Automated Solutions for PFAS in Water

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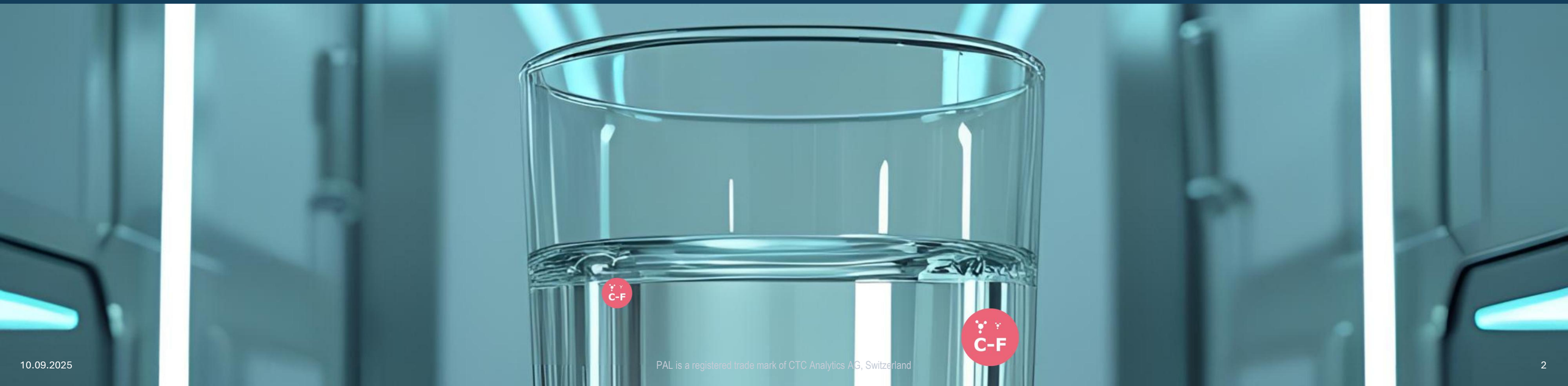
PAL SYSTEM
Ingenious sample handling

Agenda

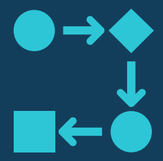
- Introduction – Regulations for PFAS in Water
- Overview – Sample Prep Techniques and Challenges
- Examples for Automated Sample Prep Workflows:
  - Online SPE
  - EPA1633 with Micro-SPE
  - Volatile PFAS with SPME
- Summary and Outlook



Regulations for PFAS in Water

USA and EU

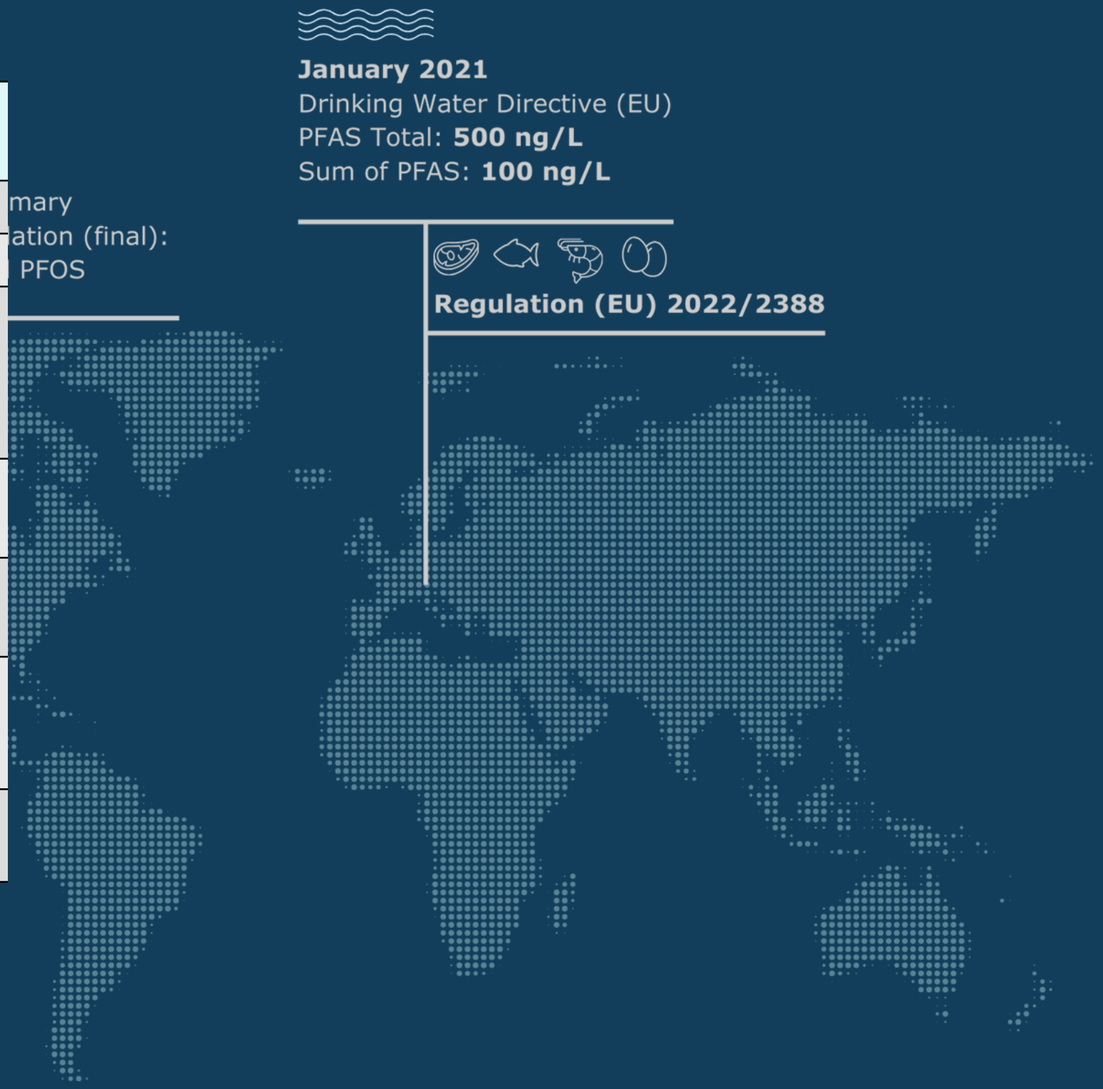
Method	Matrix Tested	No. of Analytes	Sample Prep Procedure
EPA 537.1	Drinking water	18	SPE
EPA 533	Drinking water	29	SPE
EPA 1633	wastewater, surface water, groundwater, soil, biosolids, sediment, landfill leachate, and fish tissue	40	SPE
EPA 8327	Surface water, ground water, wastewater influent and effluent	24	Direct injection
ASTM D7979	Surface water, ground water, wastewater influent and effluent	21	Direct injection
EN17892	Drinking water	20	SPE / direct injection
ISO/DIS 21675 (draft)	Drinking water, sea water, fresh water, wastewater	30-40	SPE



Regulations are still evolving

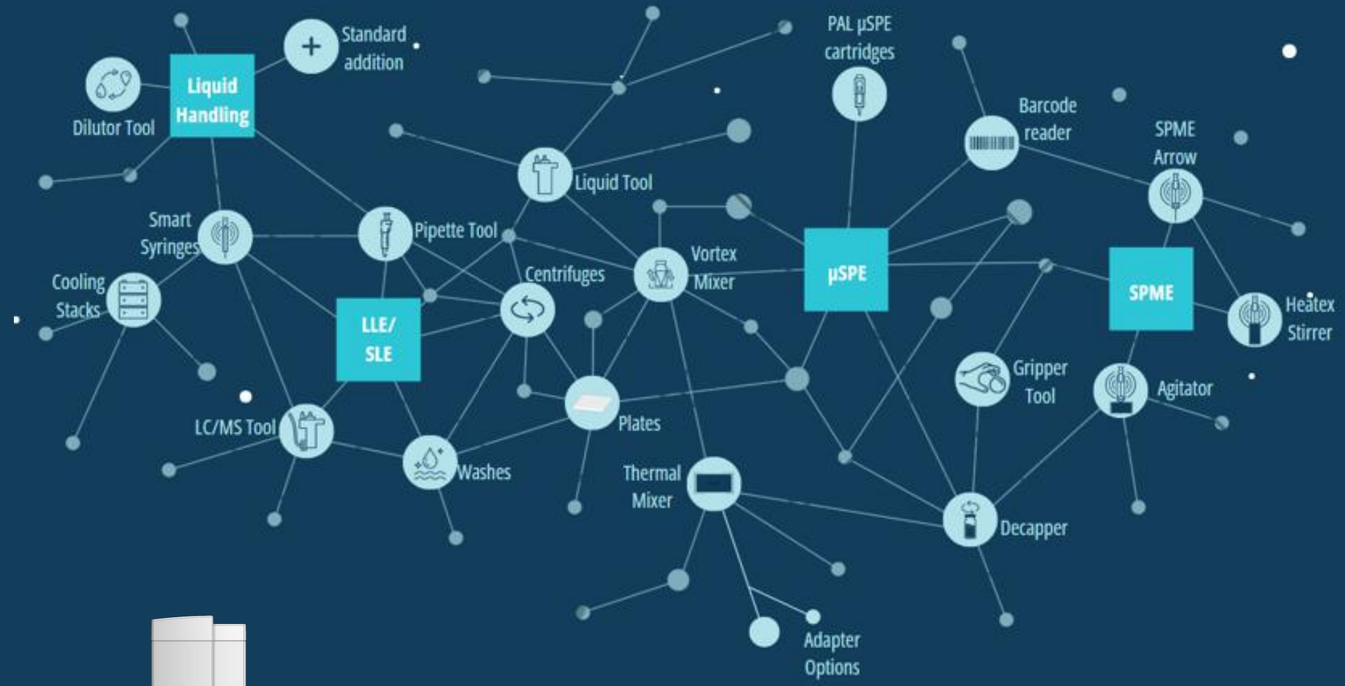
Number of analytes ↑

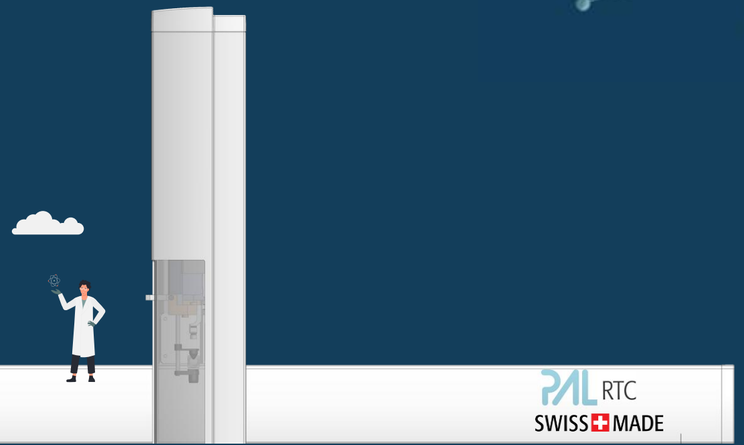
Concentration limits ↓



Key Sample Prep Techniques for PFAS in Water

SPE / Micro-SPE I Liquid-Liquid Extractions I SPME





Solid Phase Extraction (SPE)

Selectively retaining PFAS on a solid sorbent material (in a cartridge, column or well plate) while matrix interferences are washed away, followed by elution of PFAS.

- Manual SPE
- Online SPE
- Micro-SPE (µSPE)

Dispersive Liquid-Liquid Micro Extraction (DLLME)

Miniaturized version of liquid-liquid extraction, dispersion of organic extracts into droplets, followed by rapid extraction into organic phase → Dispersive Liquid-Liquid Micro Extraction (automated).

SPME (Solid Phase Micro Extraction)

Adsorption of analytes onto a coated Fiber or Arrow device, typically from sample headspace (for volatiles) or direct immersion.

Major Challenges in Sample Preparation

Why Sample Prep is Critical for Reliable PFAS Results

Major **Challenges**

Ultra-low Concentration
Demands high concentrating factor, clean background and high sensitivity.

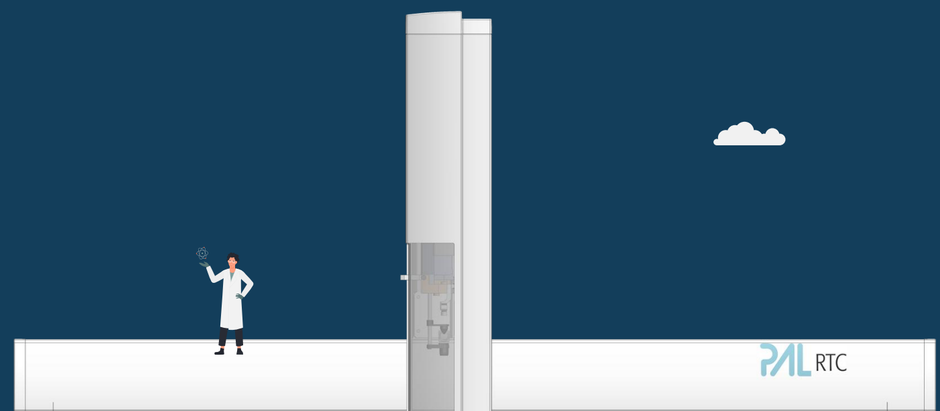
Contamination Risk
Requires thourough handling

Manual inconsistency
Operator variability undermines reproducibility.

60-70%

of **lab errors** happen in the **pre-analytical** phase, i.e., sample handling phase, introducing variability.

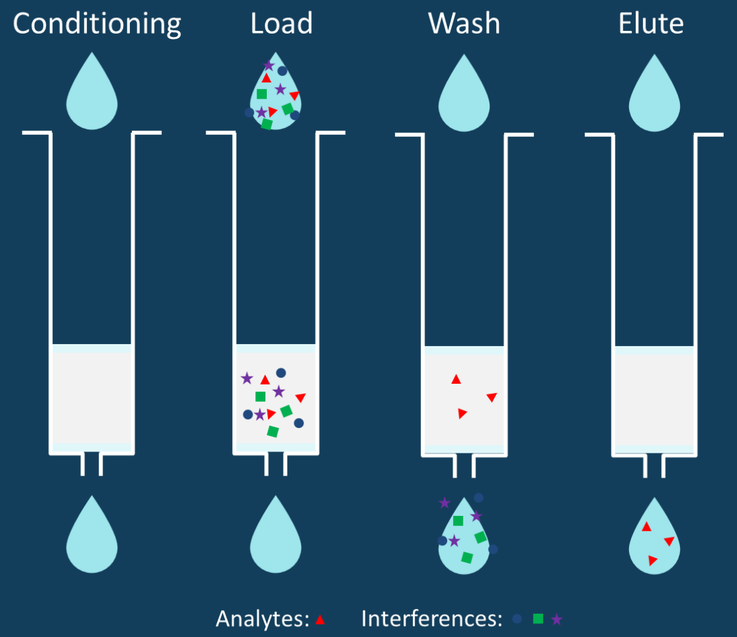
Garbage in –
Garbage out.

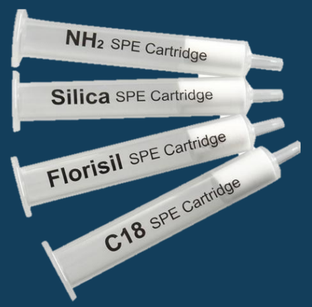


Online SPE for PFAS in Water

What is Online SPE?

Offline SPE

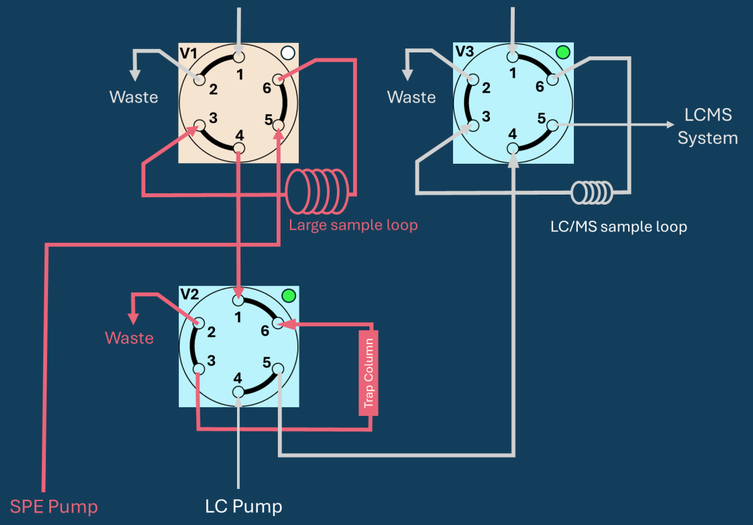




Offline SPE	Online SPE
Disposable SPE cartridges with different sorbents	SPE columns with typically 1-4.6mm id, 10-50mm length, with different packing materials
Manual multi-step sample handling	Automated sample handling
High consumption of sample and solvents	Low consumption of sample and solvents
Low sample throughput	Very high sample throughput

Online SPE

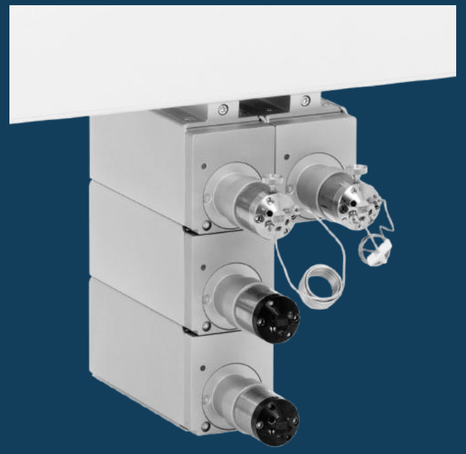
Use automated valves switching to load, wash and elute samples



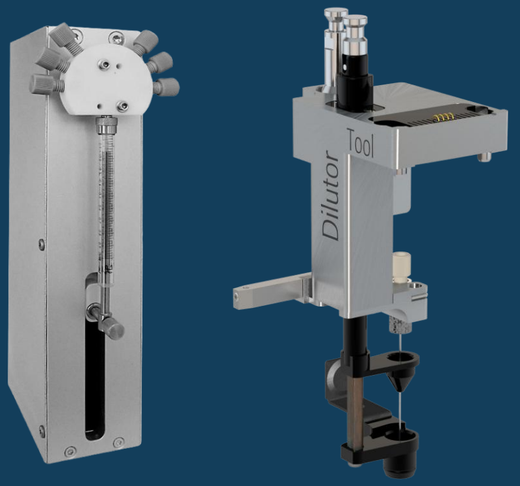
Online SPE for PFAS in Water

Hardware Setup

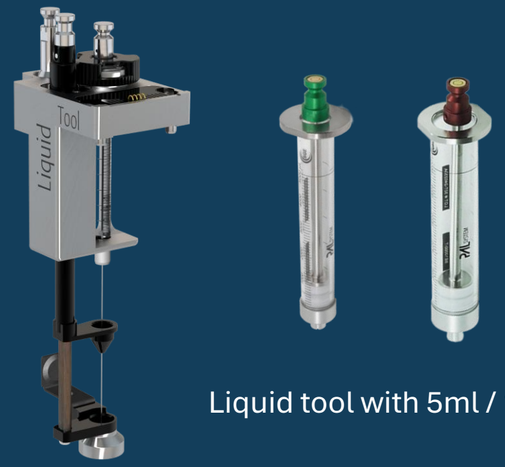
Multiple Valves + Dilutor or Liquid Tool with large Syringe (for large volume injection)



6-port & 10-port valves available, up to 9 valves on a PAL system



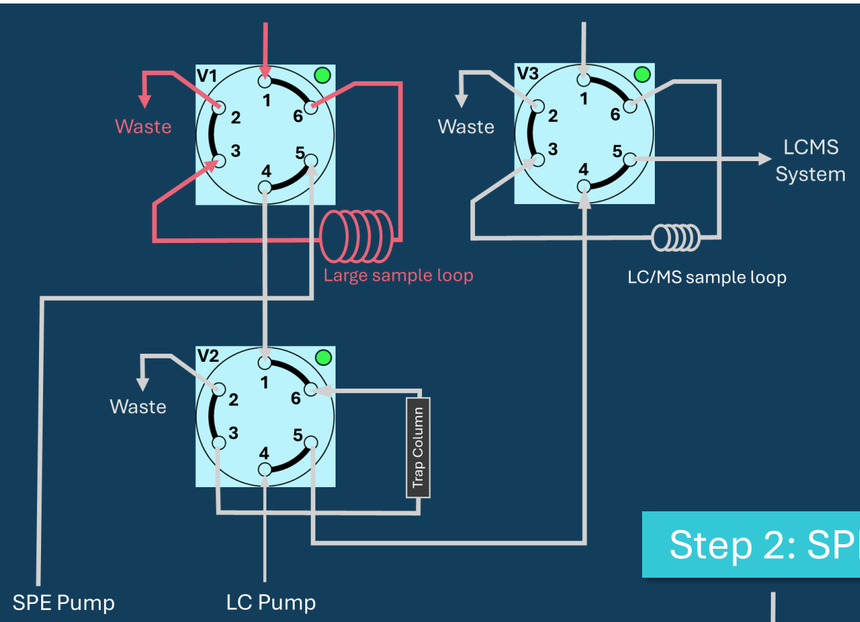
Dilutor tool with dilutor module (can connect to up to 5 solvent lines)



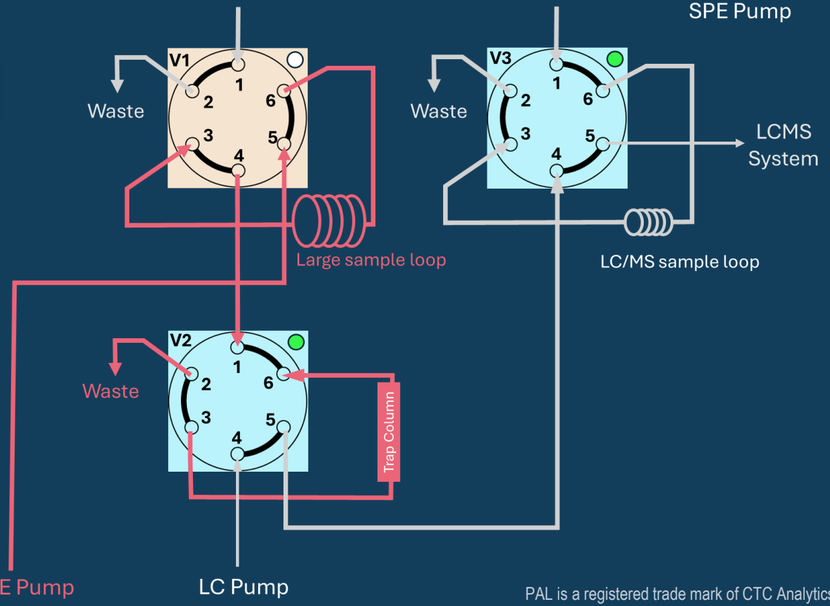
Liquid tool with 5ml / 10ml syringe

Online SPE – How it works

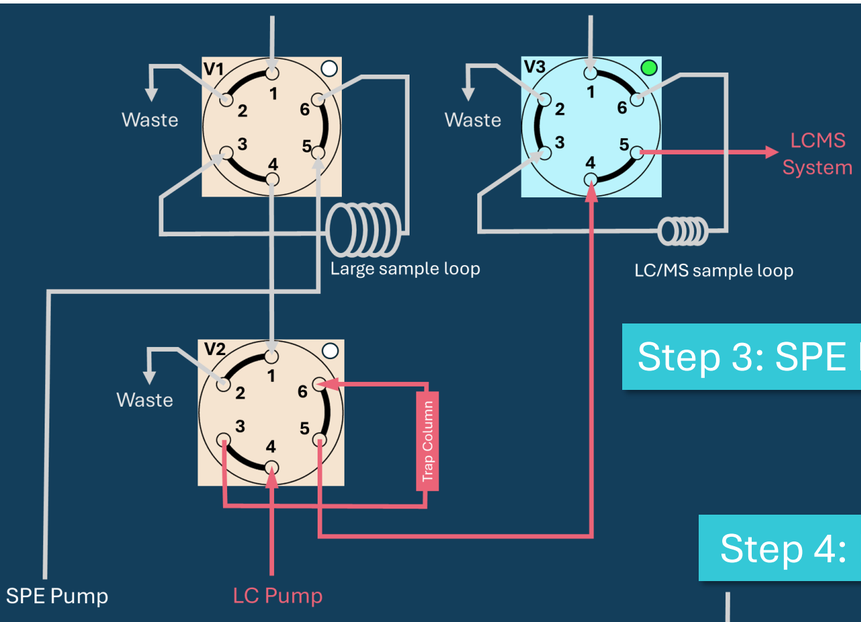
Automated Conditioning, Load, Wash, and Elute



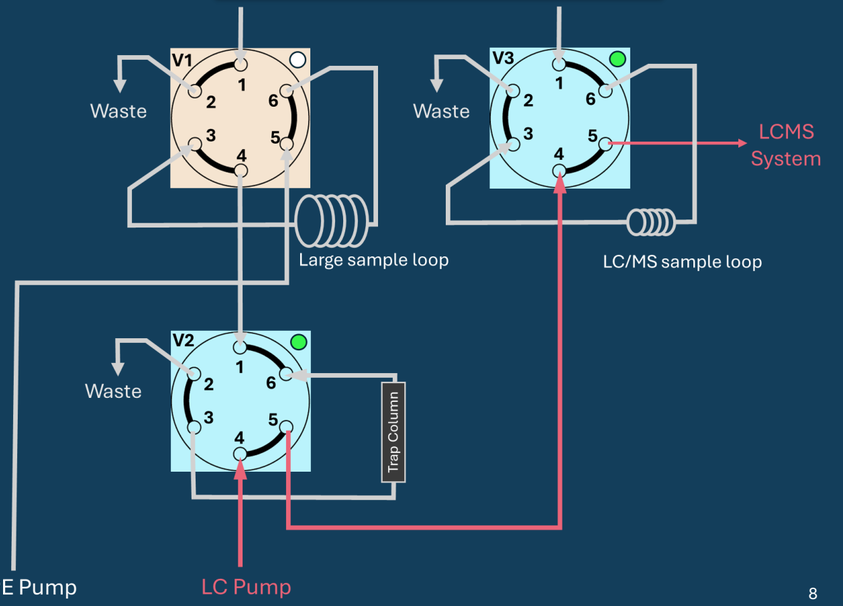
Step 1: Sample Injection



Step 2: SPE Load & Wash



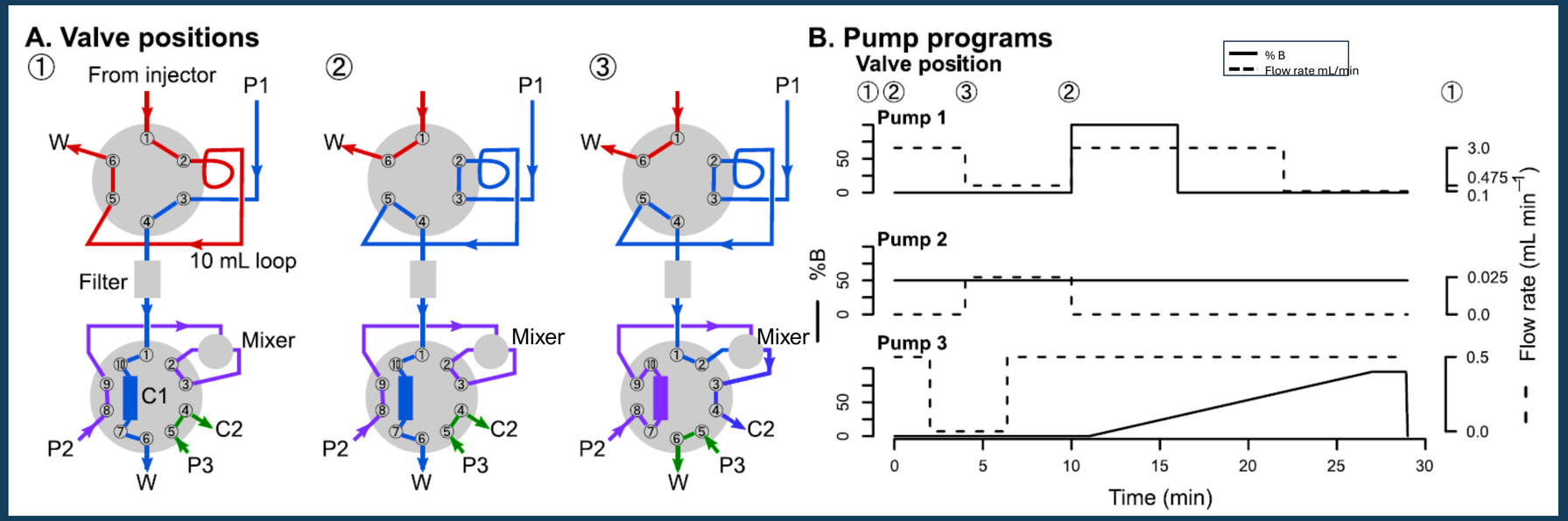
Step 3: SPE Elute



Step 4: LCMS analysis

Online SPE - Peak-focusing Approach Involving a 10-Port Valve

System Configuration



Pump 1:
A: 0.1%(v/v) acetic acid in water;
B: 2% NH_4OH in 1:1 MeOH/ACN
Pump 2:
A & B: 2% NH_4OH in a 1:1 MeOH/ACN
Pump 3:
A: water;
B: ACN

Pre- and post-run, Sample loading onto the loop → Run start, Sample loaded onto C1 → C1 back-eluted, eluent diluted with make-up flow in a mixer then onto C2 → Cleaning and re-equlibration of C1, C2 gradient-eluted onto MS → Pre- and post-run, Sample loading onto the loop

P1, P2, P3: UHPLC pumps
W: Waste ports
C1: Trapping column (AX-RP)
C2: Analytical column


High-Throughput Trace-Level Suspect Screening for Per- and Polyfluoroalkyl Substances in Environmental Waters by Peak-Focusing Online Solid Phase Extraction and High-Resolution Mass Spectrometry

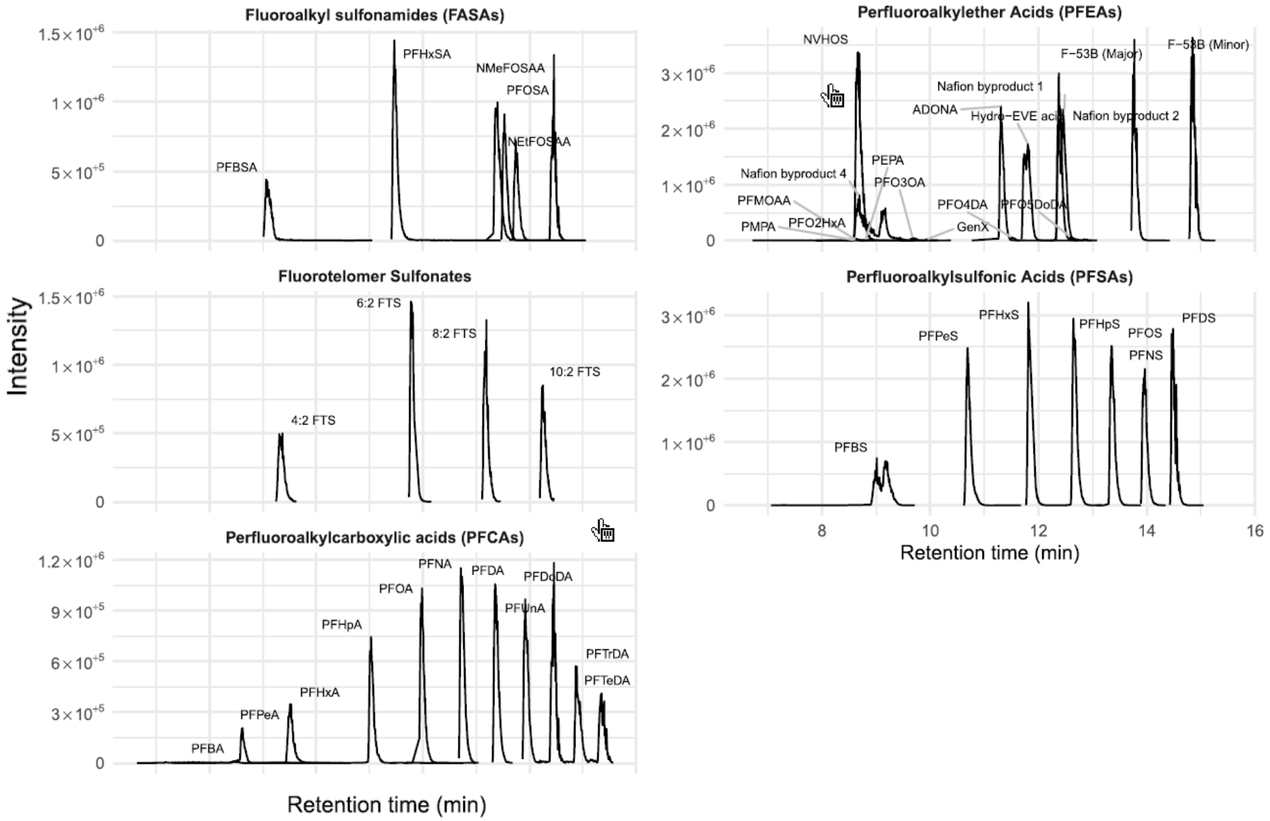
Gordon J. Getzinger* and P. Lee Ferguson*

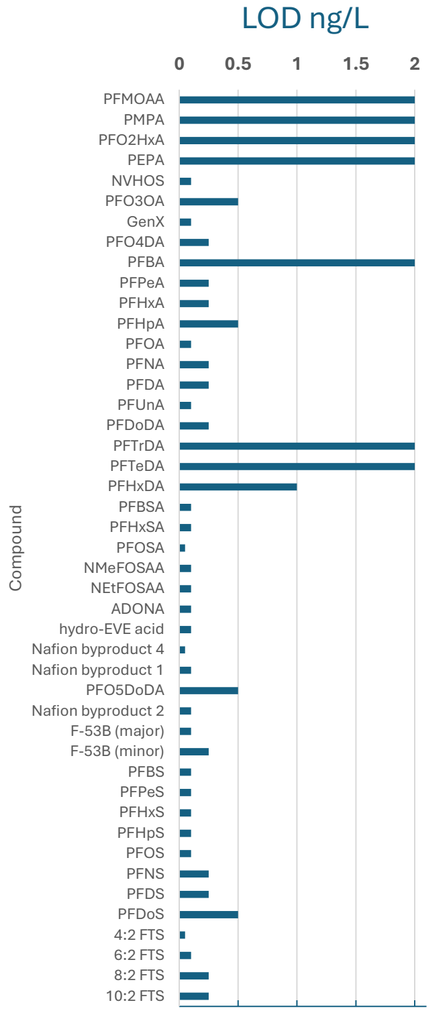
Cite This: https://doi.org/10.1021/acsestwater.0c00309 Read Online

Source: ACS EST Water 2021, 1, 5, 1240−1251, https://doi.org/10.1021/acsestwater.0c00309

Online SPE - Peak-focusing Approach Involving a 10-Port Valve

Analytical Results





Results:

The method only required only 6 mL of sample and <40 min total time for SPE and LC-HRMS analysis.

Sample focusing with back-eluting of trapping column and make-up flow led to sharp peaks.

Detection limits of 0.1-4 ng/L were reached for the 45 PFAS analytes.

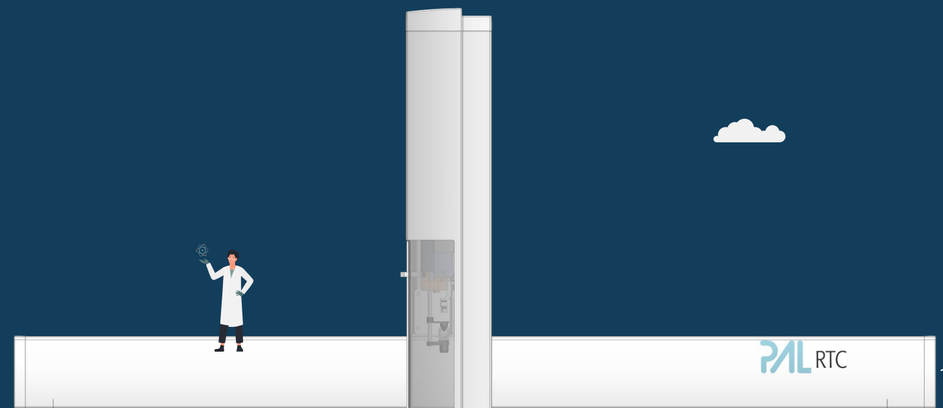
The accuracy and precision on repeated analysis of a standard reference material were typically 89-103% and <10%, respectively.

Online SPE vs. Offline SPE

VS

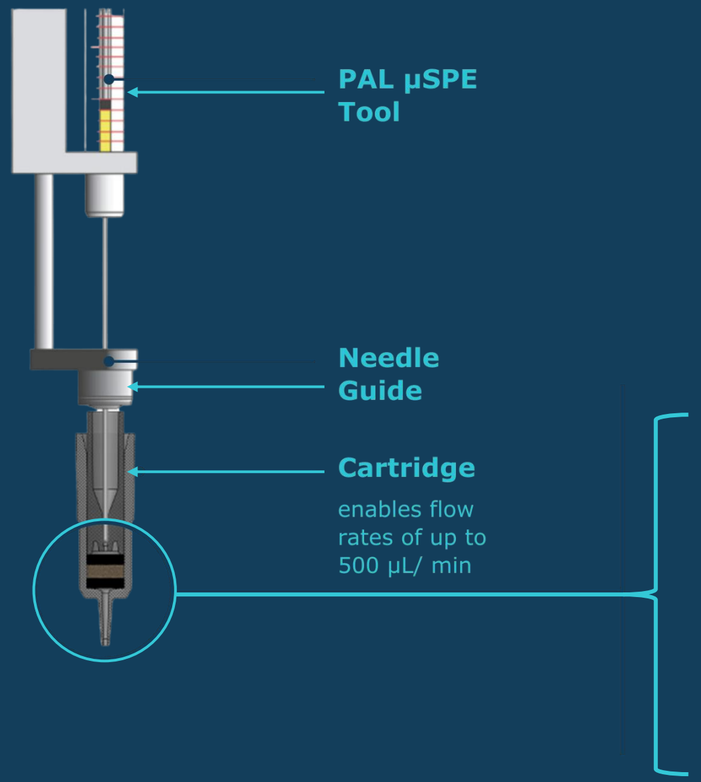
Benefits

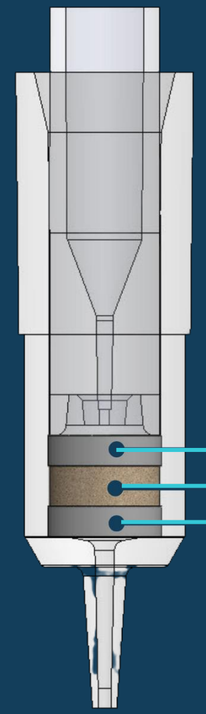
- Less sample needed
- Higher throughput
- Improved repeatability
- Reduced risk of contamination
- Transferability / Ease of use



Introduction to Micro-SPE (µSPE)

Miniturized SPE





The septumless µSPE mini-cartridge design eliminates the seal leakage issues of other vendors

- Can penetrate pre-slit septa
- Deliver **directly** into LC injection ports

bed masses can be increased to up to 100 mg

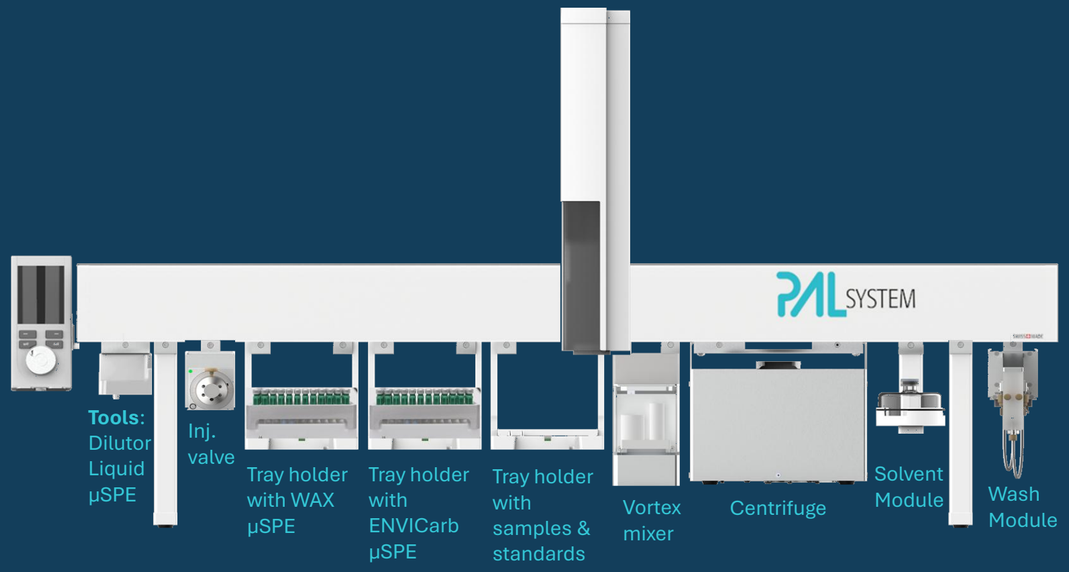
Green Chemistry

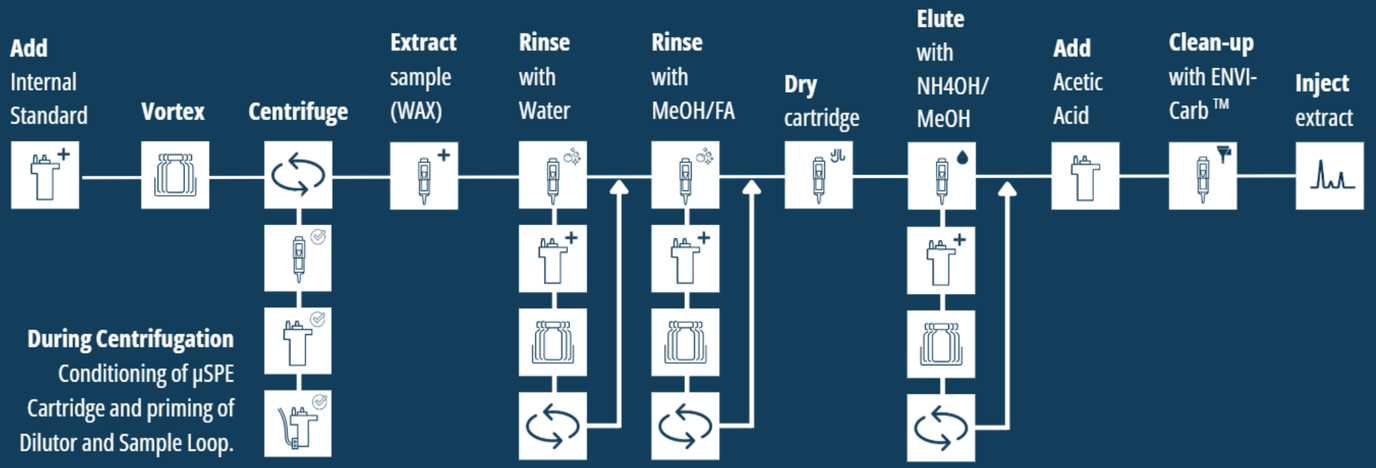
Automated

High Throughput

EPA1633 using µSPE

Fully Automated Workflow for Analyzing PFAS in Stormwater





8ml PP vial
High-recovery design

µSPE cartridges
(WAX & ENVI-Carb™)

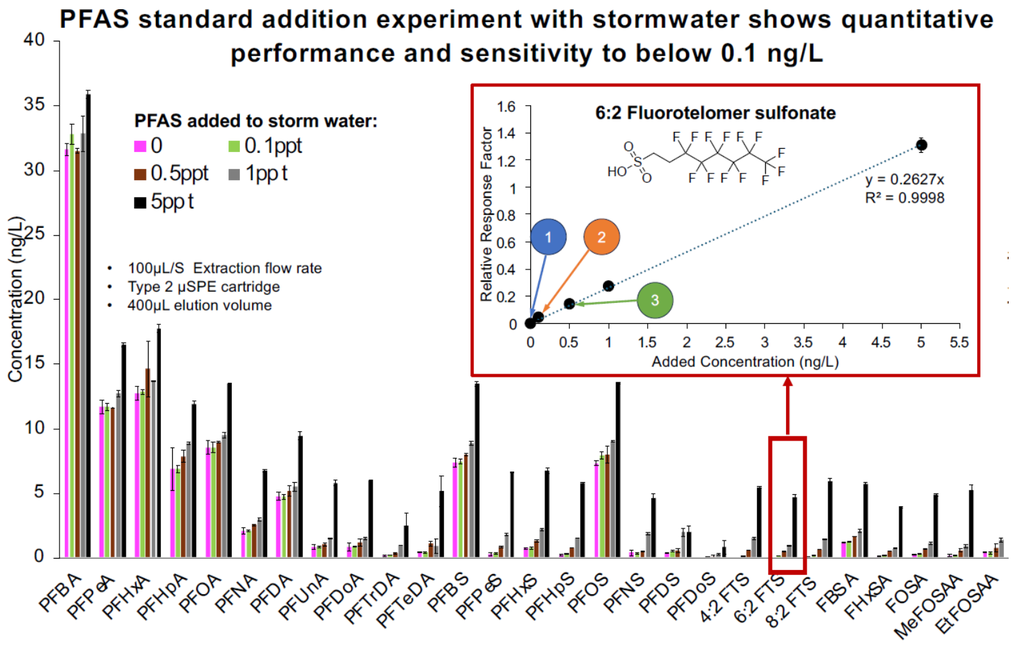
Automated:

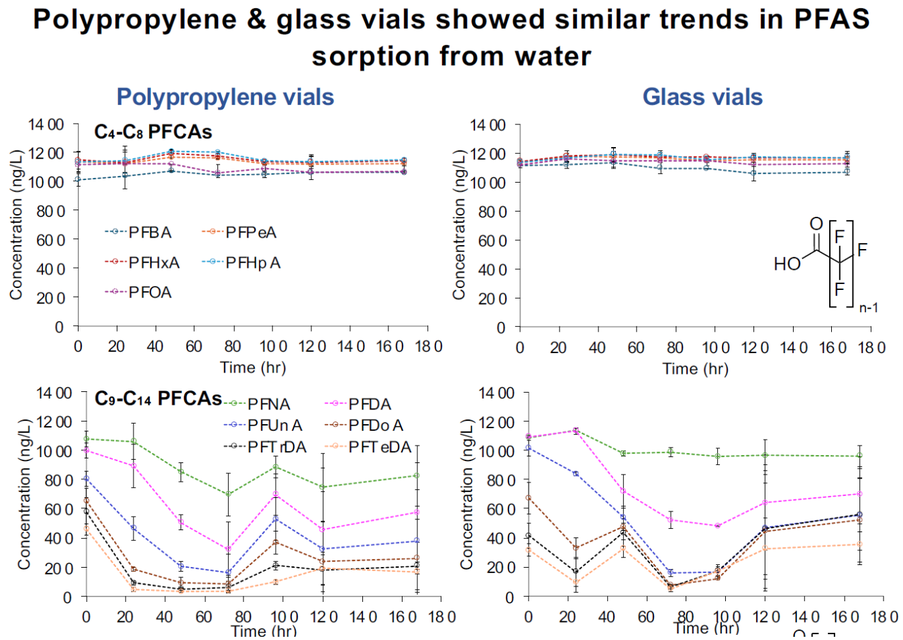
- ✓ Pre-conditioning, loading and elution of WAX µSPE
- ✓ 3x Rinsing of sample vials
- ✓ Addition of acid
- ✓ ENVI-Carb Clean-up
- ✓ Online injection into LCMS

Source: *Poster at ASMS 2025 by M. Shojaei, download at www.palsystem.com/en/pal-system-at-asms-2025*

EPA1633 using µSPE

Fully Automated Workflow for Analyzing PFAS in Stormwater



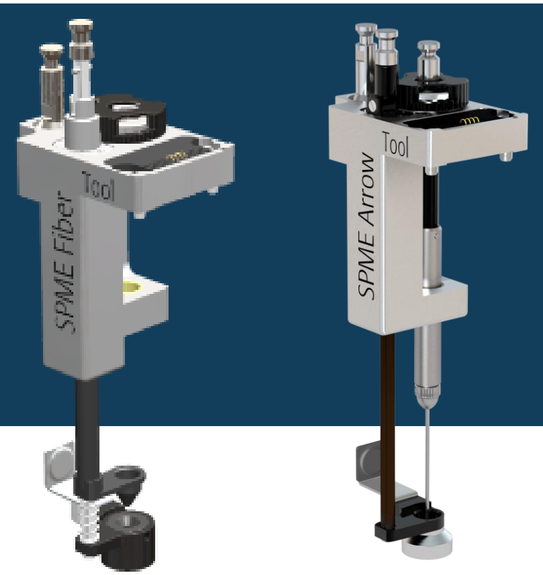


- Fully automated sample preparation and parallel analysis in < 30 minutes per sample without user intervention.
- Large-volume PP vials showed no loss of short chain PFAS (< C_8) but sorptive losses of 30-60% long chain PFAS over 6 days of sample storage (recovered with methanol rinse during sample processing).
- WAX µSPE Pro cartridges allow fast (100 µL/sec) sample loading with high recovery.
- Sample concentration factors of 20 allowed acceptable accuracy and sensitivity at concentration ⩾ 0.1 ng/L for PFAS < C_{11}.

Source: *Poster at ASMS 2025 by M. Shojaei, download at www.palsystem.com/en/pal-system-at-asms-2025*

Volatile PFAS in Water by Solid-Phase-Micro-Extraction (SPME)

- SPME effectively preconcentrates FTOHs, FOSAs, and FOSE from aqueous samples.
- Solventless thermal desorption of PFAS enhanced method performance.
- Headspace- and Direct Immersion-SPME methods were optimized and applied to simulated seawater.
- SPME-GC-MS quantified volatile PFAS at ppt concentrations with excellent repeatability.



Figures of merit of the validated HS-SPME-GC-MS method.

Analytes	LOD		LOQ		Precision (RSD, %)		Accuracy (%)		Accuracy (%) in Simulated Sea Water
	µg L^{-1}	S/N	µg L^{-1}	S/N	Intra-day[a]	Inter-day[b]	0.3 µg L^{-}	3 µg L^{-1}	1 µg L^{-1}
4:2 FTOH	0.01	6	0.05	30	2	4	111	118	99
6:2 FTOH	0.01	84	0.05	380	2	2	110	117	97
8:2 FTOH	0.01	109	0.05	494	1	2	102	116	111
Me_2FOSA	<0.005	–	0.005	426	5	11	104	92	114
EtFOSA	<0.005	–	0.005	229.	1	2	107	105	101
MeFOSE	0.1	30	0.25	145	3	7	97	98	93

Dash (–) = concentration level not within linear dynamic range.

[a] n = 3.

[b] n = 5.

Source: *H. Martínez-Pérez-Cejuela et al, Effective preconcentration of volatile per- and polyfluoroalkyl substances from gas and aqueous phase via solid phase microextraction, Analytica Chimica Acta, Volume 1345, 2025, 343746, ISSN 0003-2670, https://doi.org/10.1016/j.aca.2025.343746*

Reliable PFAS Analysis for Water Samples

Automation & The Bigger Picture

Challenges in Sample Prep

- Trace-level detection (ng/L) requires high sensitivity and minimal background contamination.
- Manual workflows are time-consuming and prone to variability.
- Complex matrices like surface or wastewater increase risk of analyte loss or interference.

Automated Workflows

- Automated workflows reduce human errors and risk of contamination.
- Direct online injection allows prep-ahead, preparing and analyzing samples in parallel.
- Configurable modules enable end-to-end automation.

Conclusion & Outlook

- Automation improves repeatability, reproducibility and traceability.
- Modular PAL workflows support evolving regulatory demands for PFAS monitoring and usage for other sample matrices such as soil and foods.

Learn more about PFAS analysis and PFAS-free consumables to reduce contaminations and subsequently background noise.

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Ingenious sample handling

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