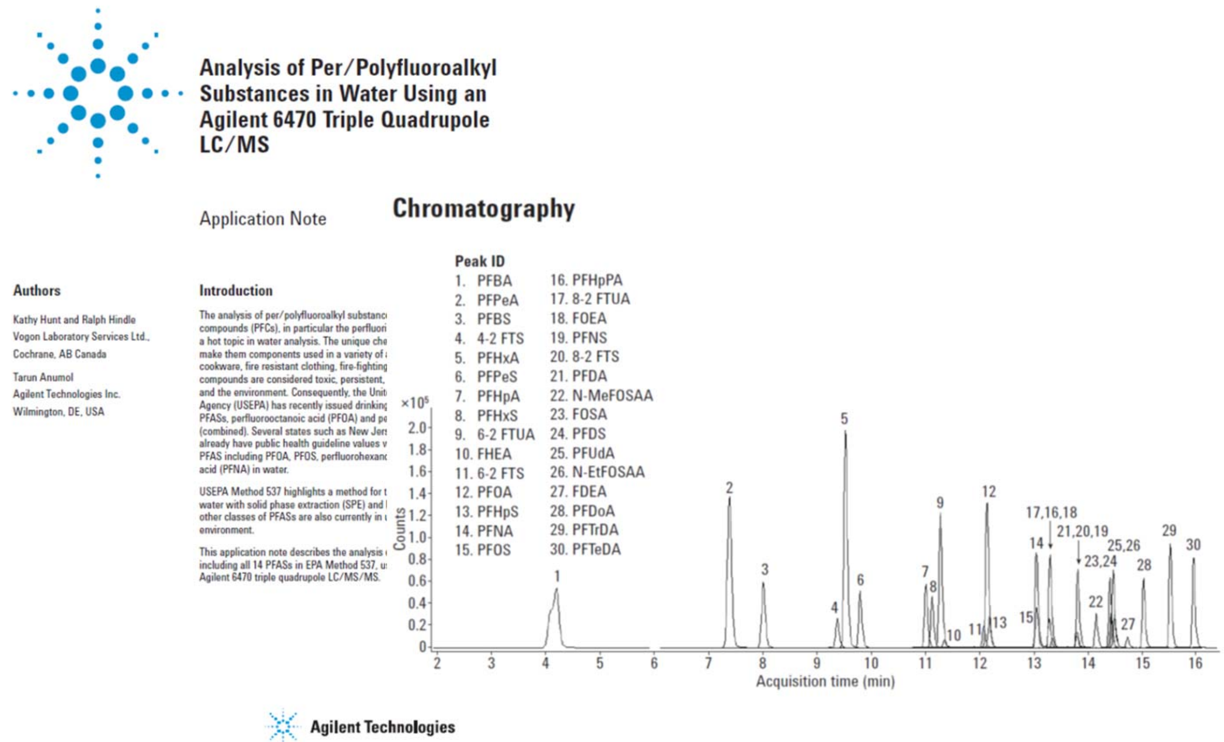


Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

Proficiency Test Report
AQA 17-08
PFAS in Soil, Water and Fish

November 2017



Analysis of Per/Polyfluoroalkyl Substances in Water Using an Agilent 6470 Triple Quadrupole LC/MS

Application Note

Authors

Kathy Hunt and Ralph Hindle
Vogon Laboratory Services Ltd.,
Cochrane, AB Canada

Tarun Anumol
Agilent Technologies Inc.
Wilmington, DE, USA

Introduction

The analysis of per/polyfluoroalkyl substanc compounds (PFCs), in particular the perfluori a hot topic in water analysis. The unique che make them components used in a variety of cookware, fire resistant clothing, fire-fighting compounds are considered toxic, persistent, and the environment. Consequently, the Unit Agency (USEPA) has recently issued drinking PFASs, perfluorooctanoic acid (PFOA) and pe (combined). Several states such as New Jer already have public health guideline values v PFAS including PFOA, PFOS, perfluorohexan acid (PFNA) in water.

USEPA Method 537 highlights a method for t water with solid phase extraction (SPE) and other classes of PFASs are also currently in environment.

This application note describes the analysis including all 14 PFASs in EPA Method 537, u Agilent 6470 triple quadrupole LC/MS/MS.

Chromatography

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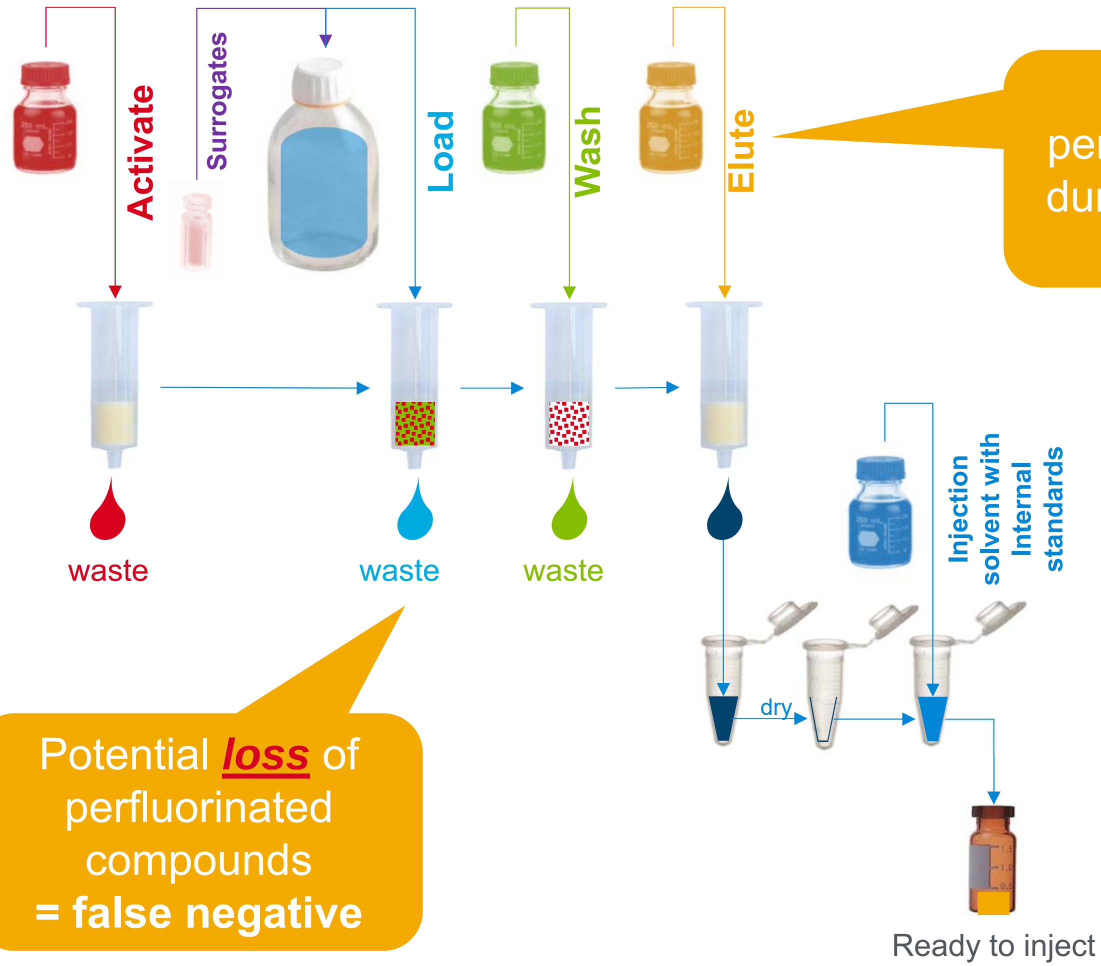
Offline SPE of environmental water samples



Potential ***gain*** of perfluorinated compounds during sample preparation
= false positive

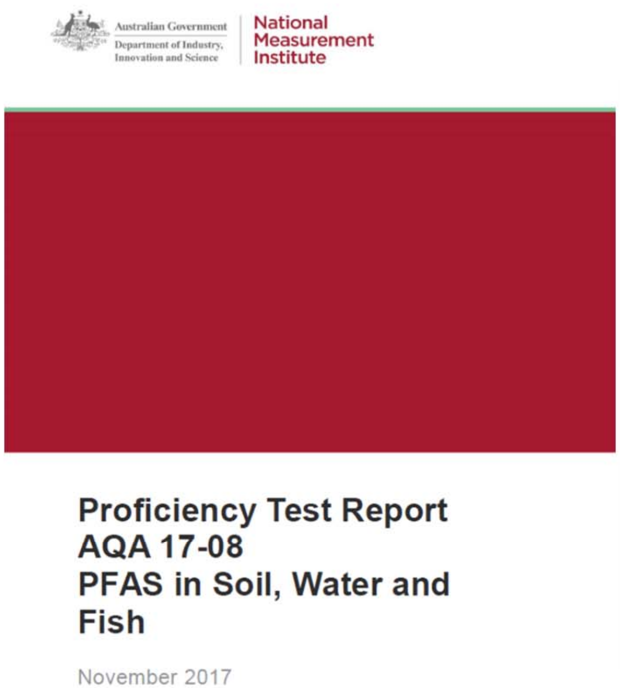
Potential ***loss*** of perfluorinated compounds
= false negative

Pain points

- Inter-operator **consistency**
- Risk of **contamination** between samples
- Preparation **Time**
- Cost of consumables
- **Waste** of expensive surrogates

AQA 17-08 NMI Proficiency test results

Results for PFOA and PFOS in spiked and incurred water samples

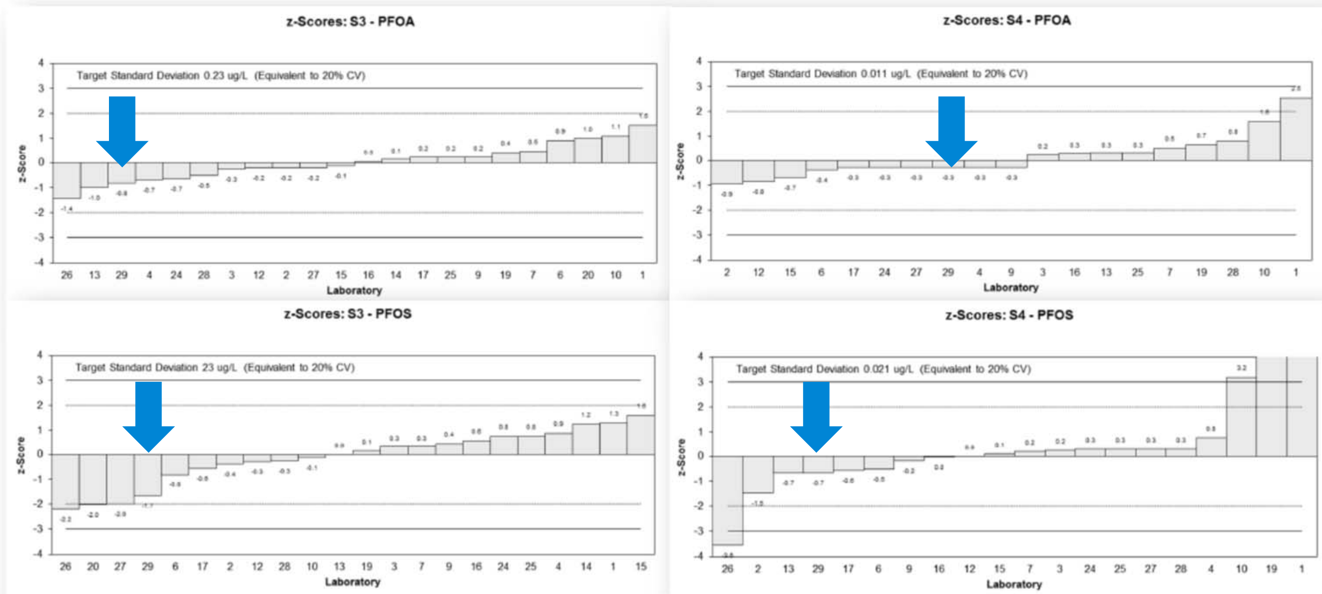

Australian Government
Department of Industry, Innovation and Science
National Measurement Institute

Proficiency Test Report
AQA 17-08
PFAS in Soil, Water and Fish

November 2017

Spiked water

Incurred water

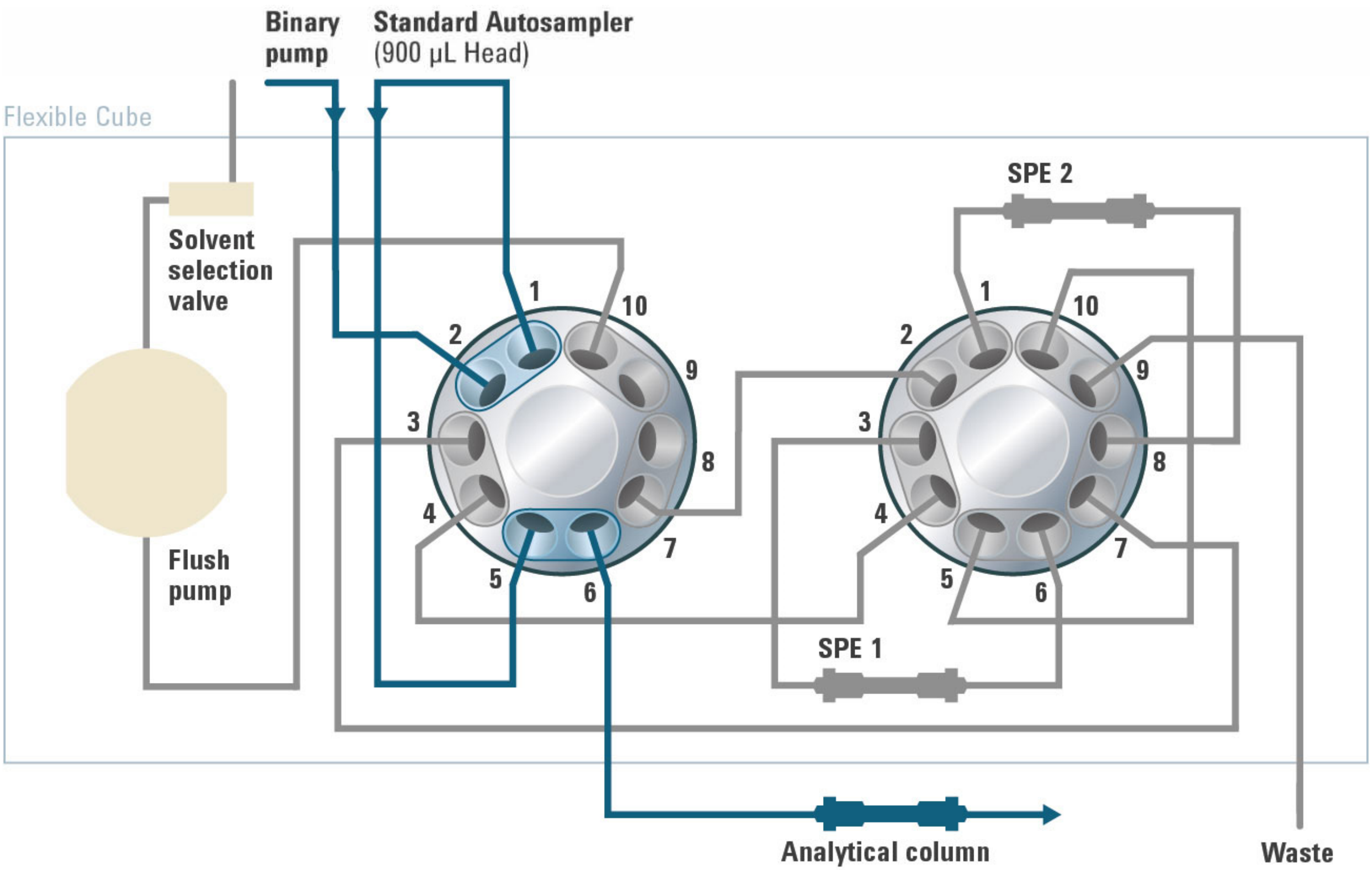


Results from offline SPE and the analytical method are good, but there must be a better way!

Online SPE with the 1290 Infinity Flexible Cube

see video at http://www.agilent.com/chem/infinity-online-spe

Online SPE with the 1290 Infinity Flexible Cube



Switch between:

1. direct inject mode (for standard LC methods and Online SPE recovery tests)
2. online SPE mode

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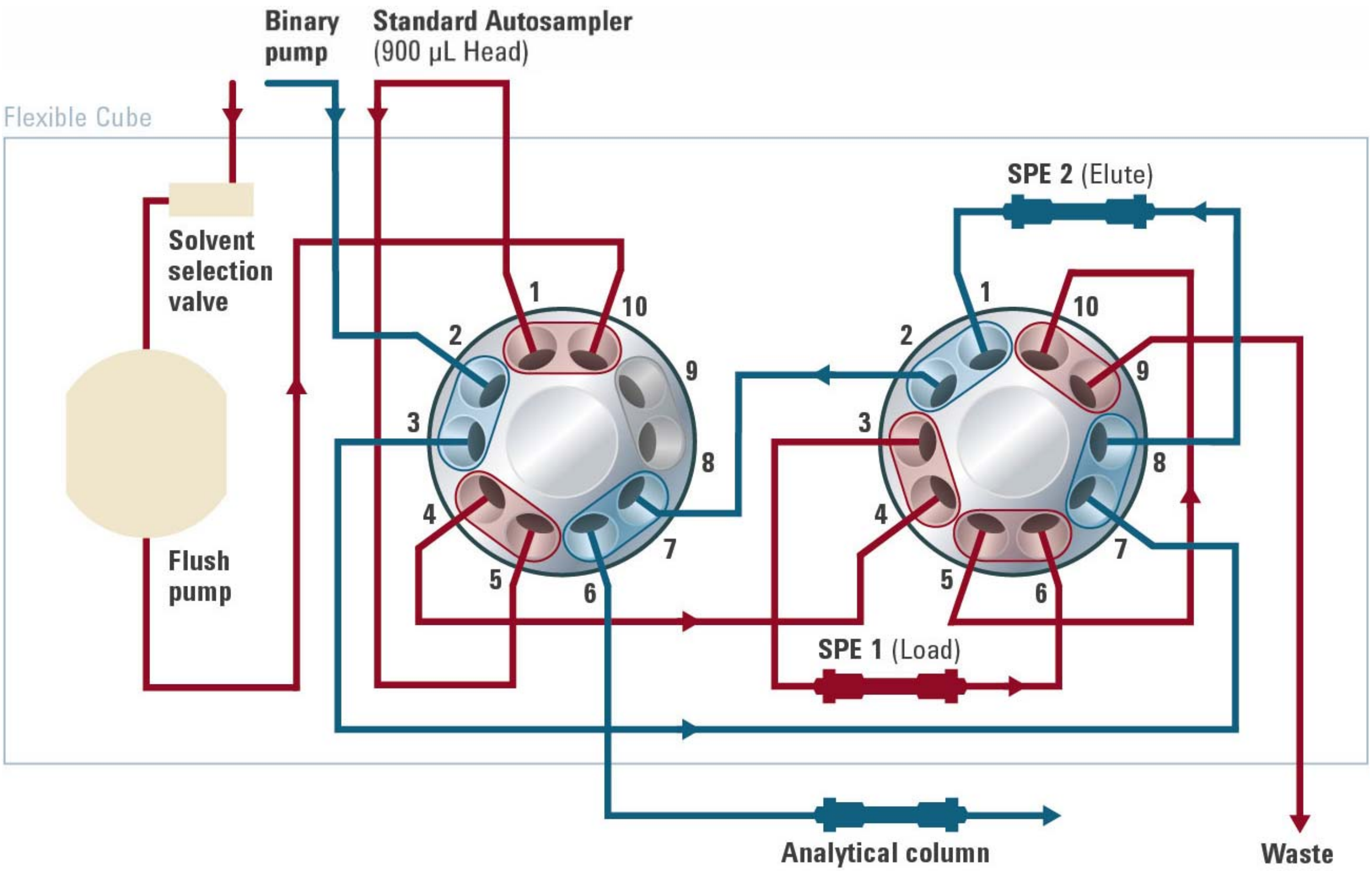
Online SPE with the 1290 Infinity Flexible Cube



In Online SPE mode, the right valve **alternates** between SPE cartridges to allow one cartridge to be prepared whilst the other is eluting.

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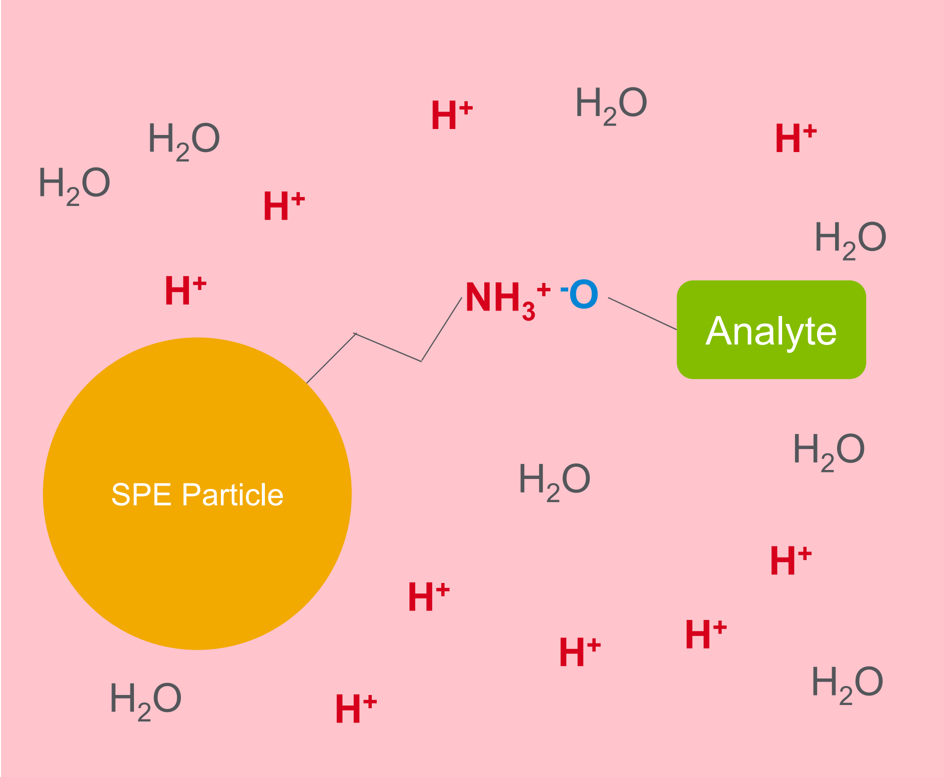
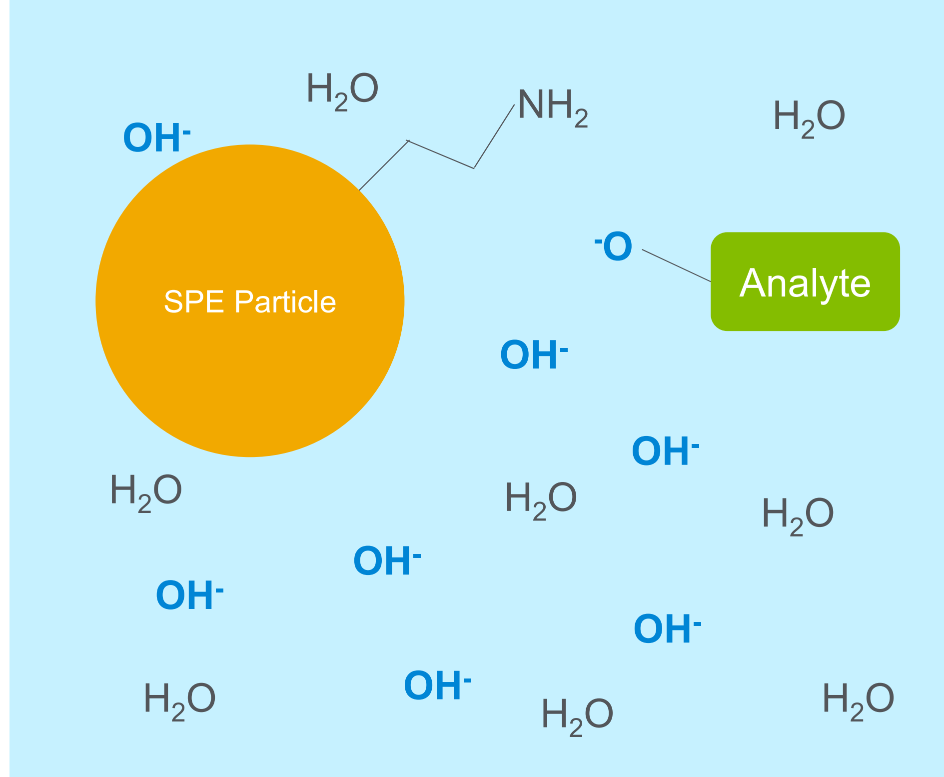
Online SPE

The chemistry we're exploiting: Weak Anion Exchange (WAX)

High acid environment (Low pH), retain analyte



Low acid environment (High pH), elute analyte



Online SPE

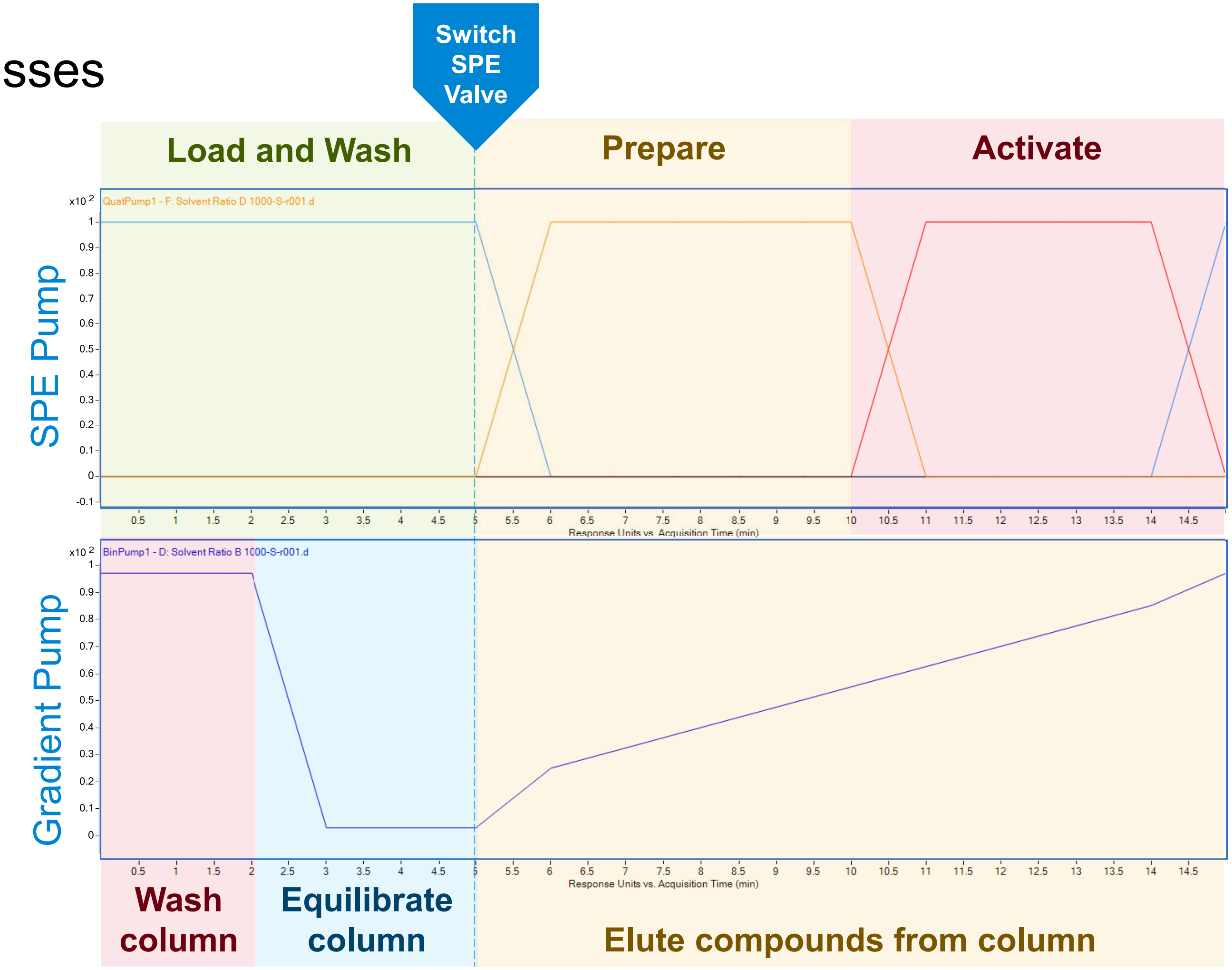
Programming parallel processes

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:

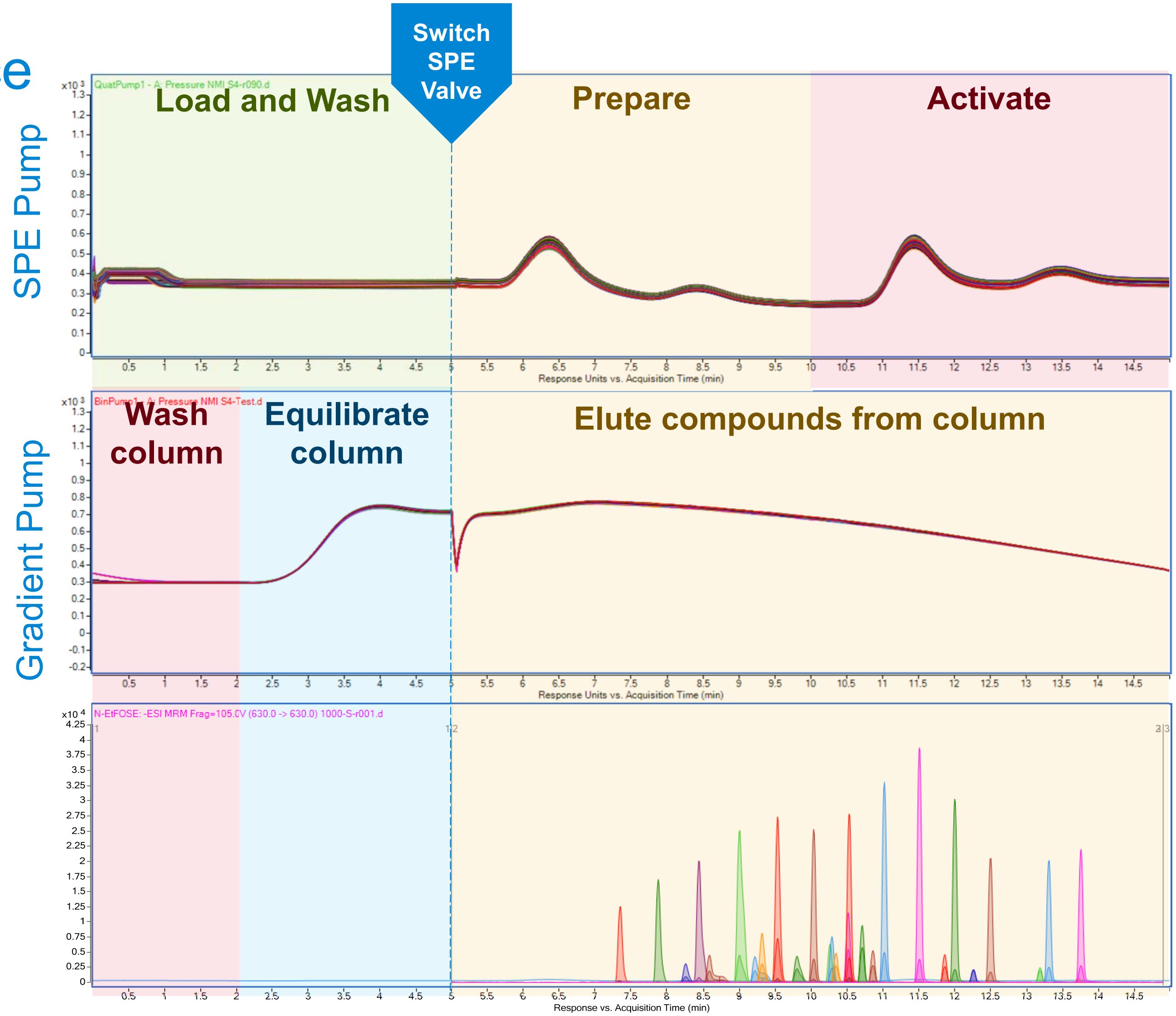
- The SPE pump prepares the sample loop and SPE sorbent, then activates the SPE, then loads the sample into the sample loop.
- The Gradient pump Elutes the compounds from the column.



Online SPE Performance

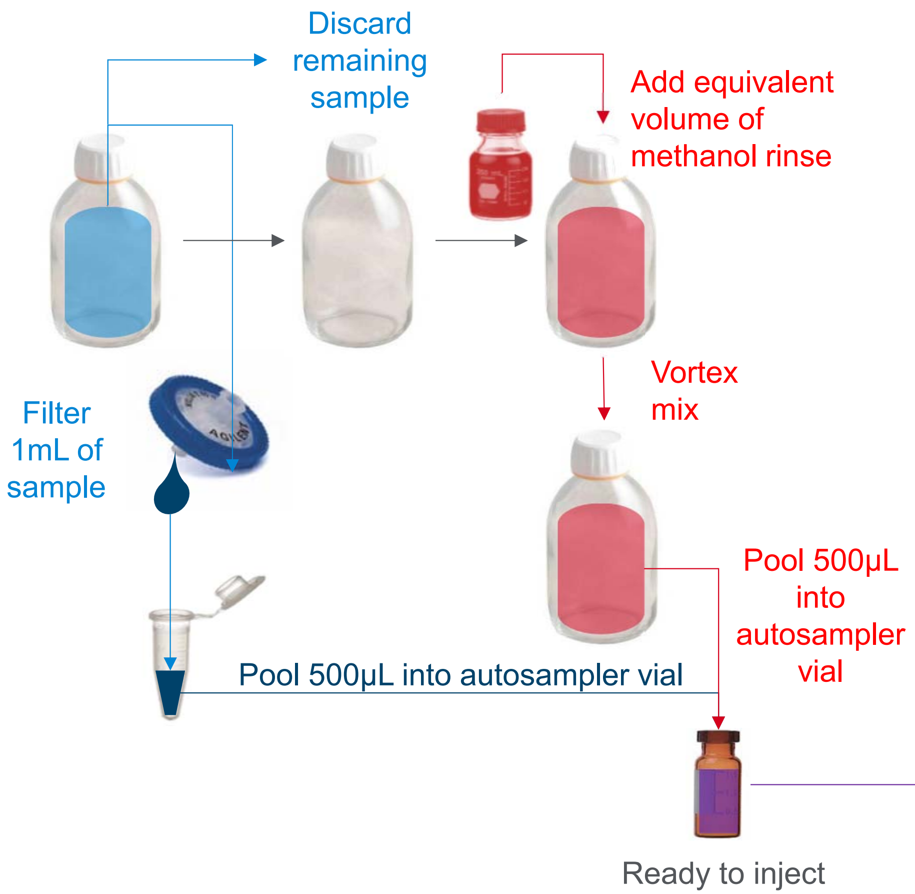
LC Performance

- Good pressure reproducibility on both the Binary and the Quaternary Pumps
- Excellent retention time reproducibility
- Great peak shape, including separation of branched isomers

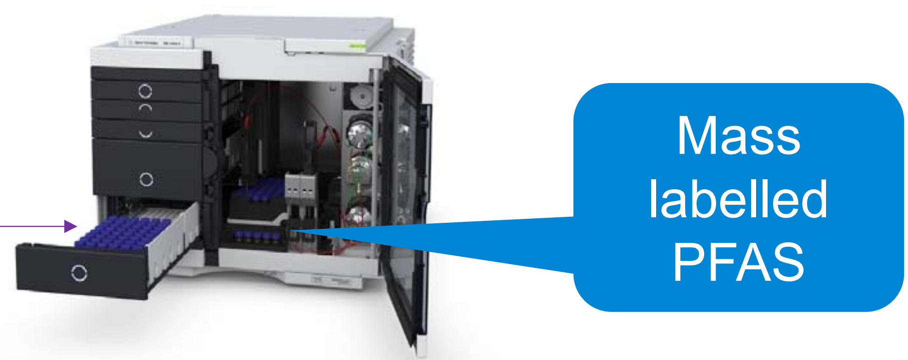


Online SPE

Simplified sample preparation for of environmental water samples



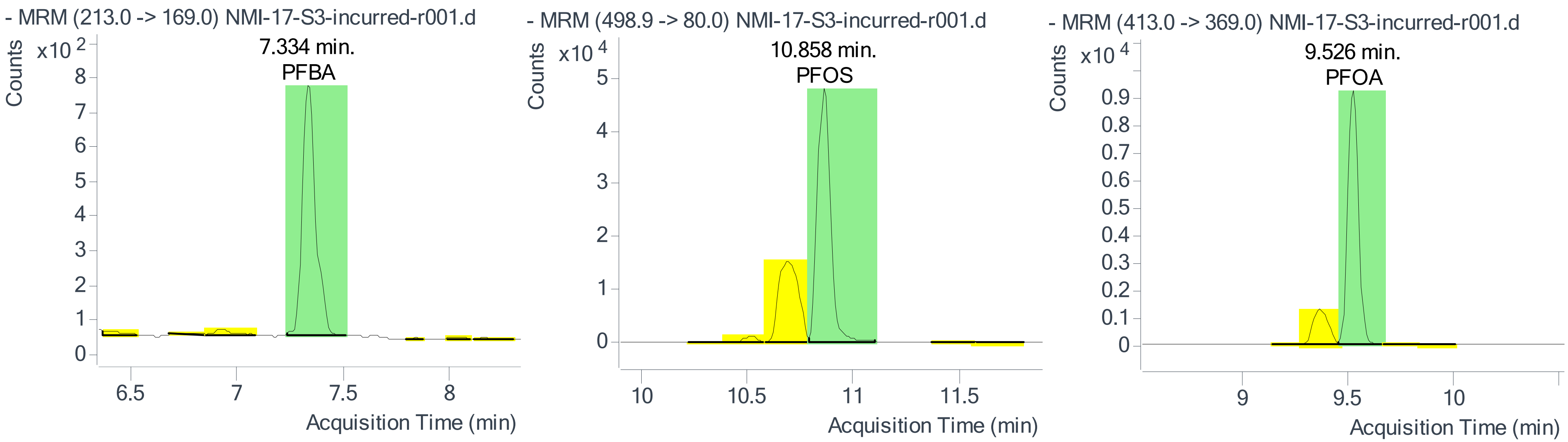
- Consumables manufactured with PTFE free materials used at all steps.
- Perfluorinated compounds can remain adhered to some sample vessels requiring methanol to rinse them off.
- Resulting dilution factor is 2
- Multisampler co-inject’s the mass-labelled PFAS with the samples



Online SPE Performance

Chromatographic resolution

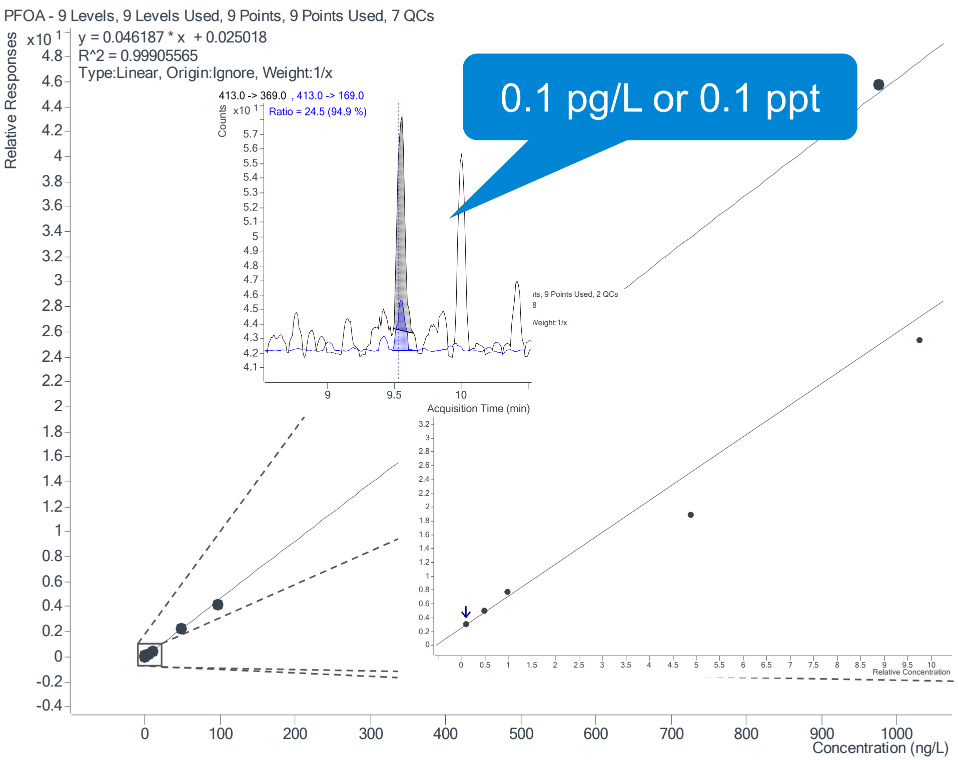
- Example EICs from NMI 17 S3 (incurred sample)
- Separation of branched isomers in incurred samples



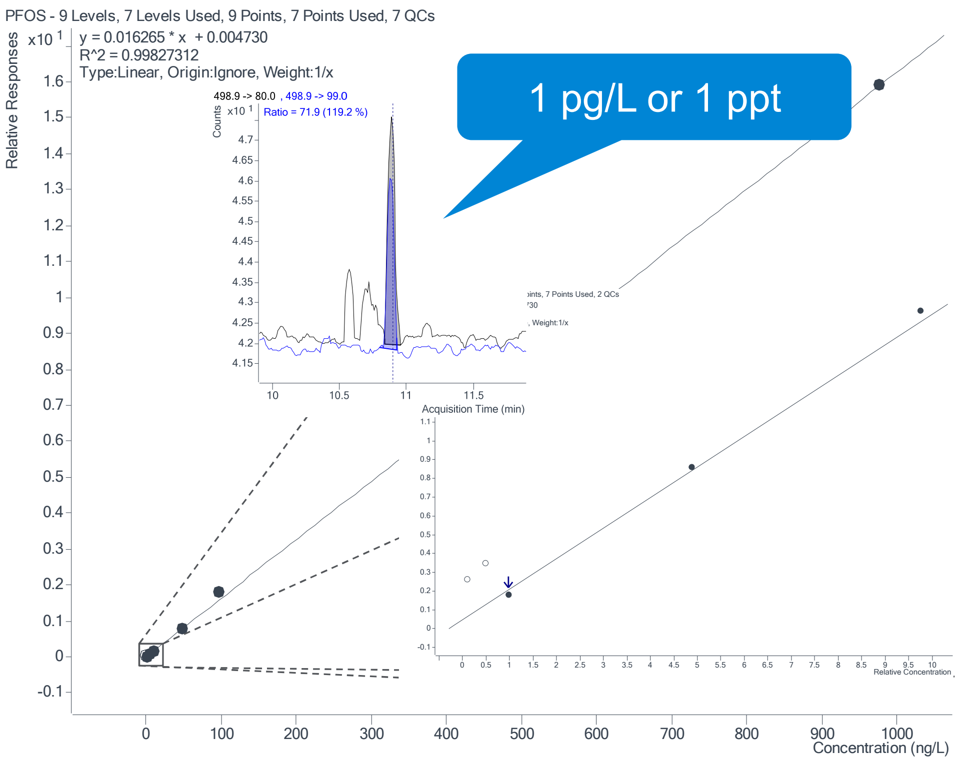
Online SPE Performance

Linearity, dynamic range and sensitivity on a 6470 system

PFOA



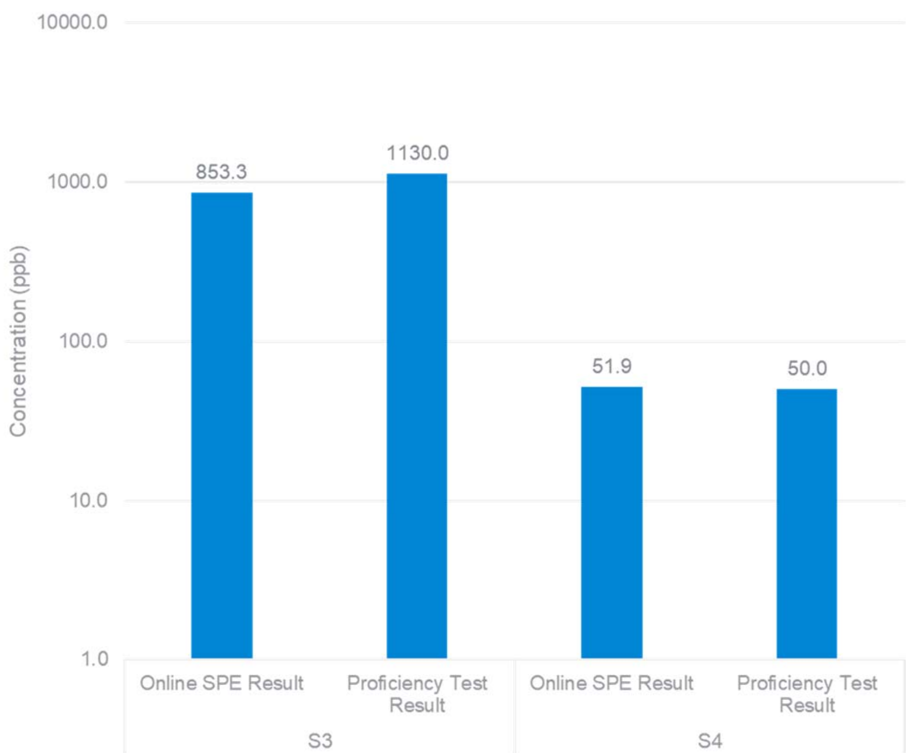
PFOS



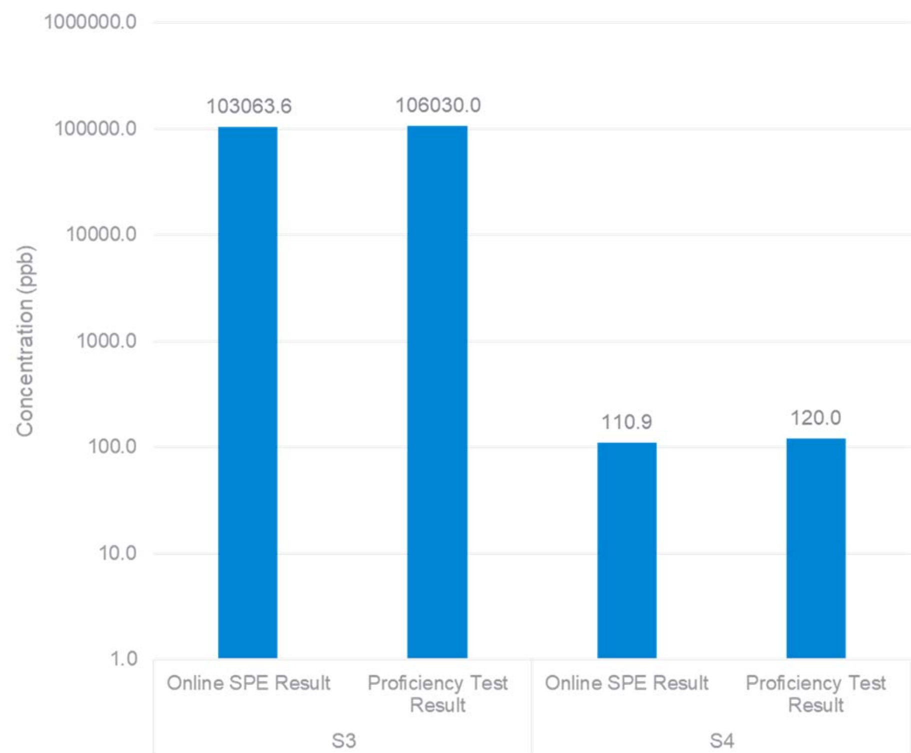
Online SPE comparison to Proficiency test NMI-17 results

Comparison of the final calculated comparison

PFOA comparison



PFOS comparison



Online SPE Performance

Robustness worklist

Sample Name	Sample Position	Method	Data File	Sample Type	Level
DB-S	No Injection	180730 PFCs OnlineSPE MRM.m	DB-S-r001.d	DoubleBlank	
DB-S	No Injection	180730 PFCs OnlineSPE MRM.m	DB-S-r002.d	DoubleBlank	
B-S	P1-F9	180730 PFCs OnlineSPE MRM.m	B-S-r001.d	Blank	
B-S	P1-F9	180730 PFCs OnlineSPE MRM.m	B-S-r002.d	Blank	
01-S	P1-A1	180730 PFCs OnlineSPE MRM.m	01-S-r001.d	Calibration	0.1
05-S	P1-A2	180730 PFCs OnlineSPE MRM.m	05-S-r001.d	Calibration	0.5
1-S	P1-A3	180730 PFCs OnlineSPE MRM.m	1-S-r001.d	Calibration	1
5-S	P1-A4	180730 PFCs OnlineSPE MRM.m	5-S-r001.d	Calibration	5
10-S	P1-A5	180730 PFCs OnlineSPE MRM.m	10-S-r001.d	Calibration	10
50-S	P1-A6	180730 PFCs OnlineSPE MRM.m	50-S-r001.d	Calibration	50
100-S	P1-A7	180730 PFCs OnlineSPE MRM.m	100-S-r001.d	Calibration	100
500-S	P1-A8	180730 PFCs OnlineSPE MRM.m	500-S-r001.d	Calibration	500
1000-S	P1-A9	180730 PFCs OnlineSPE MRM.m	1000-S-r001.d	Calibration	1000
NMI-18-S3	multiple	180730 PFCs OnlineSPE MRM.m	NMI-18-S3-r001.d	Sample	NMI-S3
NMI-18-S4	multiple	180730 PFCs OnlineSPE MRM.m	NMI-18-S4-r001.d	Sample	NMI-S4

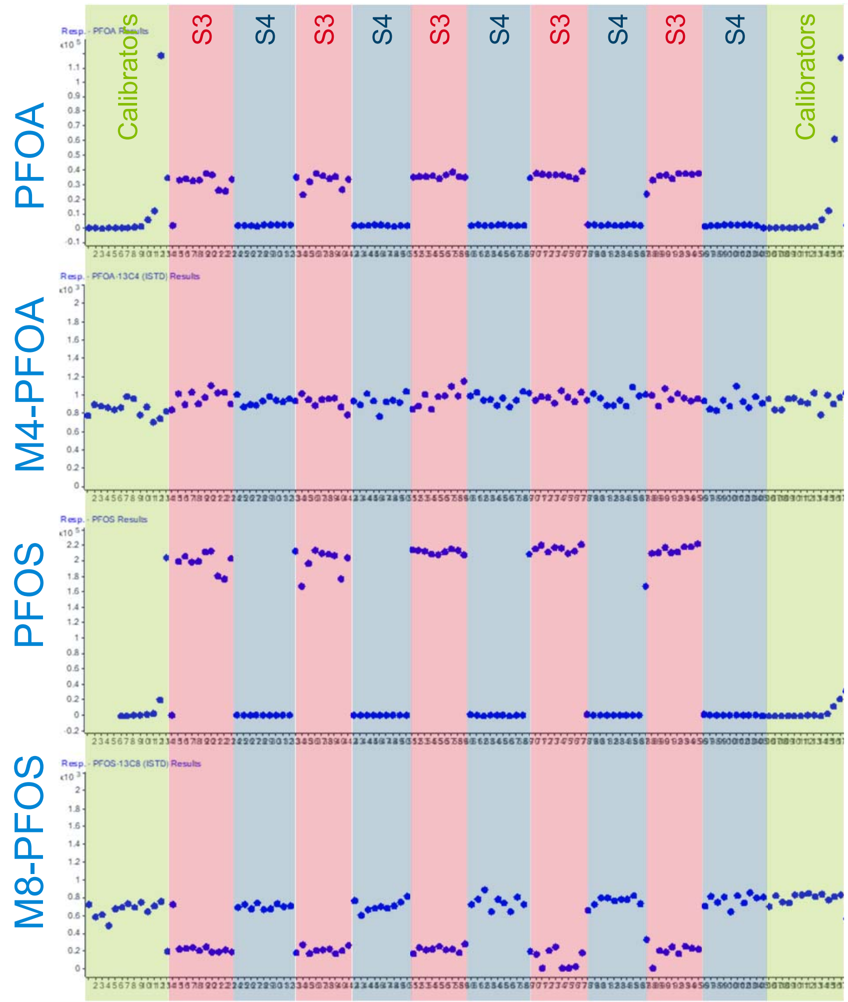
Replicated 45x each

Online SPE performance

Robustness results

PFOA and PFOS and their mass labeled internal standards showed reproducibility over replicate injections.

Injection Volume = 475 µL



Notes:

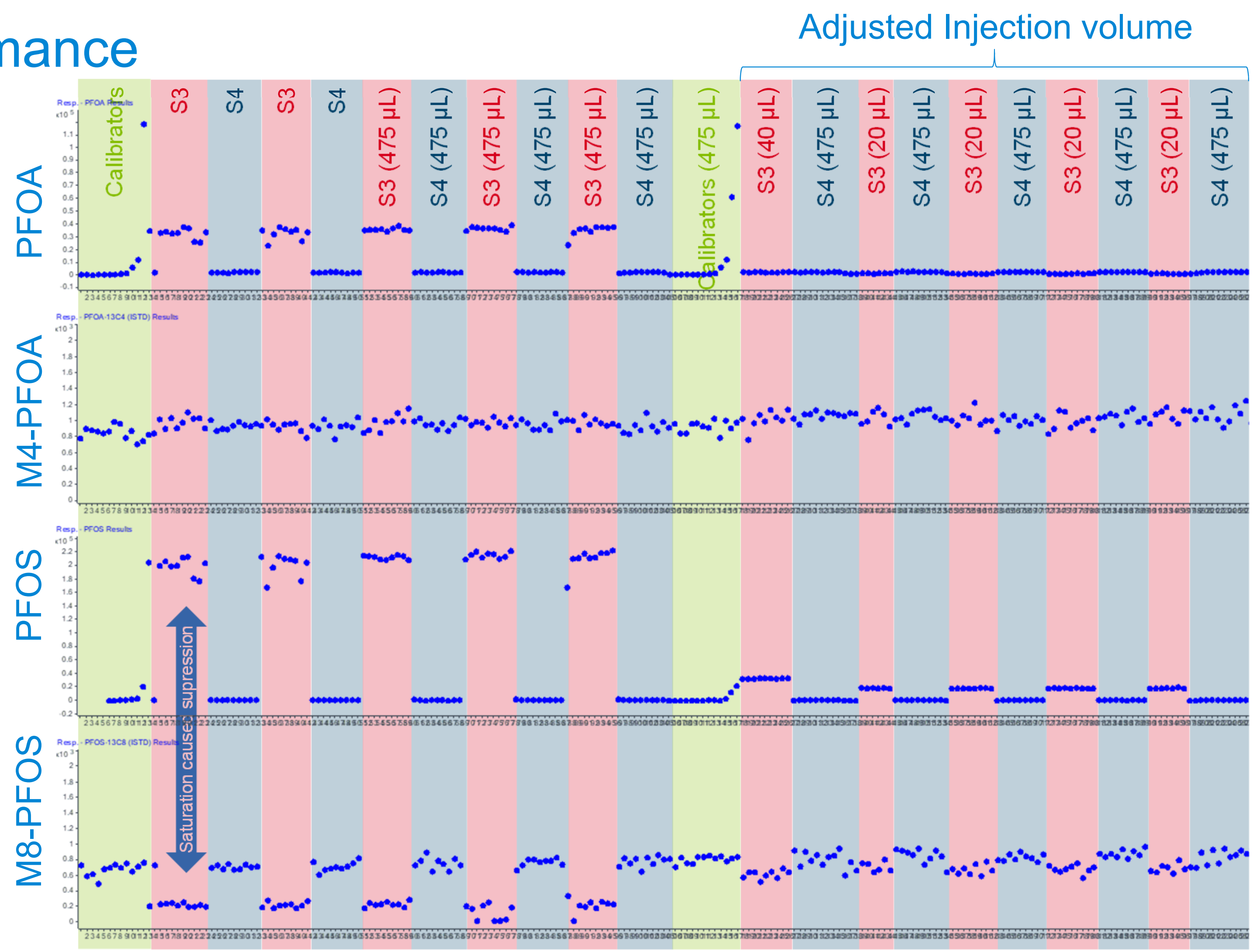
- PFOA Calibration range
- M4-PFOA response is consistent in cals and smps
- Saturated peak, S3 sample needs to be diluted
- PFOS Calibration range
- Suppression of M8-PFOS caused by saturated PFOS peak

Online SPE performance

Robustness

Continue worklist with adjusted injection volume for S3 to effectively 'dilute' the sample to quantitate PFOS within the calibration range

180 sample injections and calibrators



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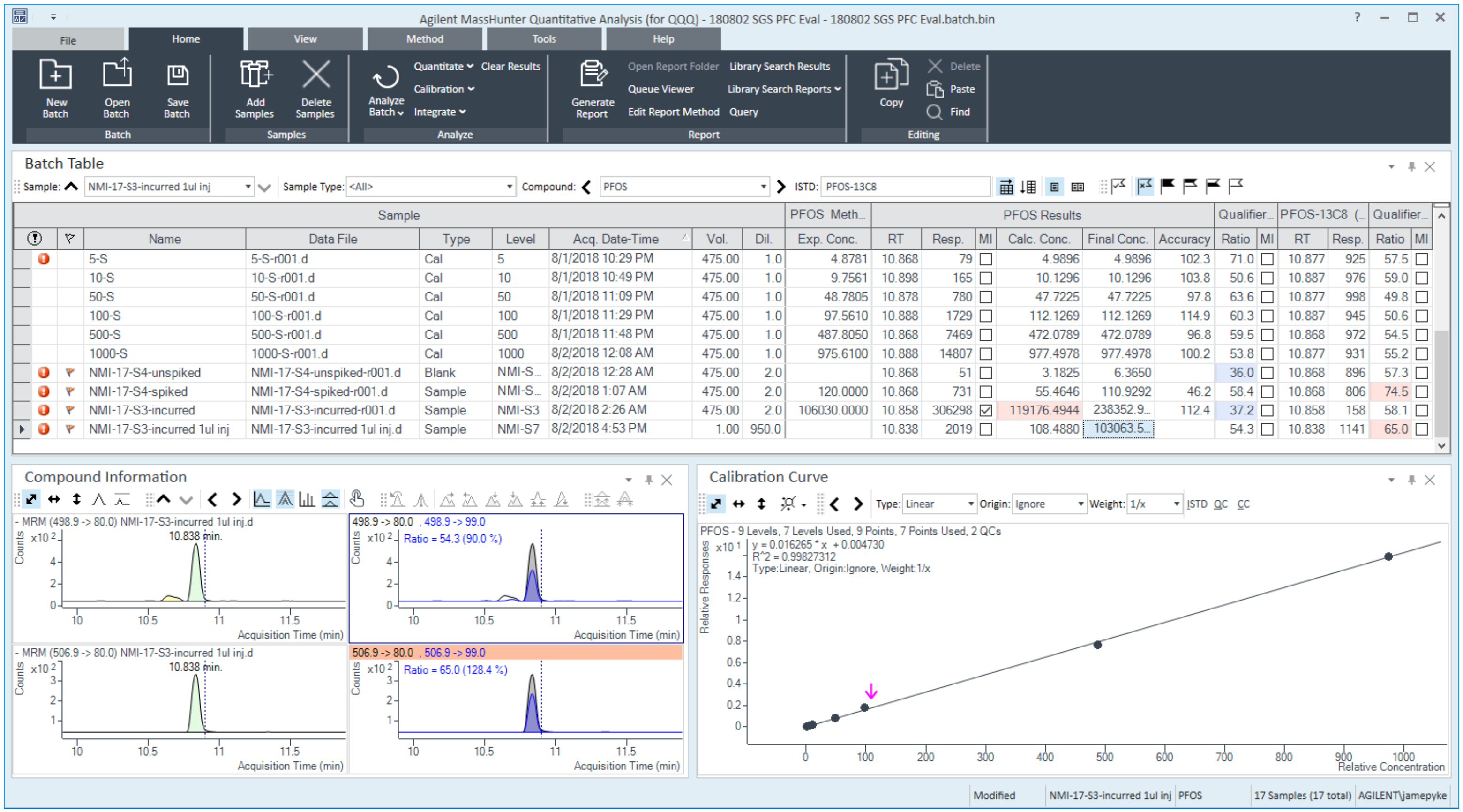
Online SPE comparison to NMI-17 proficiency test results

Calculated Final Concentrations (ng/L or ppt)



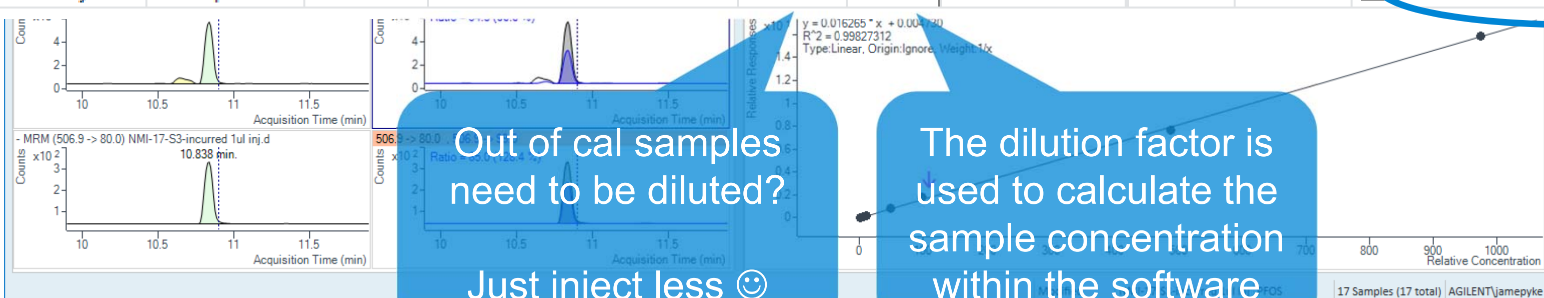
Name	Data File	Type	Level	Acq. Date-Time	Vol.	Dil.	Exp. Conc.	RT	Resp.	MI	Calc. Conc.	Final Conc.	Accuracy	Qualifier Ratio	MI	PFOS-13C8 RT	PFOS-13C8 Resp.	Qualifier Ratio	MI
5-S	5-S-r001.d	Cal	5	8/1/2018 10:29 PM	475.00	1.0	4.8781	10.868	79	☐	4.9896	4.9896	102.3	71.0	☐	10.877	925	57.5	☐
10-S	10-S-r001.d	Cal	10	8/1/2018 10:49 PM	475.00	1.0	9.7561	10.898	165	☐	10.1296	10.1296	103.8	50.6	☐	10.887	976	59.0	☐
50-S	50-S-r001.d	Cal	50	8/1/2018 11:09 PM	475.00	1.0	48.7805	10.878	780	☐	47.7225	47.7225	97.8	63.6	☐	10.877	998	49.8	☐
100-S	100-S-r001.d	Cal	100	8/1/2018 11:29 PM	475.00	1.0	97.5610	10.888	1729	☐	112.1269	112.1269	114.9	60.3	☐	10.887	945	50.6	☐
500-S	500-S-r001.d	Cal	500	8/1/2018 11:48 PM	475.00	1.0	487.8050	10.868	7469	☐	472.0789	472.0789	96.8	59.5	☐	10.868	972	54.5	☐
1000-S	1000-S-r001.d	Cal	1000	8/2/2018 12:08 AM	475.00	1.0	975.6100	10.888	14807	☐	977.4978	977.4978	100.2	53.8	☐	10.877	931	55.2	☐
NMI-17-S4-unspiked	NMI-17-S4-unspiked-r001.d	Blank	NMI-S...	8/2/2018 12:28 AM	475.00	2.0		10.868	51	☐	3.1825	6.3650		36.0	☐	10.868	896	57.3	☐
NMI-17-S4-spiked	NMI-17-S4-spiked-r001.d	Sample	NMI-S...	8/2/2018 1:07 AM	475.00	2.0	120.0000	10.868	731	☐	55.4646	110.9292	46.2	58.4	☐	10.868	806	74.5	☐
NMI-17-S3-incurred	NMI-17-S3-incurred-r001.d	Sample	NMI-S3	8/2/2018 2:26 AM	475.00	2.0	106030.0000	10.858	306298	☑	119176.4944	238352.9...	112.4	37.2	☐	10.858	158	58.1	☐
NMI-17-S3-incurred 1ul inj	NMI-17-S3-incurred 1ul inj.d	Sample	NMI-S7	8/2/2018 4:53 PM	1.00	950.0		10.838	2019	☐	108.4880	103063.5...		54.3	☐	10.838	1141	65.0	☐

Online SPE comparison to NMI-17 proficiency test results

Calculated Final Concentrations (ng/L or ppt)

Agilent MassHunter Quantitative Analysis (for QQQ) - 180802 SGS PFC Eval - 180802 SGS PFC Eval.batch.bin

Sample						PFOS Meth...	PFOS Results					
Data File	Type	Level	Acq. Date-Time	Vol.	Dil.	Exp. Conc.	RT	Resp.	MI	Calc. Conc.	Final Conc.	Acc
5-S-r001.d	Cal	5	8/1/2018 10:29 PM	475.00	1.0	4.8781	10.868	79	☐	4.9896	4.9896	
10-S-r001.d	Cal	10	8/1/2018 10:49 PM	475.00	1.0	9.7561	10.898	165	☐	10.1296	10.1296	
50-S-r001.d	Cal	50	8/1/2018 11:09 PM	475.00	1.0	48.7805	10.878	780	☐	47.7225	47.7225	
100-S-r001.d	Cal	100	8/1/2018 11:29 PM	475.00	1.0	97.5610	10.888	1729	☐	112.1269	112.1269	
500-S-r001.d	Cal	500	8/1/2018 11:48 PM	475.00	1.0	487.8050	10.868	7469	☐	472.0789	472.0789	
1000-S-r001.d	Cal	1000	8/2/2018 12:08 AM	475.00	1.0	975.6100	10.888	14807	☐	977.4978	977.4978	
NMI-17-S4-unspiked-r001.d	Blank	NMI-S...	8/2/2018 12:28 AM	475.00	2.0		10.868	51	☐	3.1825	6.3650	
NMI-17-S4-spiked-r001.d	Sample	NMI-S...	8/2/2018 1:07 AM	475.00	2.0	120.0000	10.868	731	☐	55.4646	110.9292	
NMI-17-S3-incurred-r001.d	Sample	NMI-S3	8/2/2018 2:26 AM	475.00	2.0	106030.0000	10.858	306298	☑	119176.4944	238352.9...	
NMI-17-S3-incurred 1ul inj.d	Sample	NMI-S7	8/2/2018 4:53 PM	1.00	950.0		10.838	2019	☐	108.4880	103063.5...	



Out of cal samples need to be diluted?

Just inject less ☺

The dilution factor is used to calculate the sample concentration within the software

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Conclusions

There are a range of options analyzing perfluorinated compounds, the online SPE option:

- is **fast and effective** at 4 samples/hour include SPE steps and data acquisition
- is **efficient** due to less consumable requirements and less wastage of the mass labeled internal standards and surrogates
- is able to **mitigates risk** by:
  - by reducing potential sample contact with PTFE materials in the lab
  - removing background perfluorinated compounds from the LC flow path and LC solvents as they are used
  - preventing cross contamination by reducing sample preparation steps

Leading to an increase **ROI** as less resources are required. The LC configuration is not dedicated to Online SPE, so is **flexible** to accommodate multiple acquisition methods.

Thank you, for your time.

Any questions?