Advancing PFAS Analysis

Robotic Automation and Streamlined LC-MS Workflows

PAL SYSTEM
Ingenious sample handling

This webinar will be recorded

Webinar Information

Microphone and webcam are **turned off**

Chat function is **turned off**

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Recording in progress

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While your microphone and camera will be disabled, your name and any questions you may ask in the chat feature will be visible to other participants.

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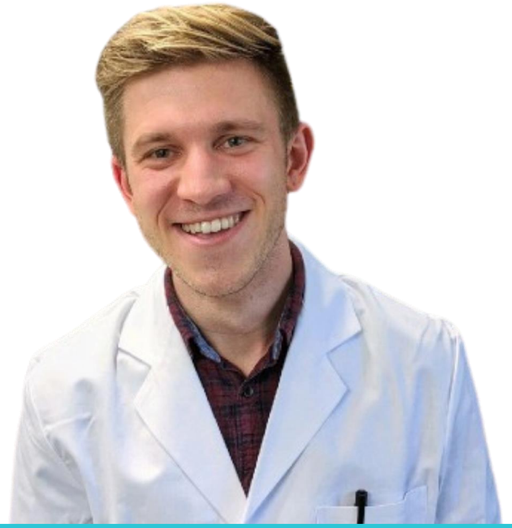
Meet today's hosts

Tiantian Li, PhD

Product Manager, CTC Analytics

- Expert in LC and LC-MS product development and marketing
- Over 10 years of experience in the field
- Currently focused on developing PFAS workflow solutions



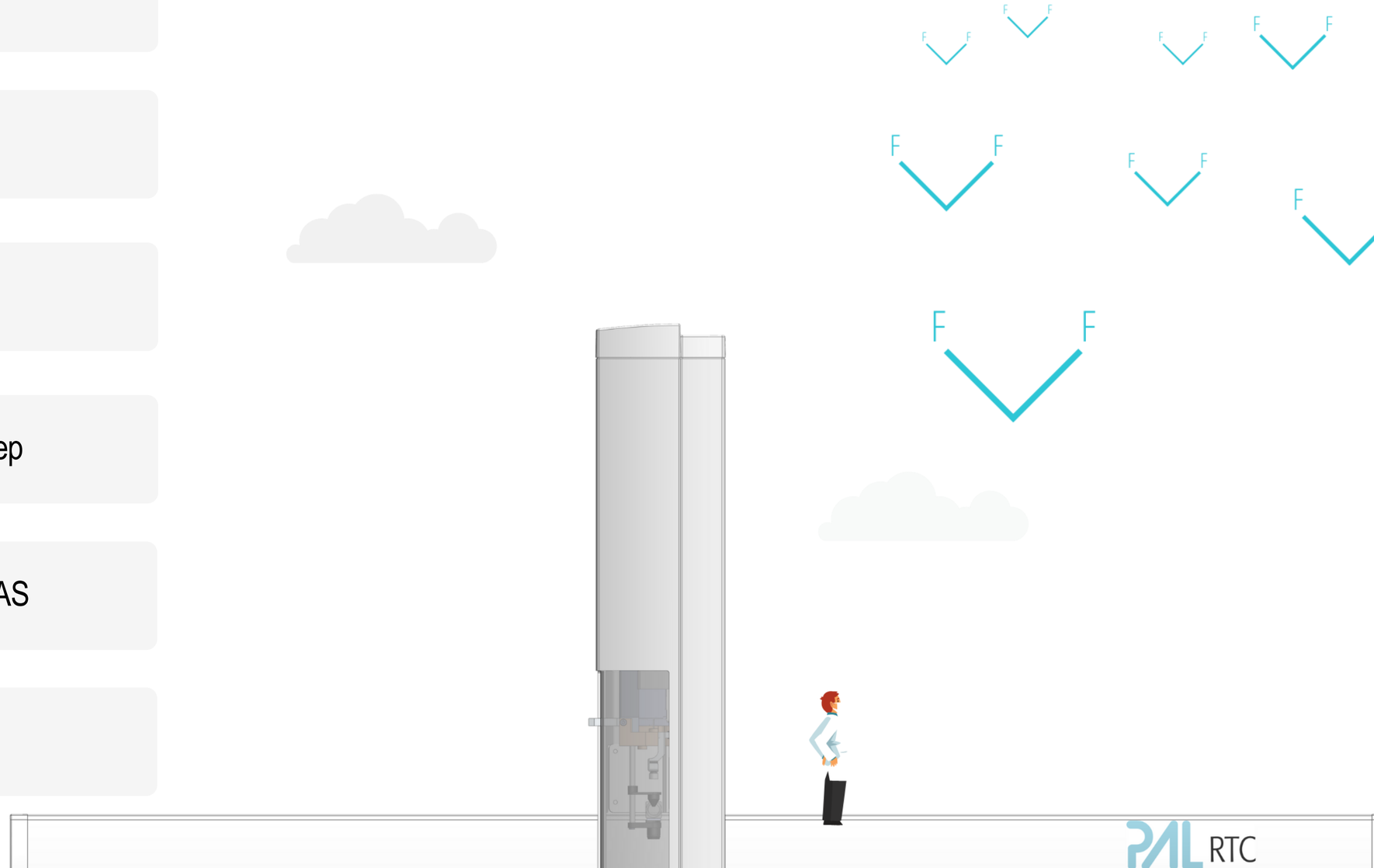
Hagen M. Gegner, PhD

Content and Digital Marketing, CTC Analytics

- Background in clinical metabolomics
- Method development in LC-MS and study design
- Focused on translating scientific content to broader audiences

Agenda

- Introduction PAL System
- PFAS – an overview
- Regulations
- PFAS Analysis and Sample Prep
- Deep Dive: Online-SPE for PFAS
- Q & A



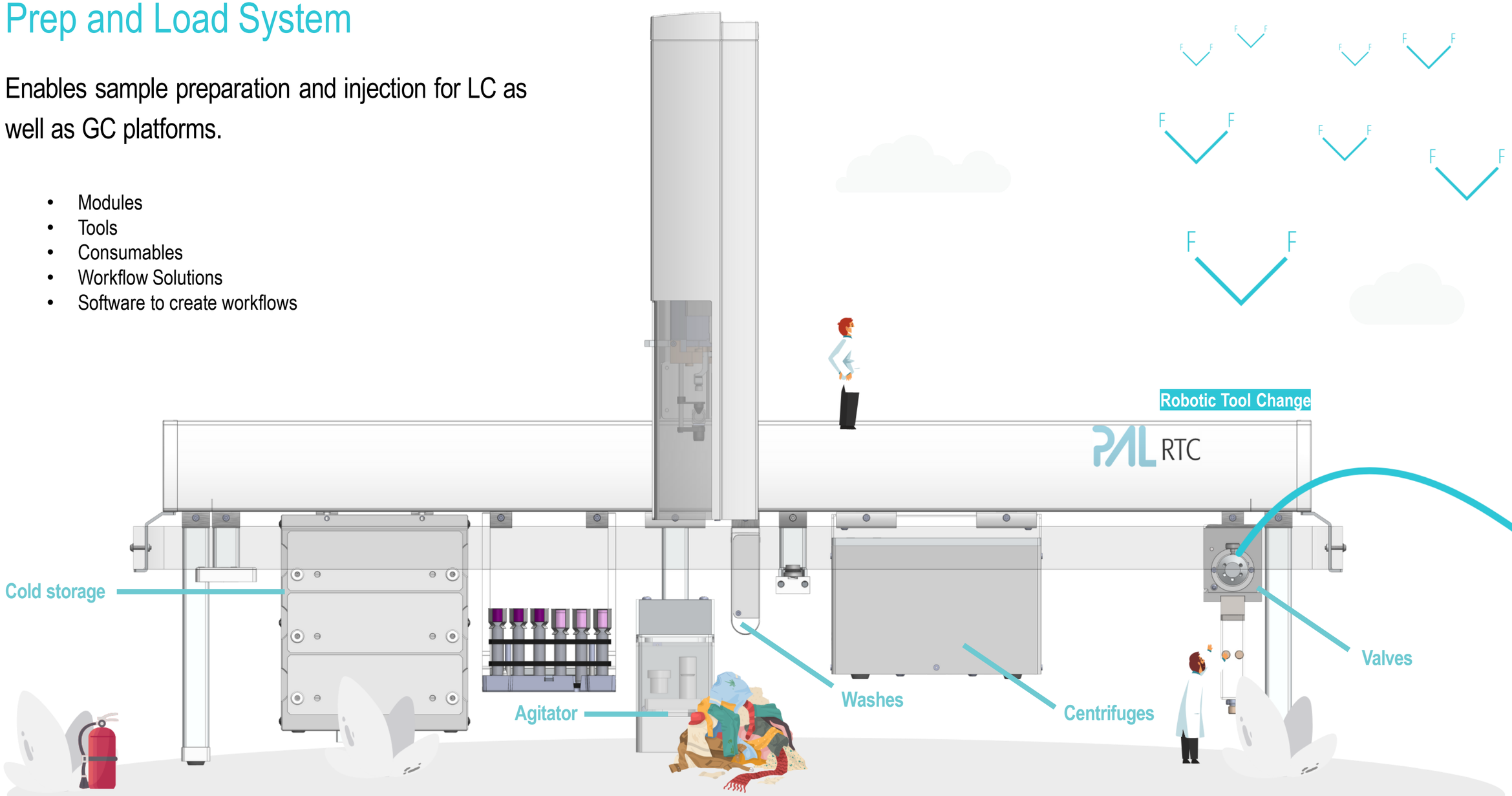
PAL System

PAL SYSTEM
Ingenious sample handling

Prep and Load System

Enables sample preparation and injection for LC as well as GC platforms.

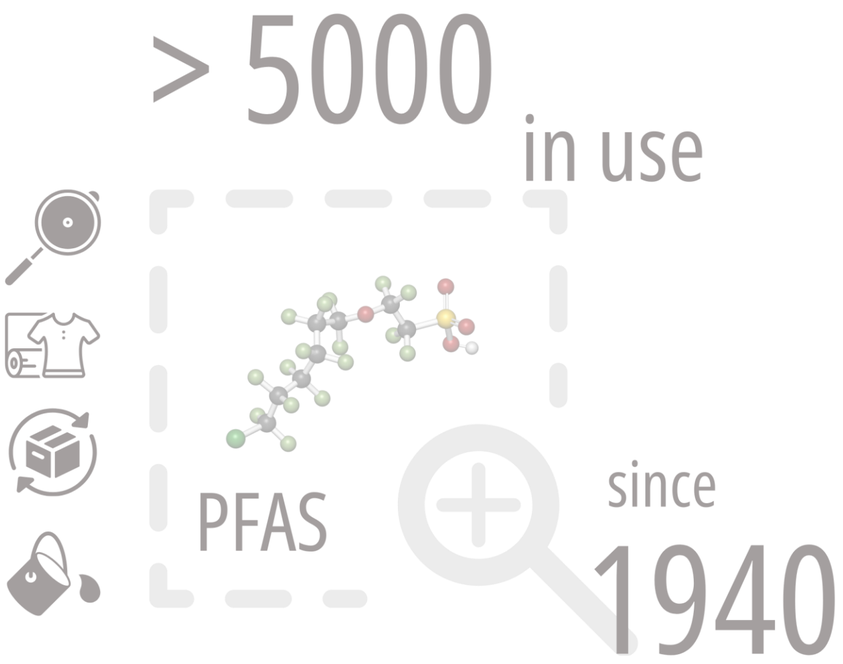
- Modules
- Tools
- Consumables
- Workflow Solutions
- Software to create workflows



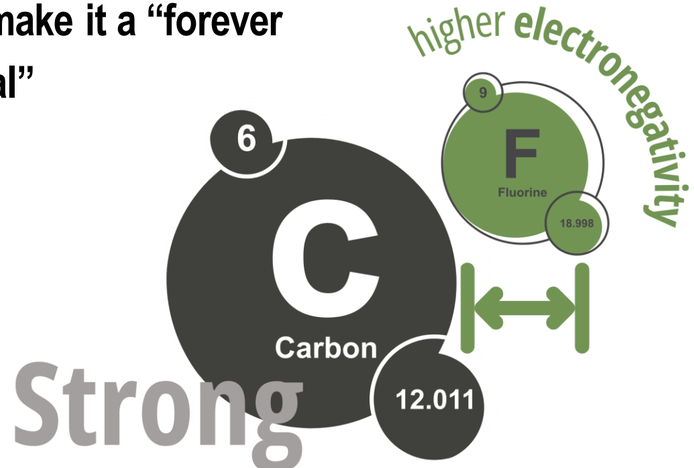
PFAS - Introduction

Per- and polyfluoroalkyl substances (PFAS)

Dhiman, S., & Ansari, N. G. (2024). A review on extraction, analytical and rapid detection techniques of Per /Poly fluoro alkyl substances in different matrices. In Microchemical Journal (Vol. 196). Elsevier Inc. https://doi.org/10.1016/j.microc.2023.109667



Multiple carbon–fluorine (C–F) bonds make it a "forever chemical"



C-F bond is resistant to

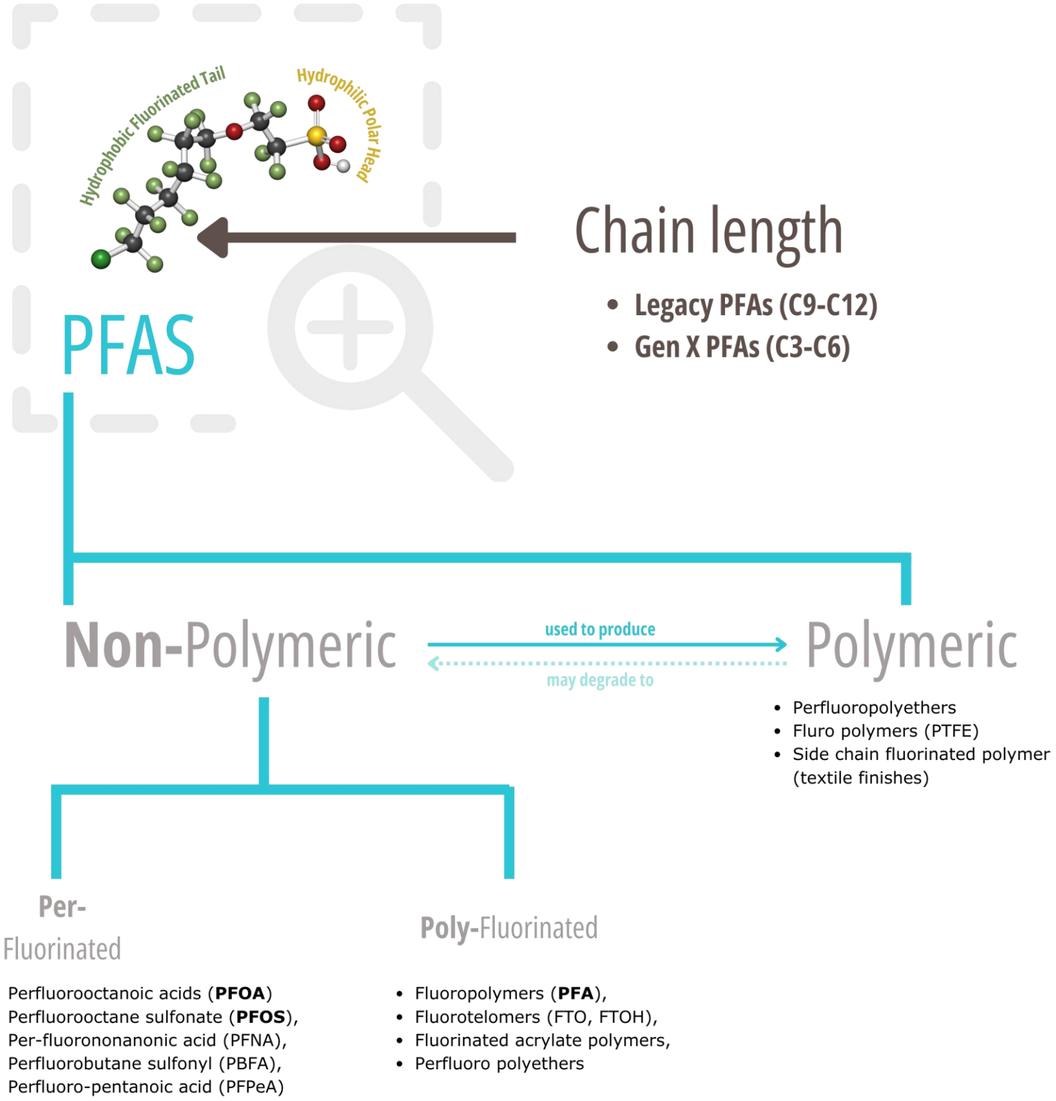
- thermal,
- chemical,
- metabolic degradation

PFAS are commonly found in various products like **food packaging, textiles, paint** and more, but their stability poses risks to both the environment and human health

PFAS - Introduction

Per- and polyfluoroalkyl substances (PFAS)

Dhiman, S., & Ansari, N. G. (2024). A review on extraction, analytical and rapid detection techniques of Per /Poly fluoro alkyl substances in different matrices. In Microchemical Journal (Vol. 196). Elsevier Inc. https://doi.org/10.1016/j.microc.2023.109667



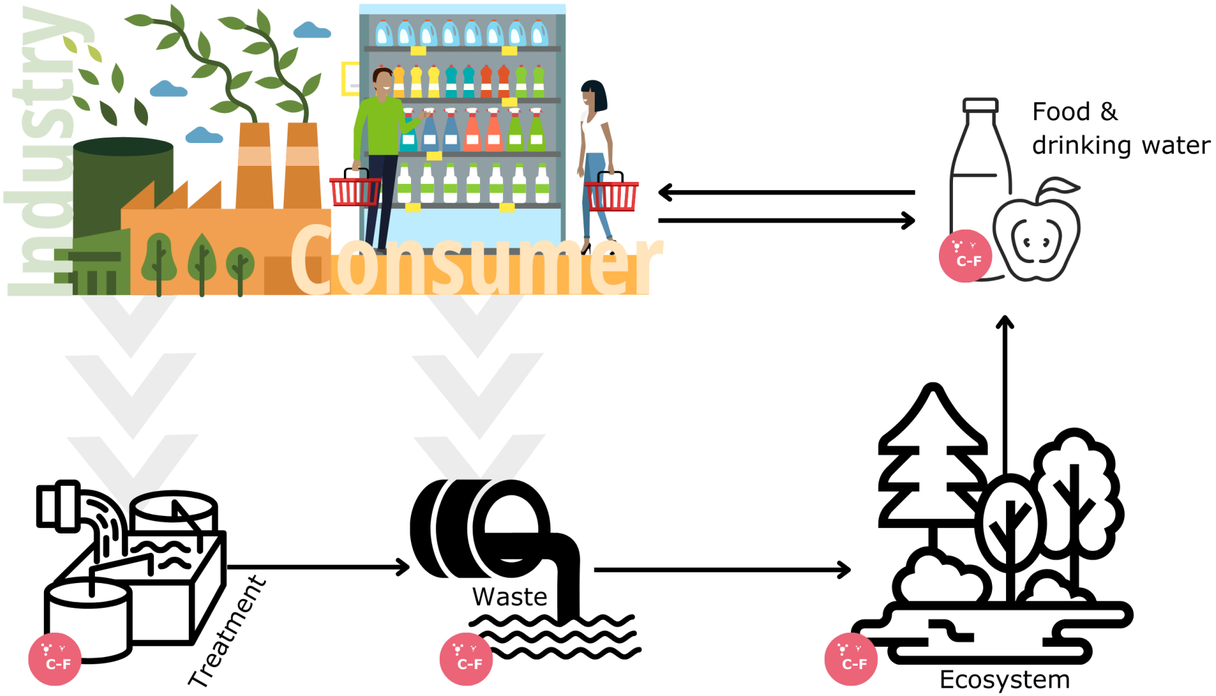
Perfluorooctanoic acid (PFOA) and **perfluorooctane sulfonate (PFOS)**

- highly persistent,
- leading to bioaccumulation
- potential harm to aquatic organisms and human health

PFAS - Introduction

PFAS enter the environment and humans through various pathways
e.g. industrial releases, firefighting activities, and disposal of consumer products

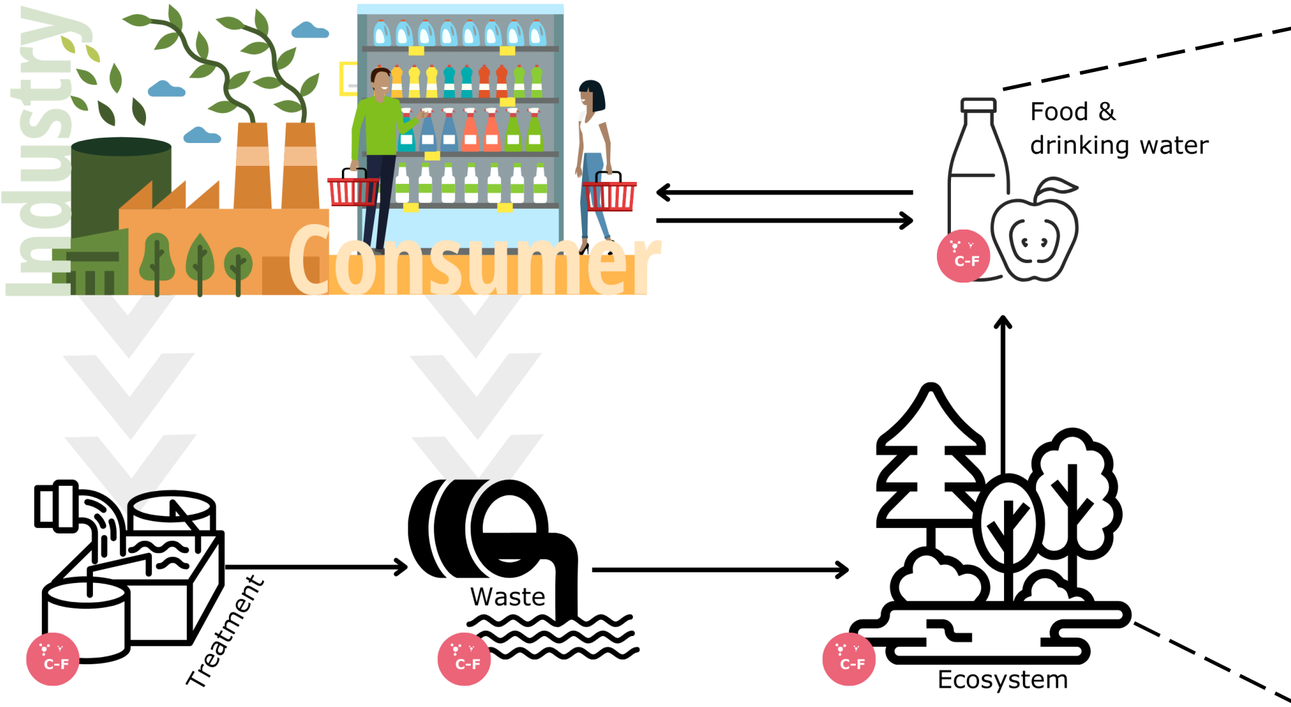
Wee, S. Y., & Aris, A. Z. (2023). Revisiting the “forever chemicals”, PFOA and PFOS exposure in drinking water. In npj Clean Water (Vol. 6, Issue 1). Nature Research. https://doi.org/10.1038/s41545-023-00274-6

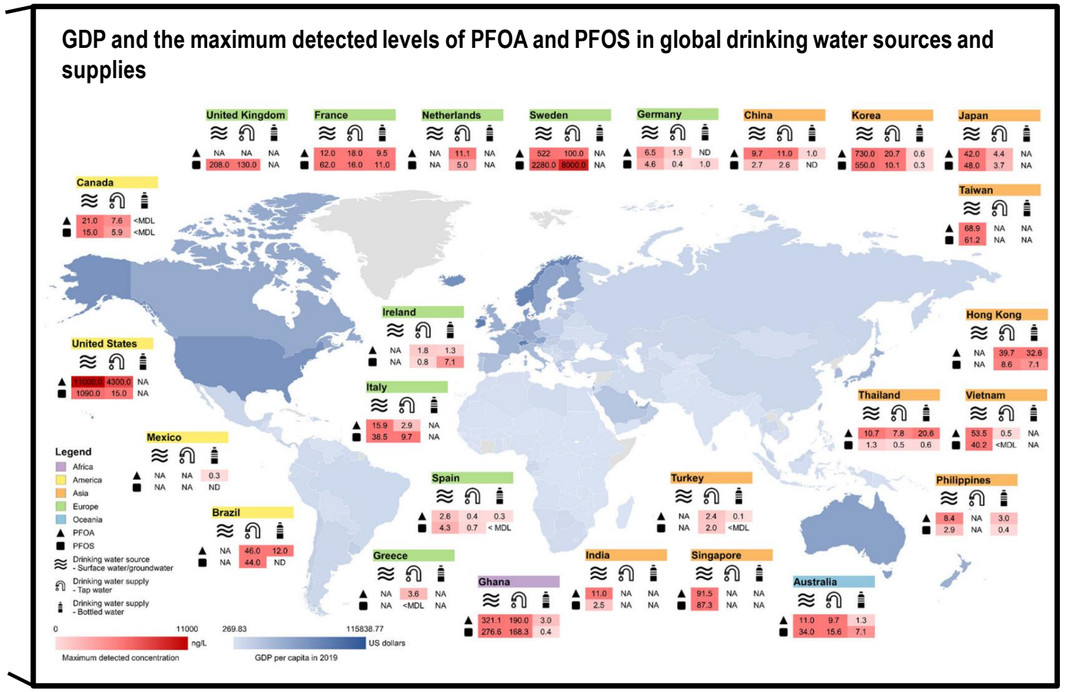


PFAS - Introduction

A global concern due to their widespread presence and persistence

Wee, S. Y., & Aris, A. Z. (2023). Revisiting the “forever chemicals”, PFOA and PFOS exposure in drinking water. In npj Clean Water (Vol. 6, Issue 1). Nature Research. https://doi.org/10.1038/s41545-023-00274-6

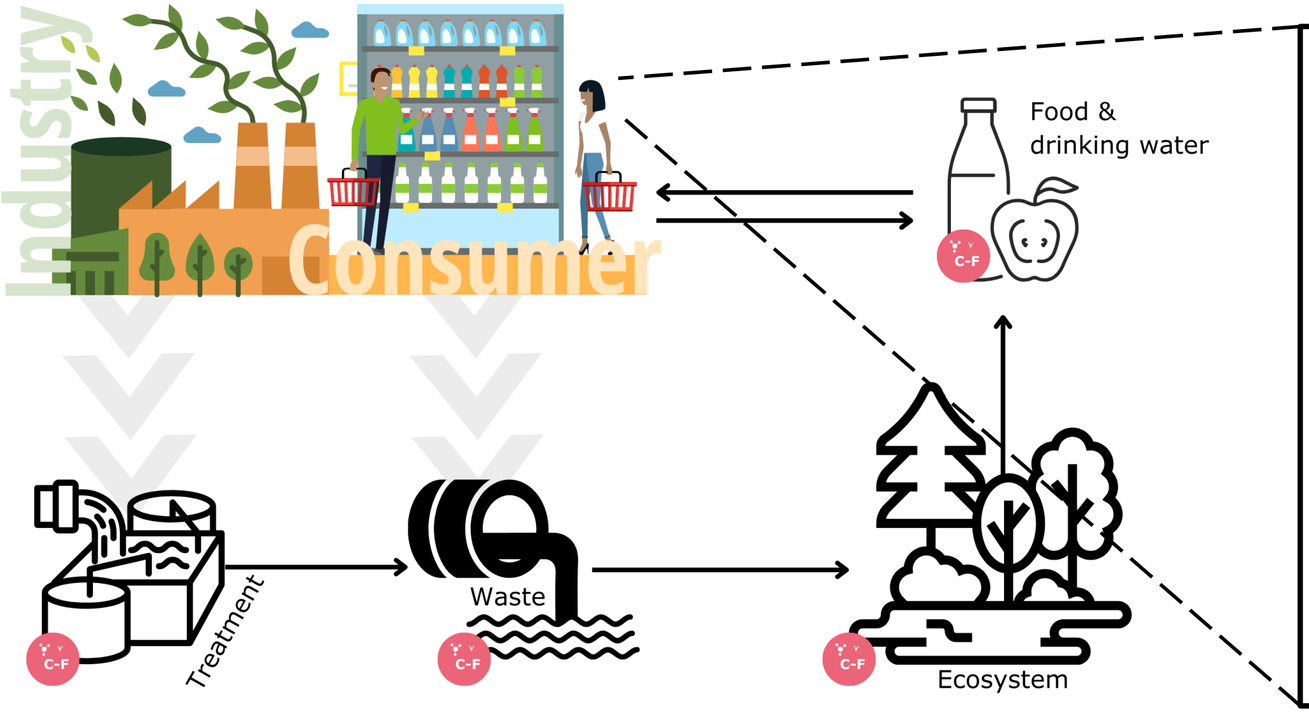




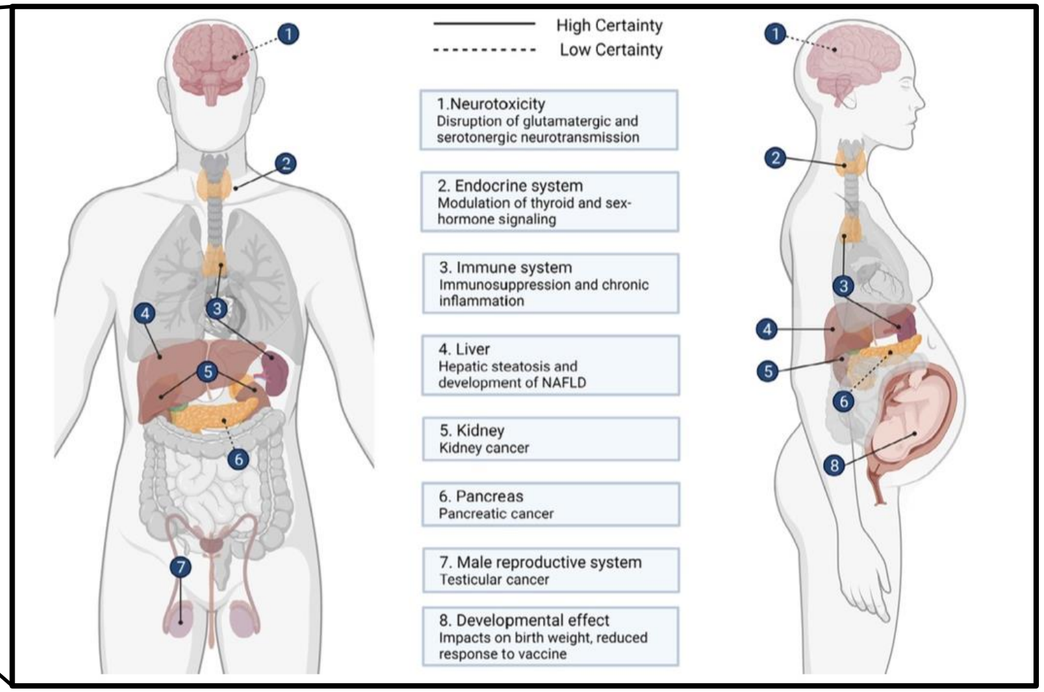
PFAS - Introduction

Elevated risks of cancer, immune responses, metabolic syndromes, developmental issues, and reproductive effects

Wee, S. Y., & Aris, A. Z. (2023). Revisiting the "forever chemicals", PFOA and PFOS exposure in drinking water. In npj Clean Water (Vol. 6, Issue 1). Nature Research. https://doi.org/10.1038/s41545-023-00274-6



UC Davis Dept of Molecular Biosciences (pagesuite-professional.co.uk)

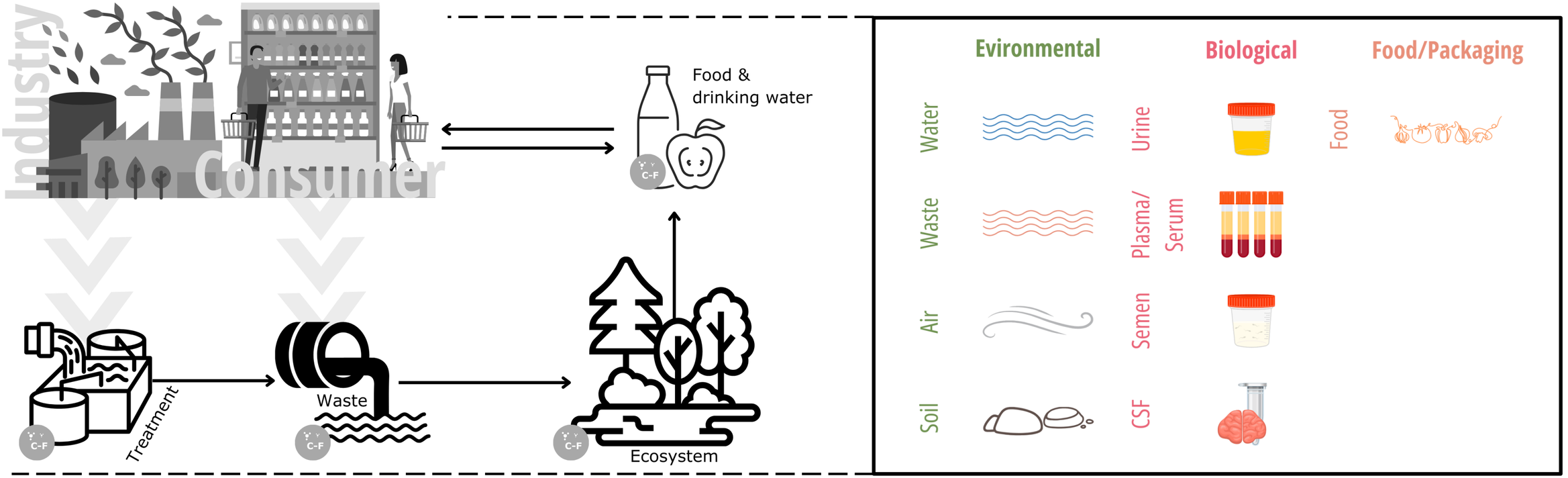


- Additive and synergistic effects of PFAS mixture
- PFAS exposure exhibit potential sex-dependent variations

PFAS - Introduction

A wide array of sample types for analysis

Dhiman, S., & Ansari, N. G. (2024). A review on extraction, analytical and rapid detection techniques of Per /Poly fluoro alkyl substances in different matrices. In *Microchemical Journal* (Vol. 196). Elsevier Inc. https://doi.org/10.1016/j.microc.2023.109667



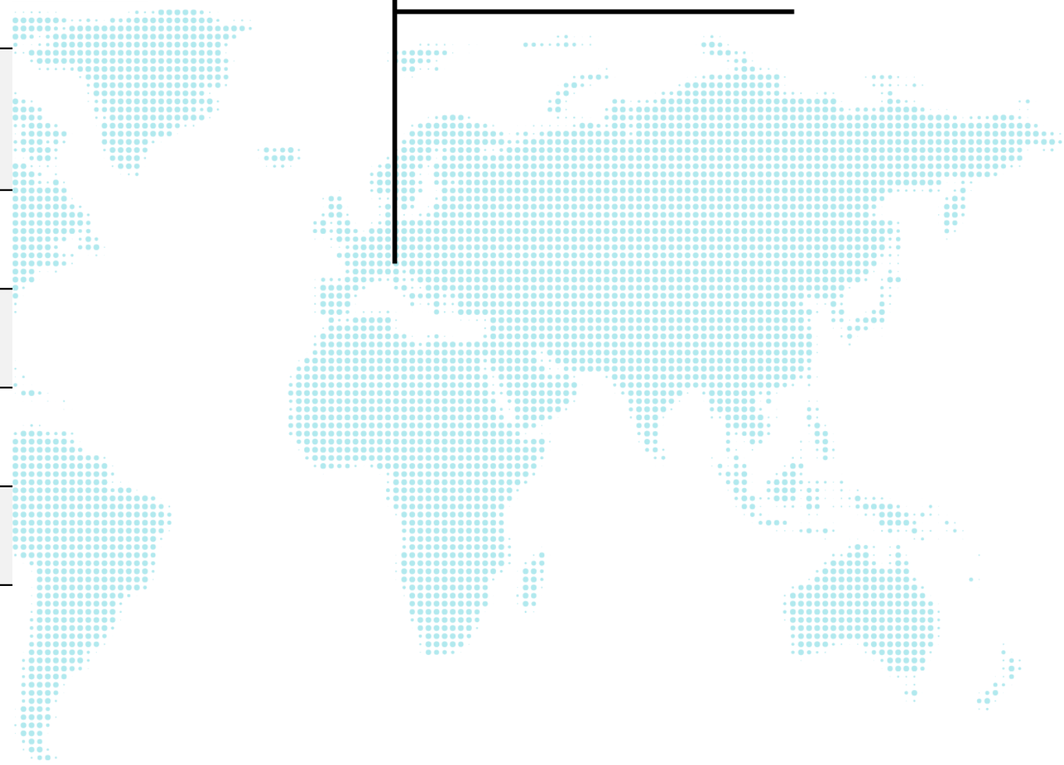
PFAS - Regulations

USA and EU

Method	Matrix Tested	No. of Analytes	Sample Prep Procedure
EPA 537	Drinking water	14	SPE
EPA 537.1	Drinking water	18	SPE
EPA 533	Drinking water	29	SPE
EPA 1633 (draft)	wastewater, surface water, groundwater, soil, biosolids, sediment, landfill leachate, and fish tissue	40	SPE
EPA 8327 (draft)	Surface water, ground water, wastewater influent and effluent	24	Dilute & shoot
ASTM D7979	Surface water, ground water, wastewater influent and effluent	21	Dilute & shoot
ASTM D7968	Soil and solids	21	Organic extraction with MeOH
ISO/DIS 21675 (draft)	Drinking water, sea water, fresh water, wastewater	30	SPE

January 2021
Drinking Water Directive (EU)
PFAS Total: **500 ng/L**
Sum of PFAS: **100 ng/L**

Regulation (EU) 2022/2388

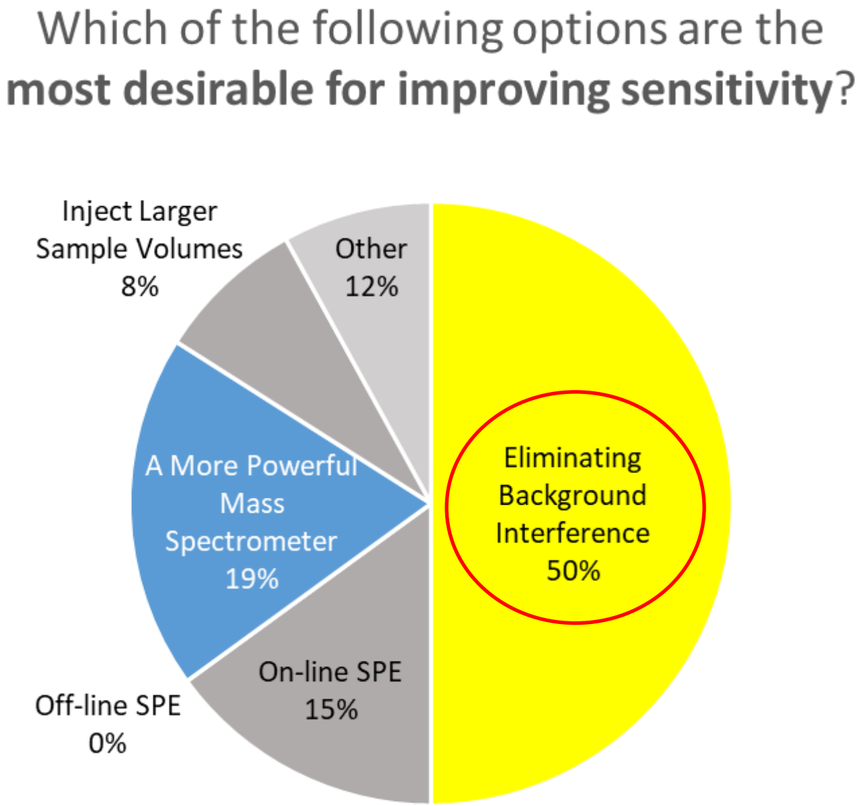


Regulations are still evolving

Number of analytes ↑
Concentration limits ↓

Challenges in PFAS Analysis

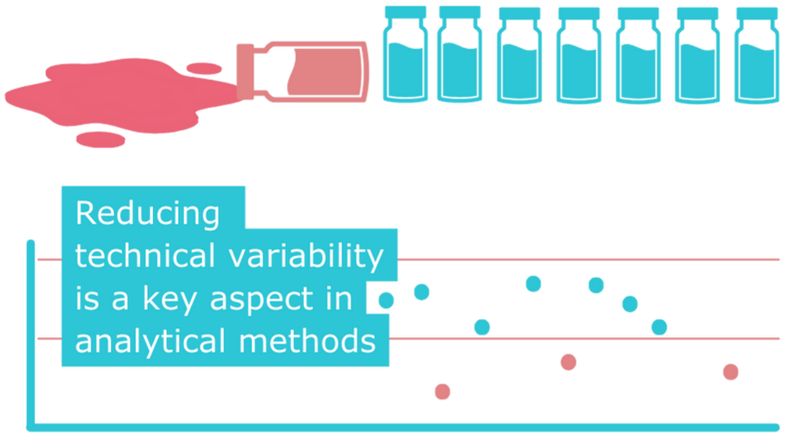
How to reach the Low Detection Limit required?

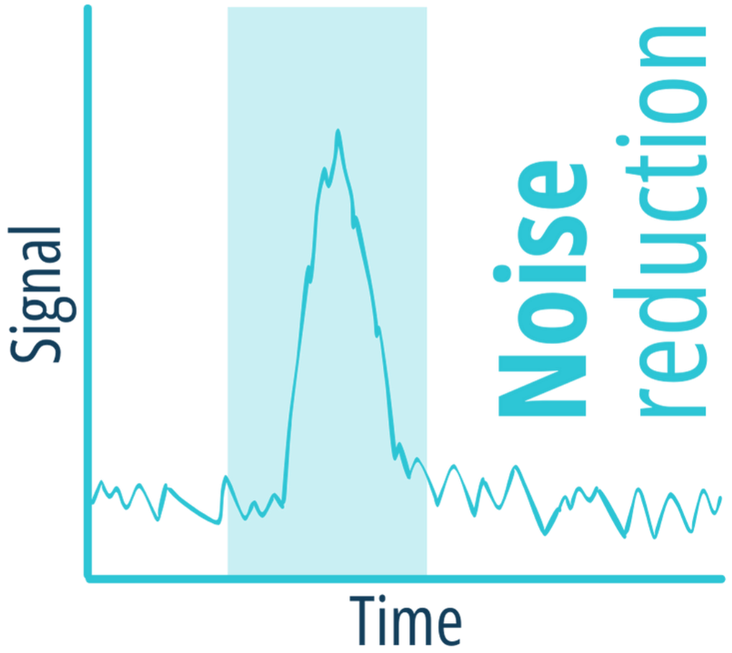


Source: *Trends and Challenges in PFAS Analysis (theanalyticalscientist.com)*

Challenges in PFAS Analysis

Reducing technical variability and noise through automation





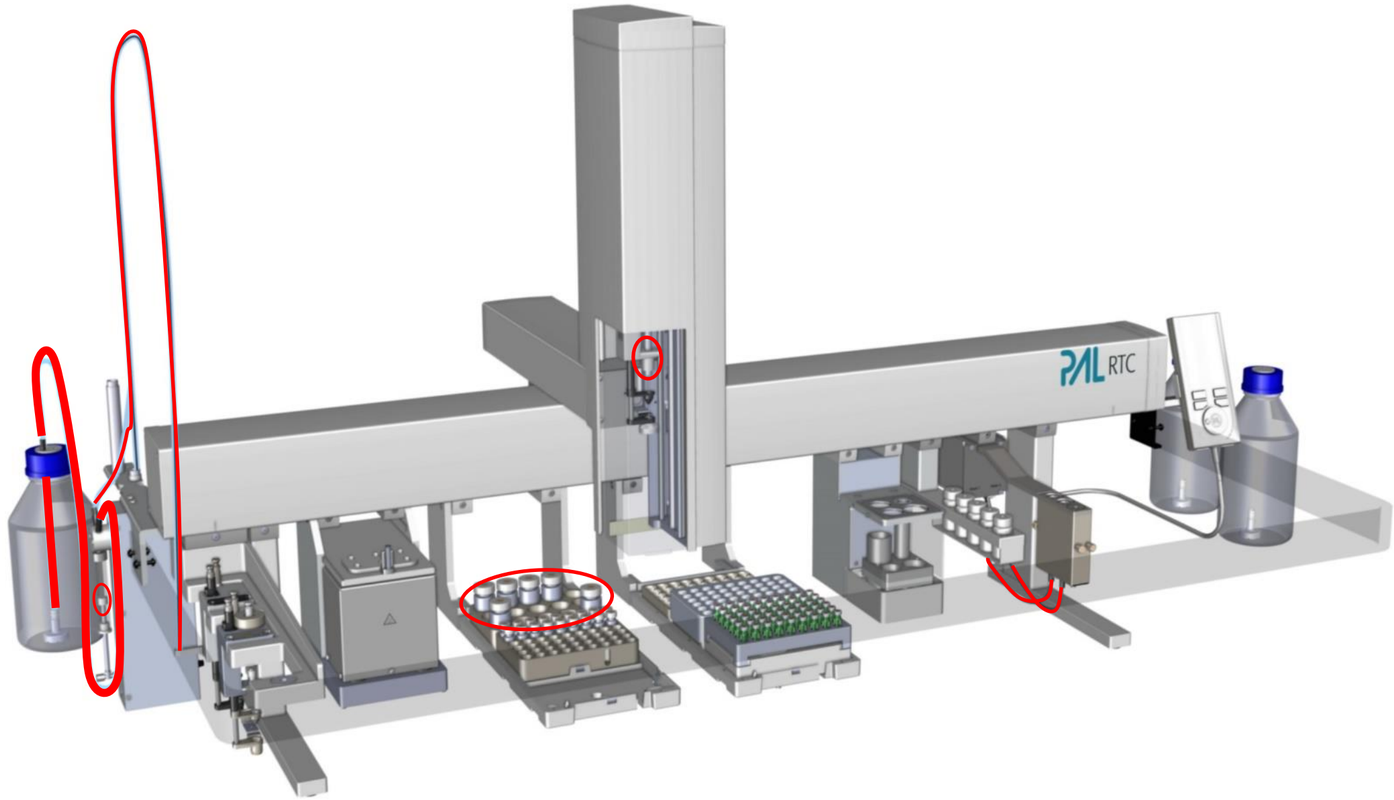
Benefits of automation in PFAS Analysis

- Traceability and Reproducibility: Logs actions (QC/QA) for reliable data across batches and labs.
- Reduced Variability: Robust and timed workflows for routine analysis.
- Increased Sample Throughput: Automates tedious tasks for faster analysis – 24/7.
- Precision Handling: Minimizes manual errors for accurate and reliable results.
- Contamination Control: Dedicated consumables and workflows reduce cross-contamination risks.

Background Interference

Potential sources of PFAS from a PAL System

- Tubings and Loops from:
  - LCMS Tool (PFA tubings, FEP loop)
  - Dilutor
  - Fast Wash
  - HF-Fast Wash
- Wash cup
- Syringes with PTFE plunger
- Sample vials and caps



Background Interference

Reduce background contamination with PFAS-free products

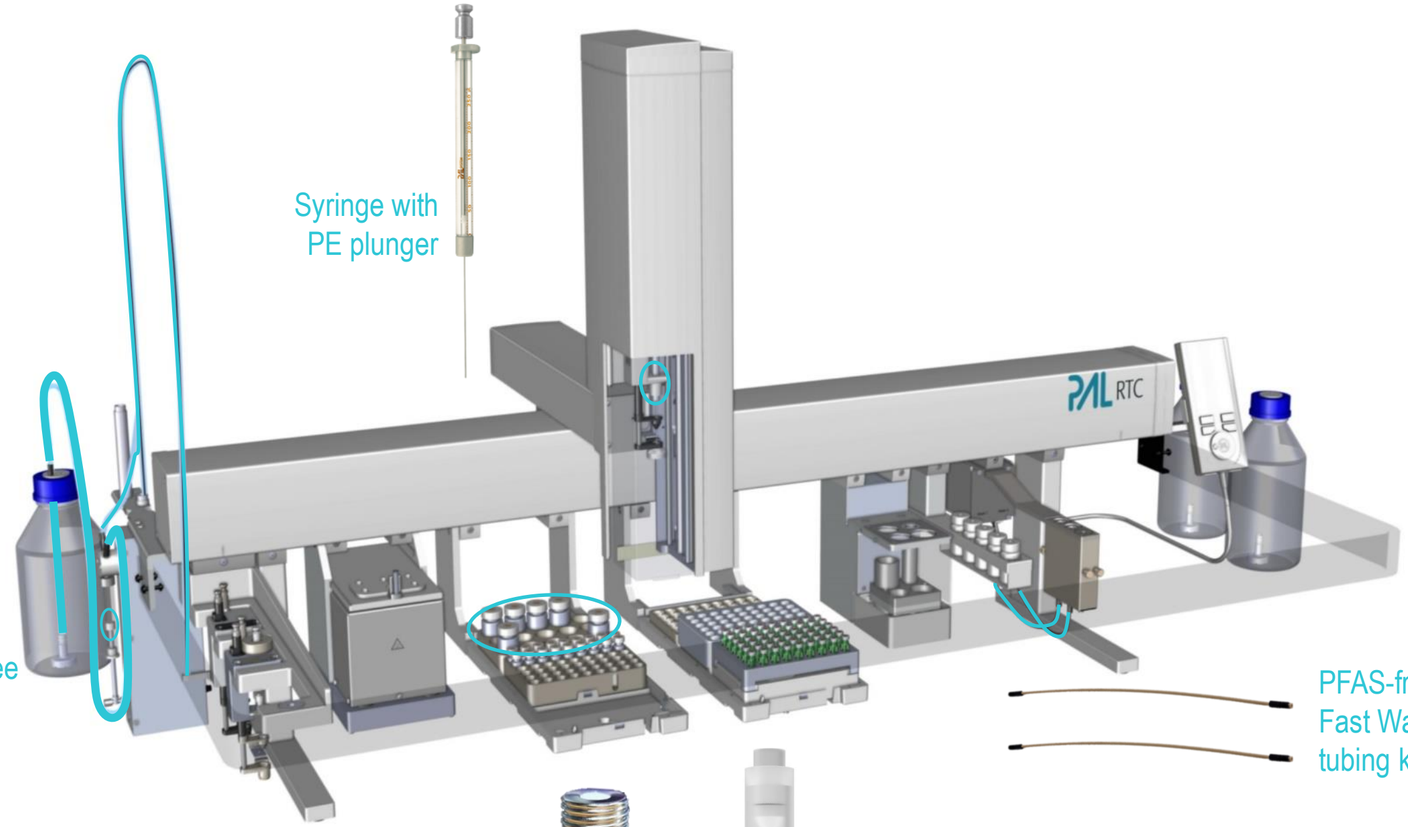
Use the PAL Product Finder and search for “PFAS-free“
www.palsystem.com/en/products/pal-product-finder/

Solvent inlet tubing kit

PFAS-free dilutor syringe

PEEK connection tubing kit

Syringe with PE plunger



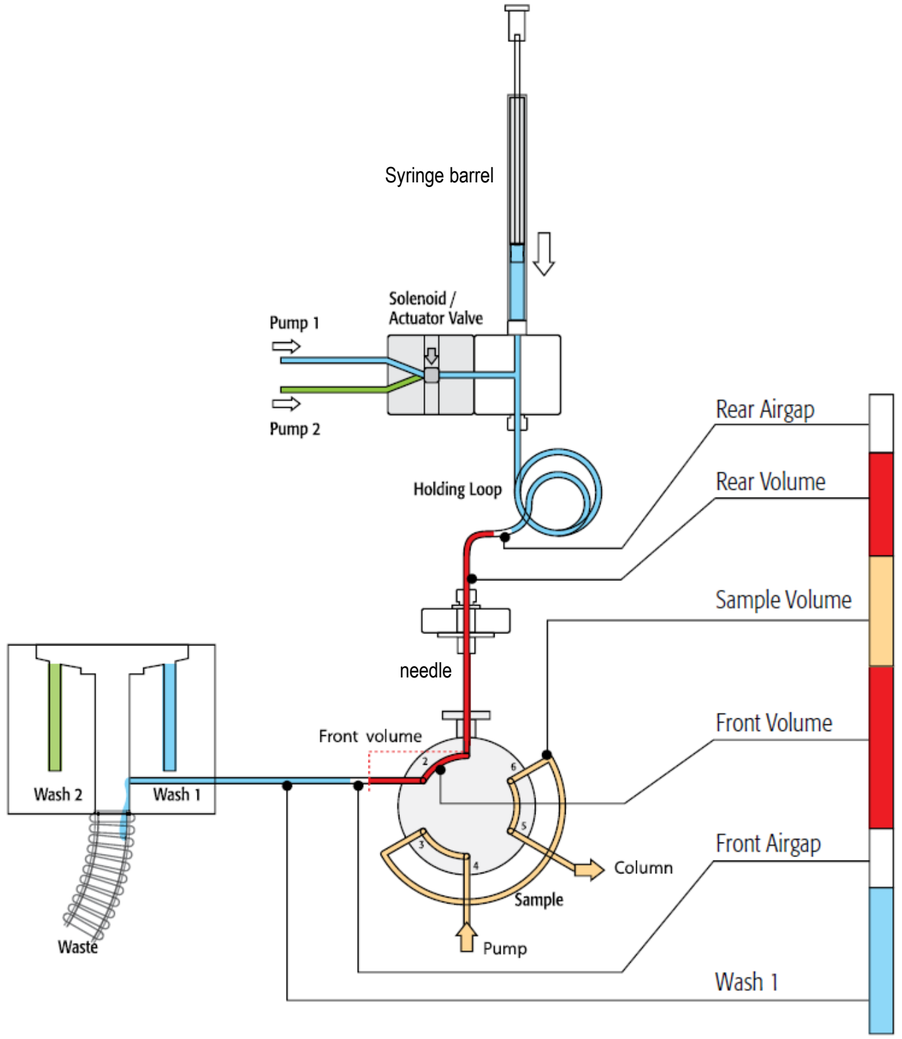
PFAS-free Fast Wash tubing kit

High recovery PP vials with conical bottom, with PFAS-free magnetic caps

µSPE cartridges with different sorbens for sample clean-up or analytes enrichment

Background Interference

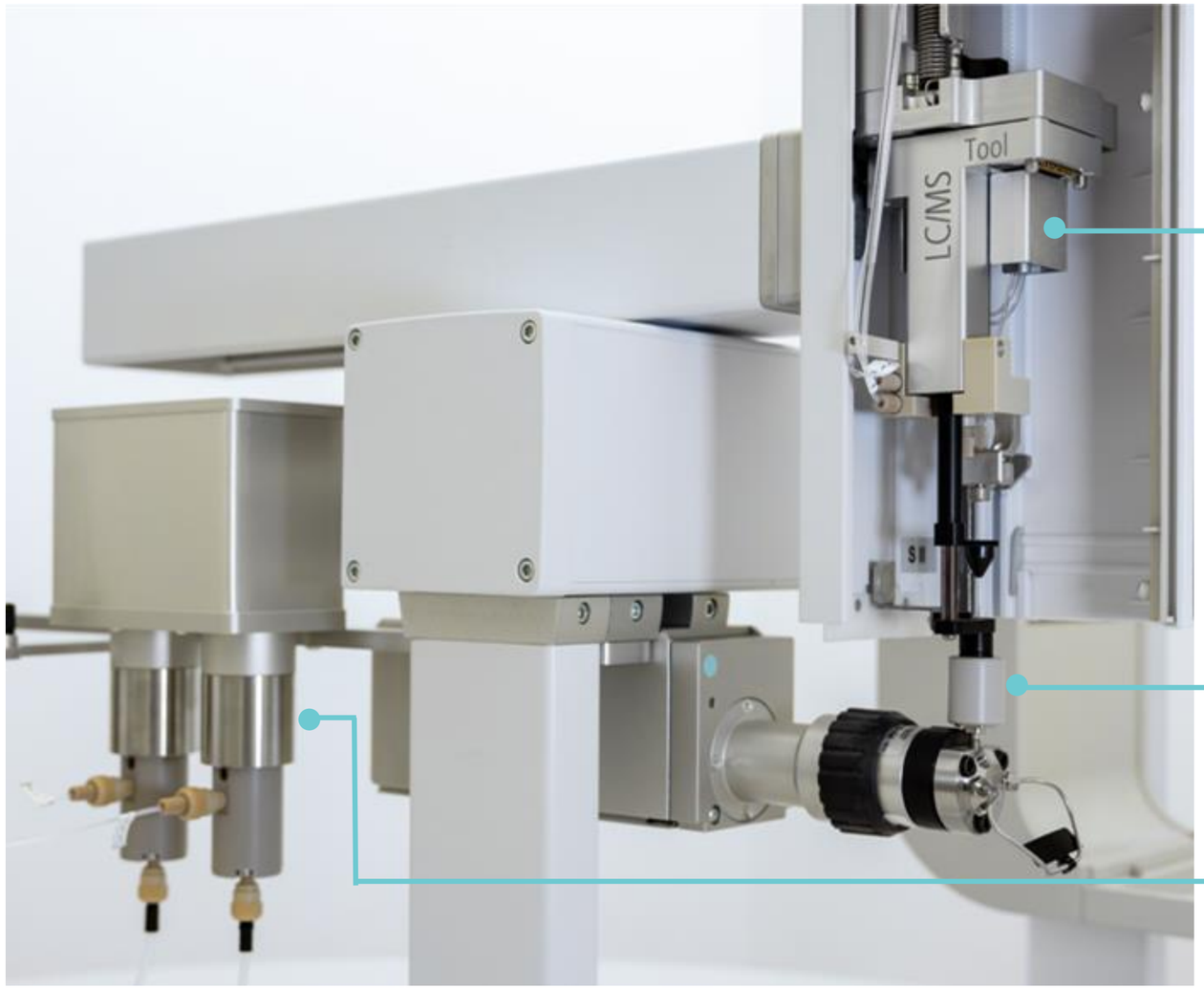
Minimize Carryover with the LC/MS Tool



- Sample does not go into the syringe, but through the holding loop
- Post-injection wash: Flush loop, needle inside and injection valve with wash solvent 2+1 → flush loop, needle inside & outside with wash solvent 2+1

Background Interference

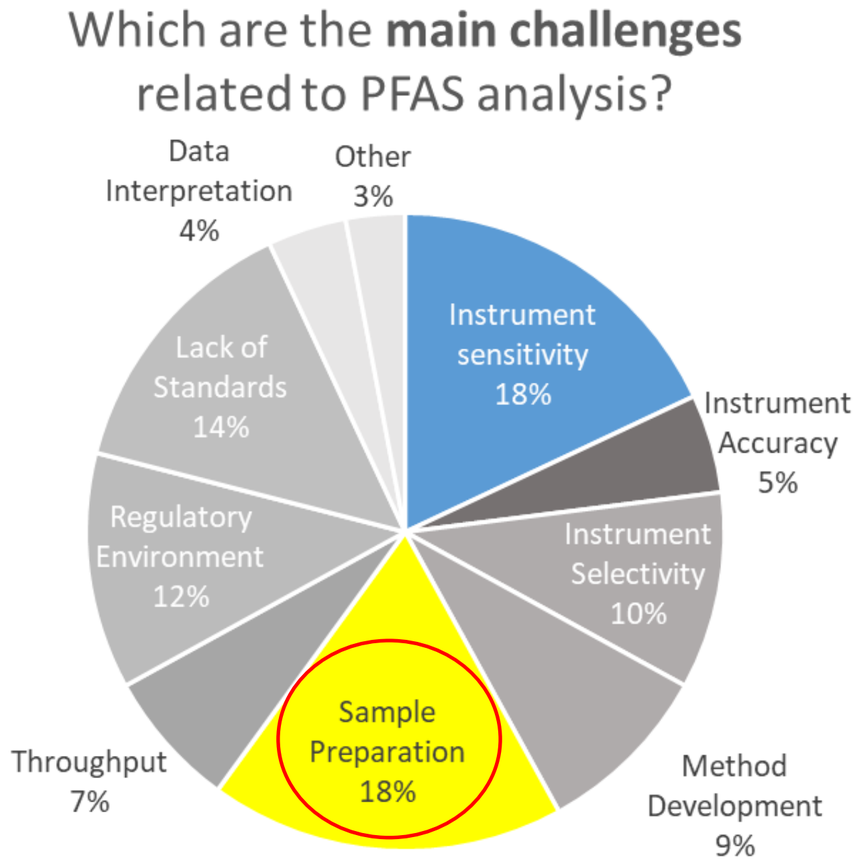
Minimize Carryover with the LC/MS Tool



- Volumetric pump connected to 2 wash solvents for post-injection wash
- Four different wash modes (high through-put, basic, extended & custom) available to suit different needs

What are the main challenges?

Community: Sample preparation is a focus area



Source: *Trends and Challenges in PFAS Analysis (theanalyticalscientist.com)*

Sample Preparation Options

Overview across PFAS analysis

Wee, S. Y., & Aris, A. Z. (2023). Revisiting the "forever chemicals", PFOA and PFOS exposure in drinking water. In npj Clean Water (Vol. 6, Issue 1). Nature Research. https://doi.org/10.1038/s41545-023-00274-6

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Biological		Extractions used			
Urine		SPE		LLE+SPE	
Plasma/ Serum		SPE	dSPE	LLE+SPE	LLE
Semen		SPE			LLE
CSF		SPE		LLE+SPE	LLE

Evironmental		Extractions used			
Water		SPE	Dilute and shoot	SPME	
Waste		SPE	Dilute/Filtrate		LLE
Air		SPE	Direct Headspace	SPME	ITEX
Soil		SPE			LLE

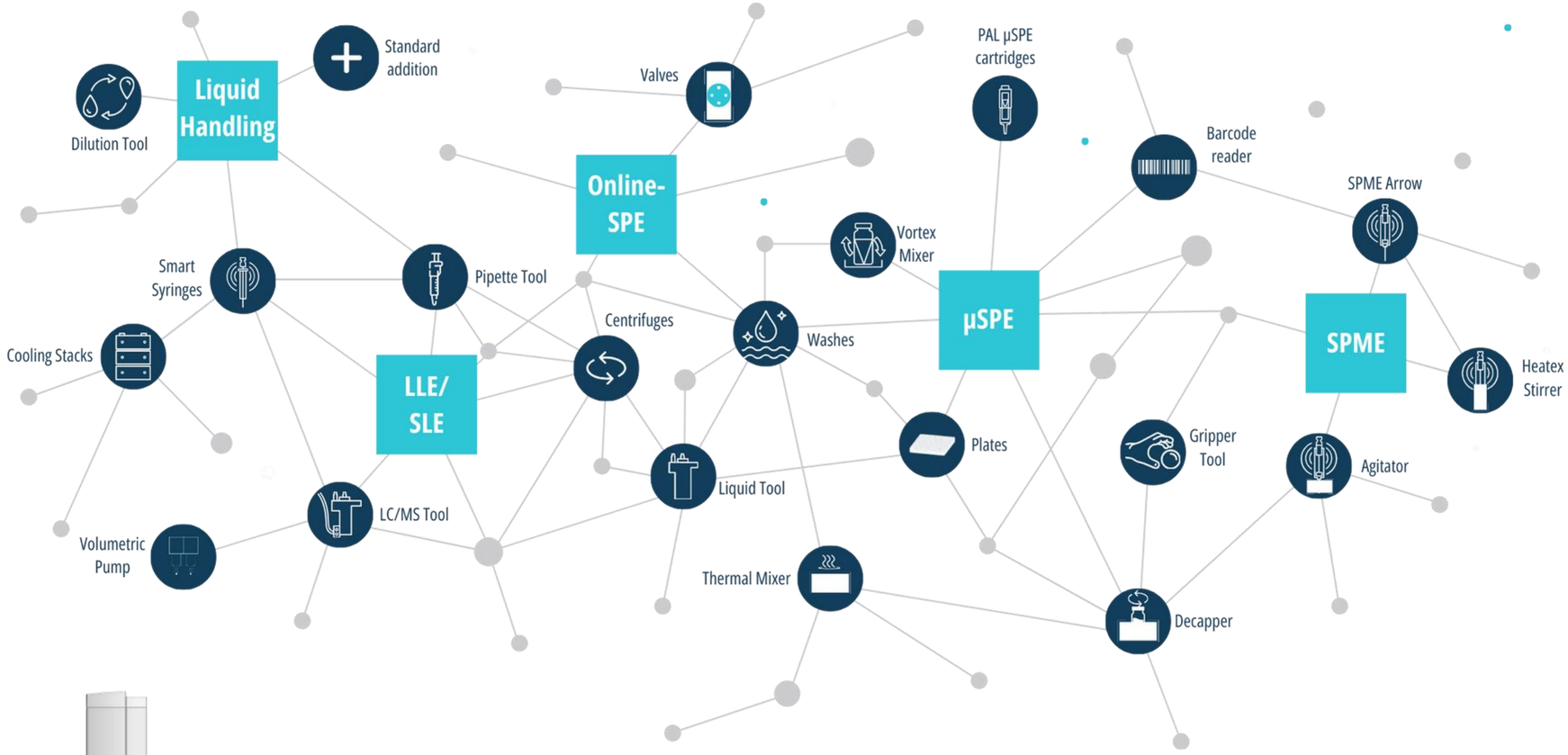
Food/Packaging		Extractions used	
Food		QuEChERS --> SPE	LLE

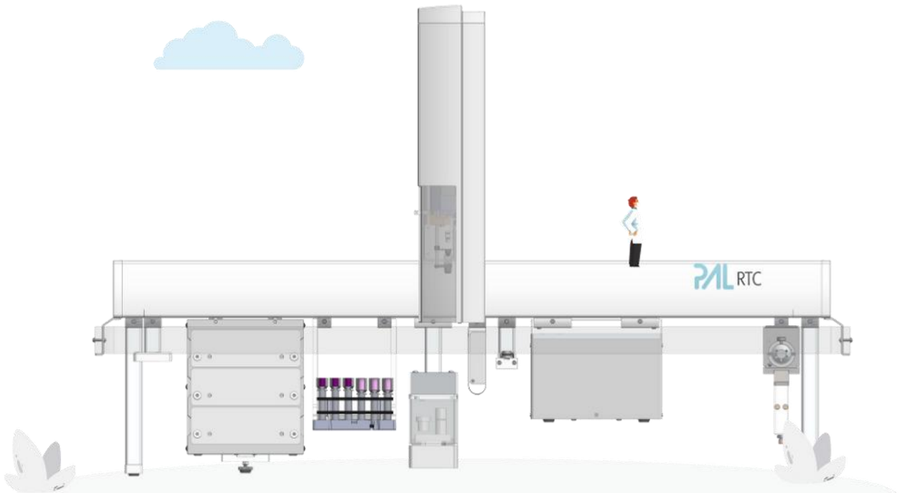
Abbreviations

Liquid-Liquid Extraction (**LLE**)
Solid-Liquid Extraction (**SLE**)
In-tube Extraction (**ITEX**)
Solid Phase Extraction (**SPE**)
Solid phase micro-extraction (**SPME**)
Dispersive SPE (**dSPE**)
Quick easy cheap effective rugged and safe extraction (**QuEChERS**)

PAL Toolbox for PFAS Analysis

PAL SYSTEM
Ingenious sample handling





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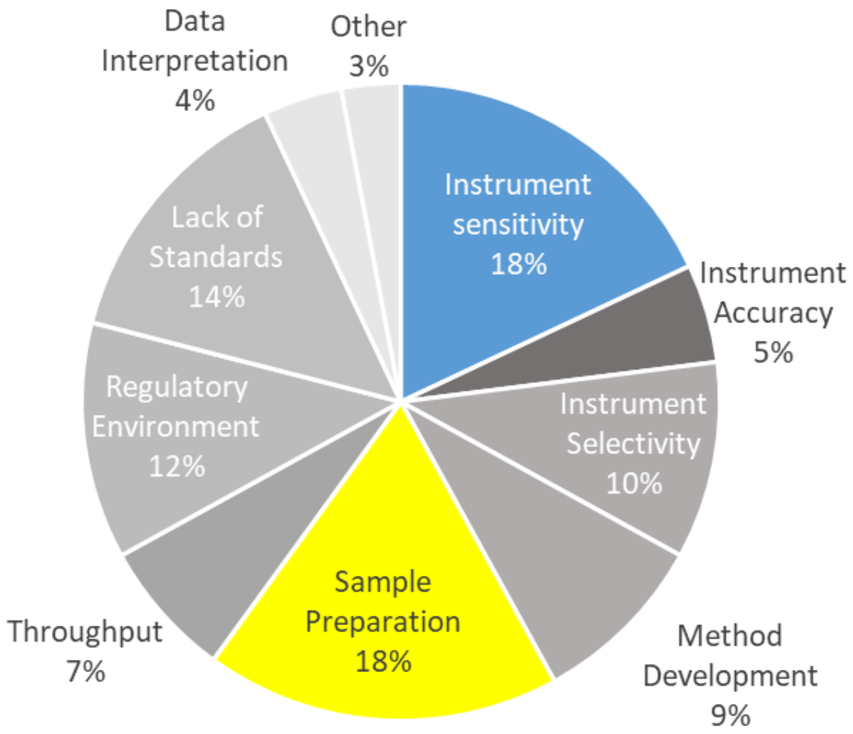
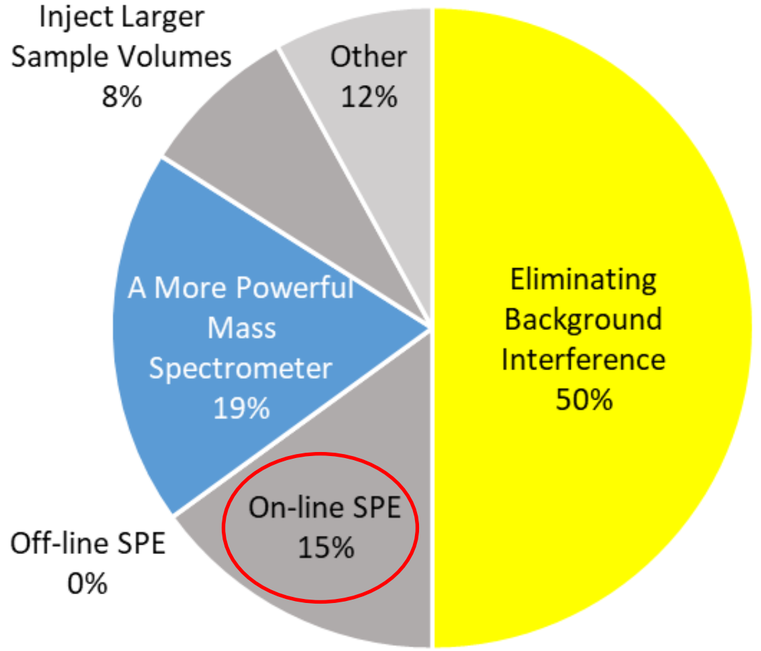
Challenges in PFAS Analysis

Focus on Sample Preparation: Online SPE

Which are the **main challenges** related to PFAS analysis?



Which of the following options are the **most desirable for improving sensitivity**?

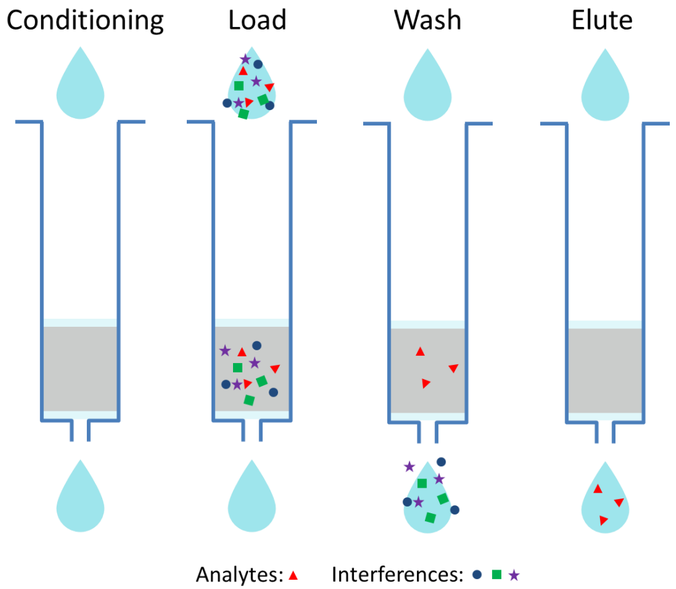


Source: *Trends and Challenges in PFAS Analysis (theanalyticalscientist.com)*

Deep Dive: Online SPE for PFAS in Water

What is Online SPE?

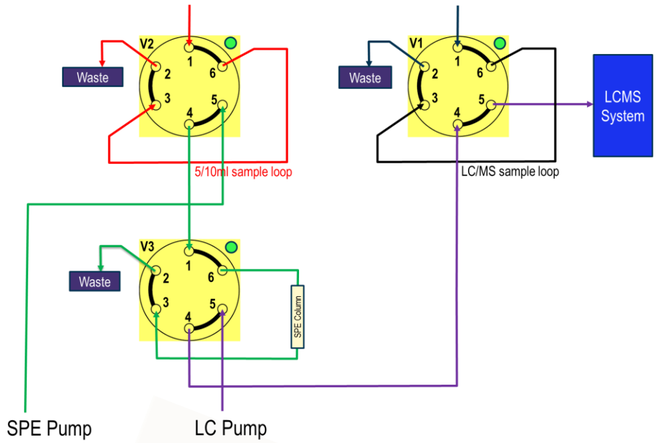
Offline SPE

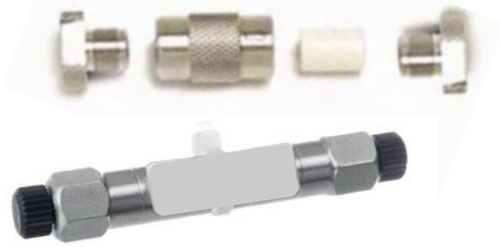


Offline SPE	Online SPE
Disposable SPE cartridges with different sorbents	SPE columns with typically 1-4.6mm id, 10-50mm length, with different packing materials
Manual multi-step sample handling	Automated sample handling
High consumption of sample and solvents	Low consumption of sample and solvents
Low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

Online SPE

Use automated valves switching to load, wash and elute samples





Deep Dive: Online SPE for PFAS in Water

Multiple Valves

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

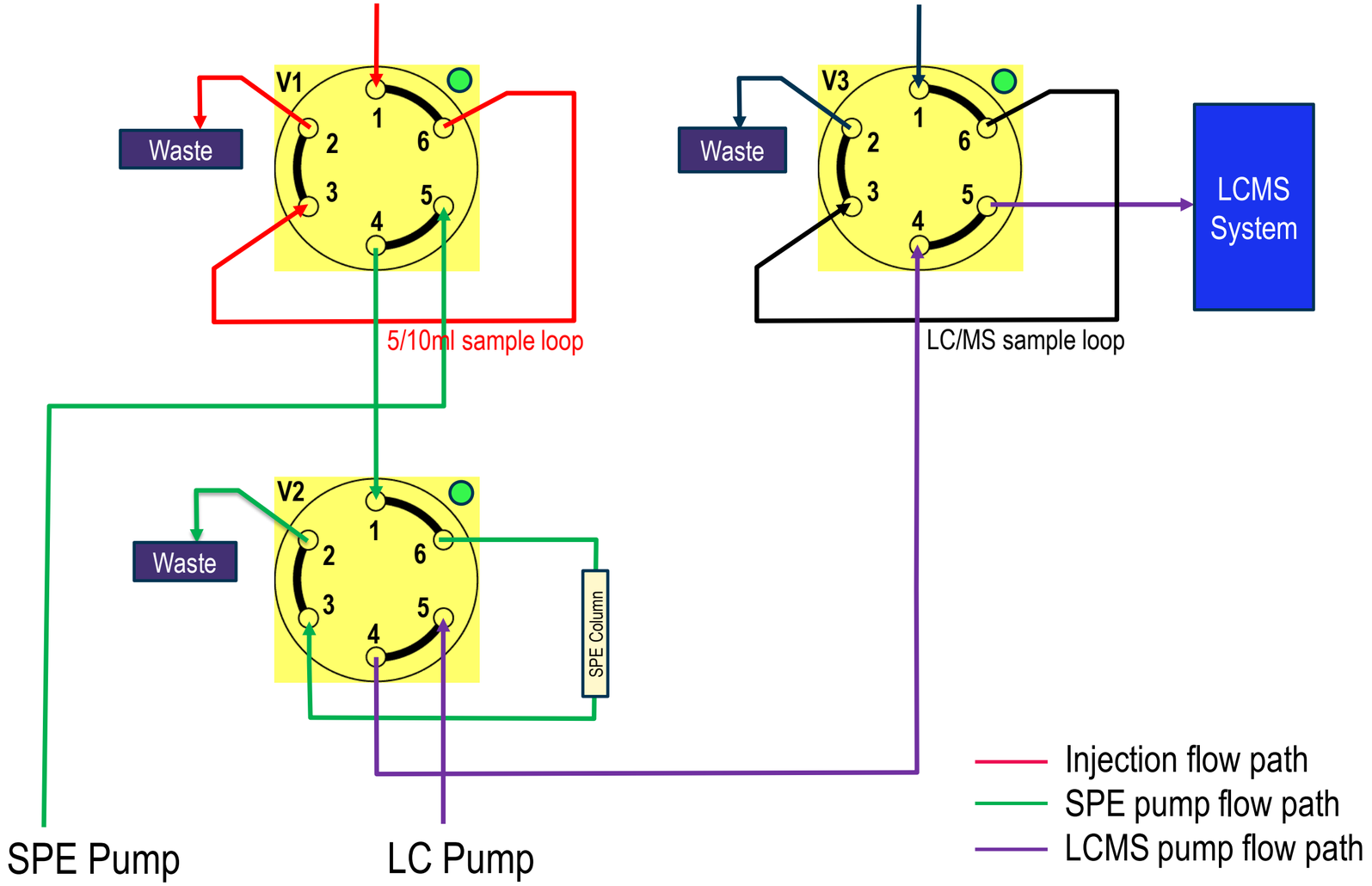
+ Dilutor or Liquid Tool with large Syringe (for large volume injection)

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

Liquid tool with 5ml / 10ml syringe

Deep Dive: Online SPE for PFAS in Water

Online SPE principle

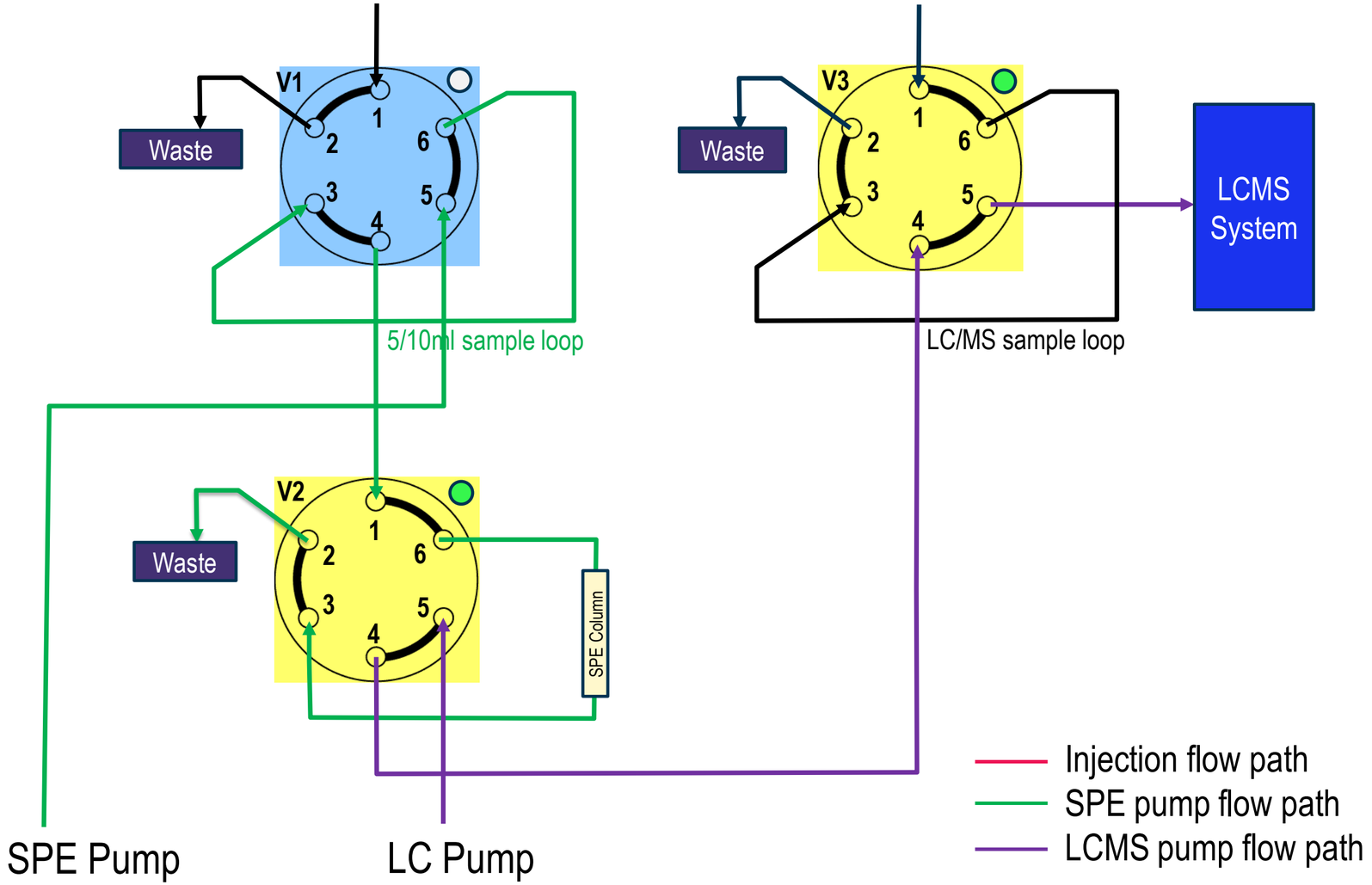


Loop Injection

Inject sample into valve loop

Deep Dive: Online SPE for PFAS in Water

Online SPE principle

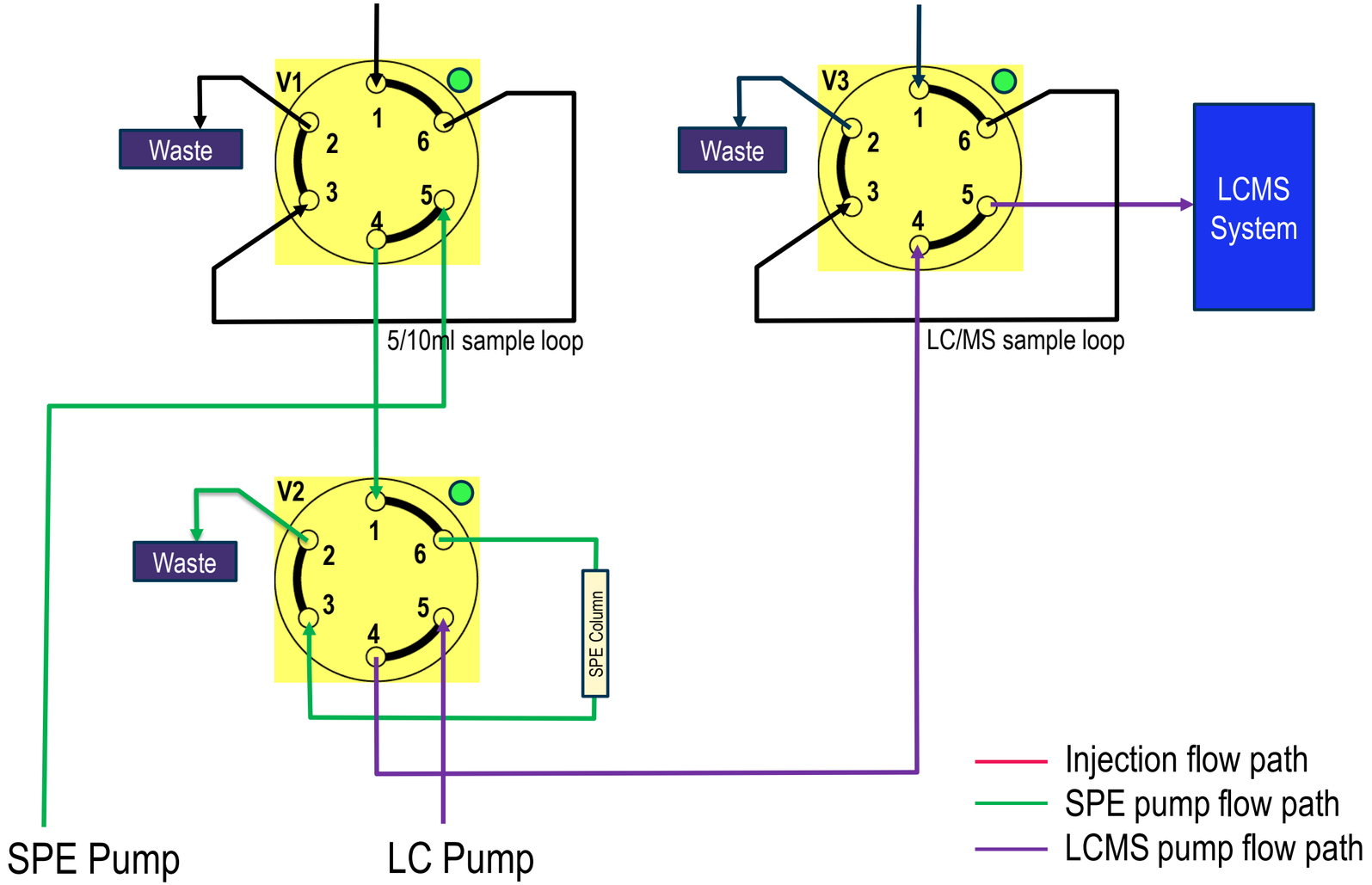


Injection Load

Load sample onto SPE

Deep Dive: Online SPE for PFAS in Water

Online SPE principle

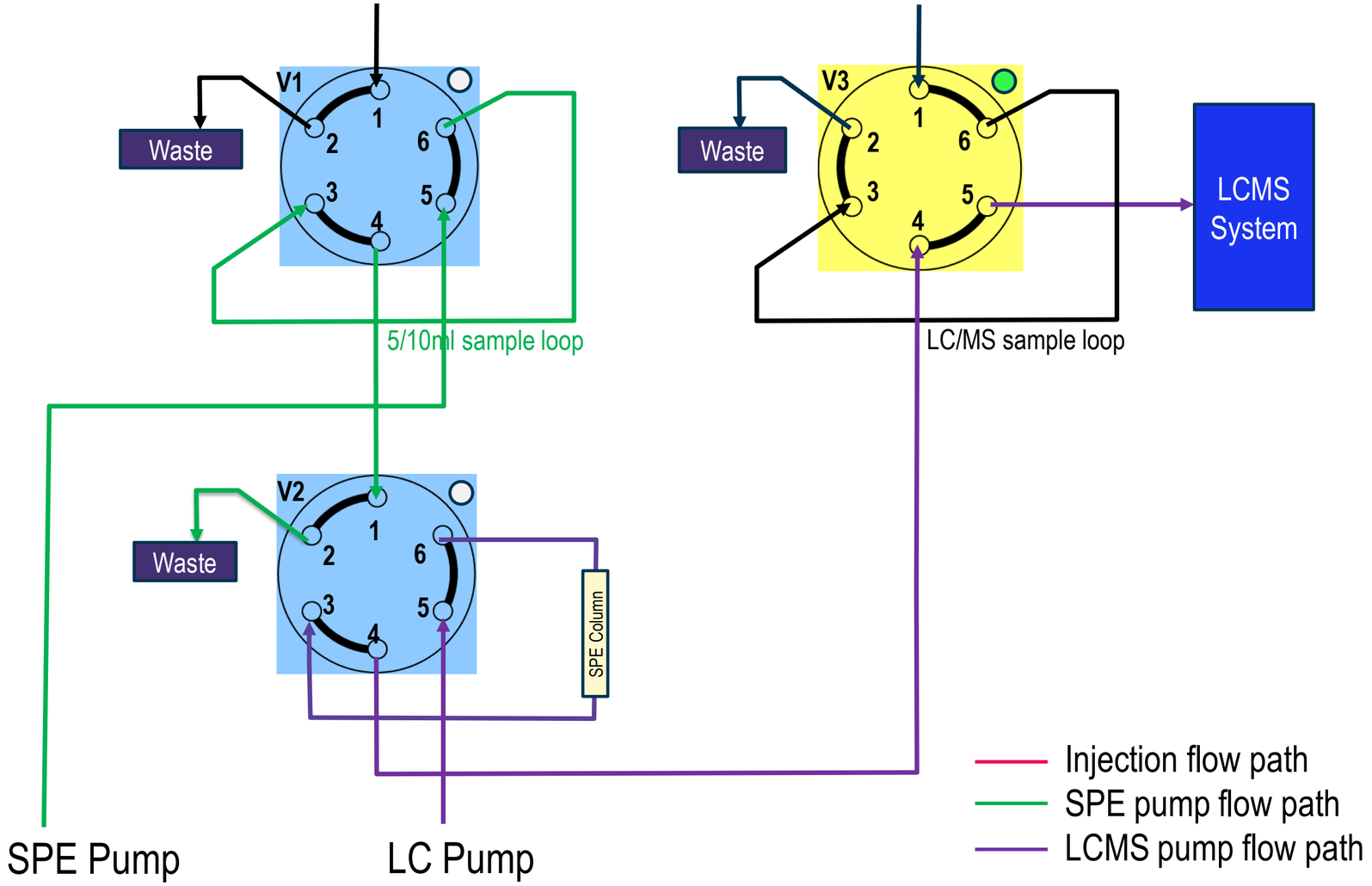


Sample Wash

Wash sample with a gradient by the SPE pump

Deep Dive: Online SPE for PFAS in Water

Online SPE principle

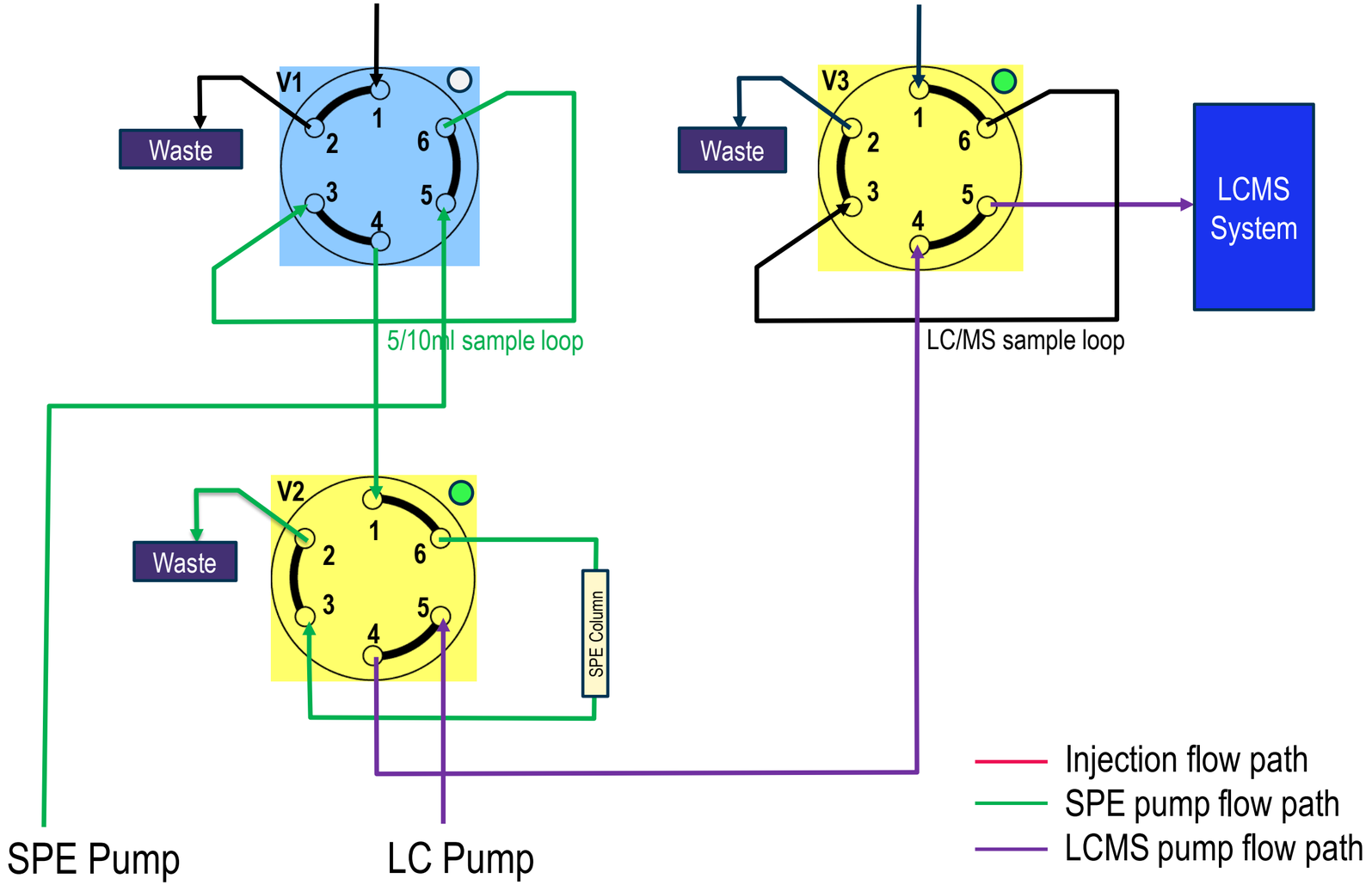


Elution onto analytical column

Elute sample into LCMS with a gradient by the LC pump

Deep Dive: Online SPE for PFAS in Water

Online SPE principle

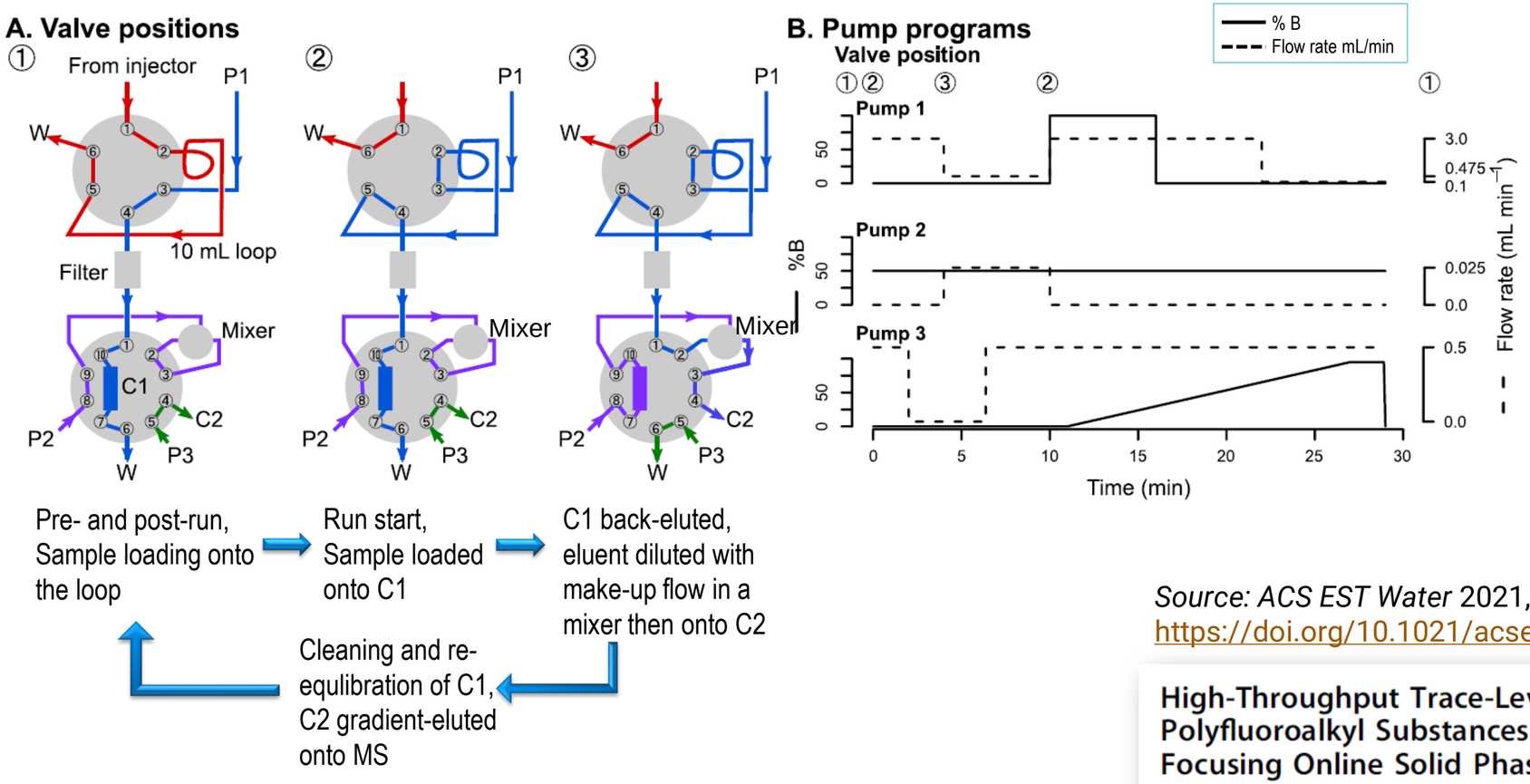


Pre-/Post-Elution Position

Optional valve cleaning

Deep Dive: Online SPE for PFAS in Water

Peak-focusing Approach Involving a 10-Port Valve



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

***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

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

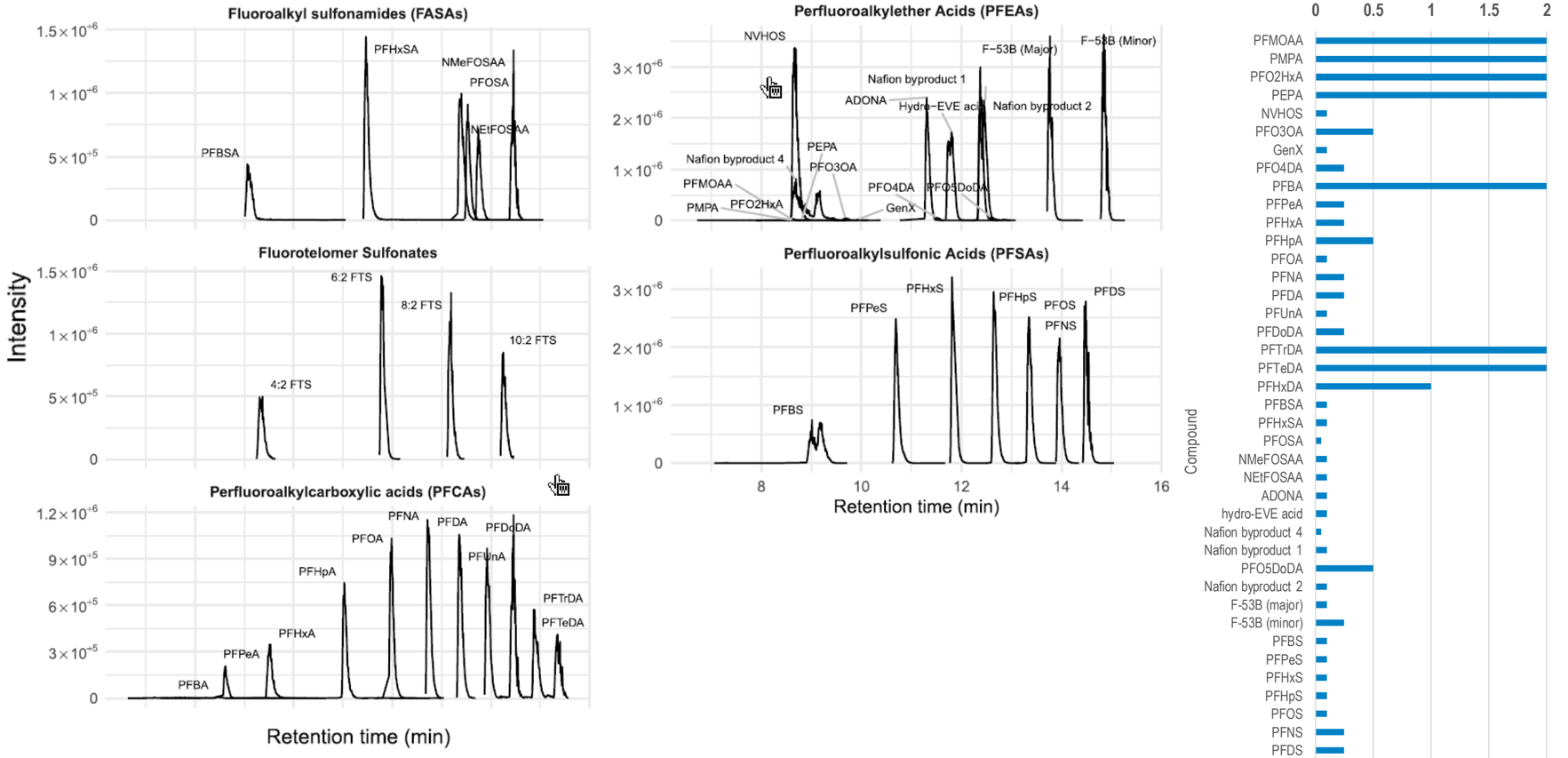

High-Throughput Trace-Level Suspect Screening for Per- and Polyfluoroalkyl Substances in Environmental Waters by Peak-Focusing Online Solid Phase Extraction and High-Resolution Mass Spectrometry

Gordon J. Getzinger* and P. Lee Ferguson*

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

Deep Dive: Online SPE for PFAS in Water

Peak-focusing Approach Involving a 10-Port Valve - 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.

Deep Dive: Online SPE for PFAS in Water

Online SPE

VS

Offline SPE

Benefits

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

Deep Dive: Online SPE → Micro-SPE

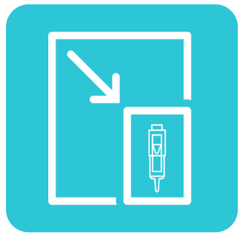
Following Green Analytical Chemistry

Green
Analytical
Chemistry

Waste Reduction
Minimizing solvent use and sample size (amount).

Resource Conservation
Lower energy consumption and reduced reliance on hazardous reagents.

Enhanced Safety
Reducing exposure of analysts to harmful chemicals.



PAL µSPE cartridges

Miniaturized: Designed for efficiency and reduced solvent consumption.

Automated: Seamless integration with PAL System for streamlined workflows.

High-Throughput: Ideal for processing large numbers of samples.

Versatile: Suitable for various matrices, including water, soil, and biological samples.

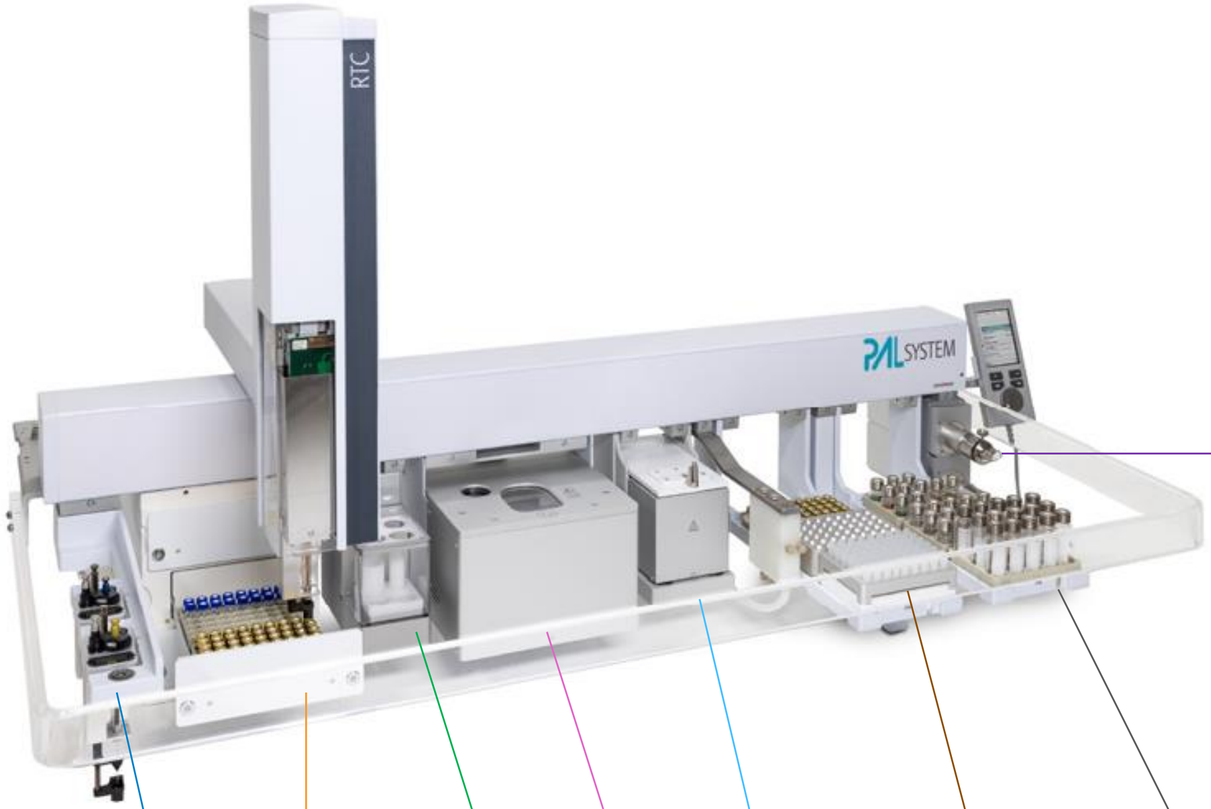
Deep Dive: µSPE for PFAS in soil

Overview

Manual

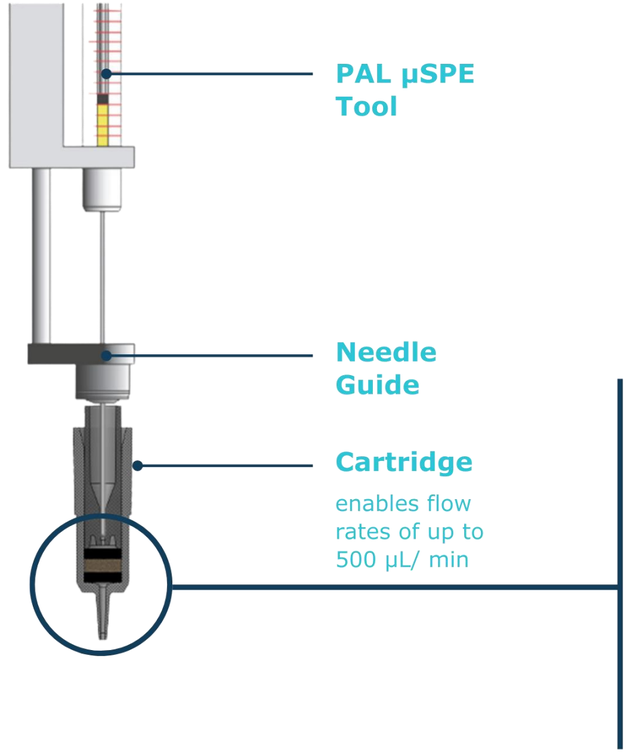
Automated by PAL

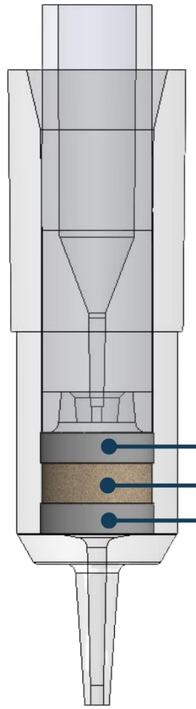
- Weigh samples into vial
- Add spiked solution with isotopically labeled standards
- Add extraction solvent (basic MeOH)
- Extraction of analytes
- Centrifuge/Filtration of supertanant
- Clean-up with Carbon SPE
- WAX SPE
- Acidification and dilution of sample
- LC/MS analysis



Deep Dive: µSPE for PFAS in soil

µSPE cartridge and workflow





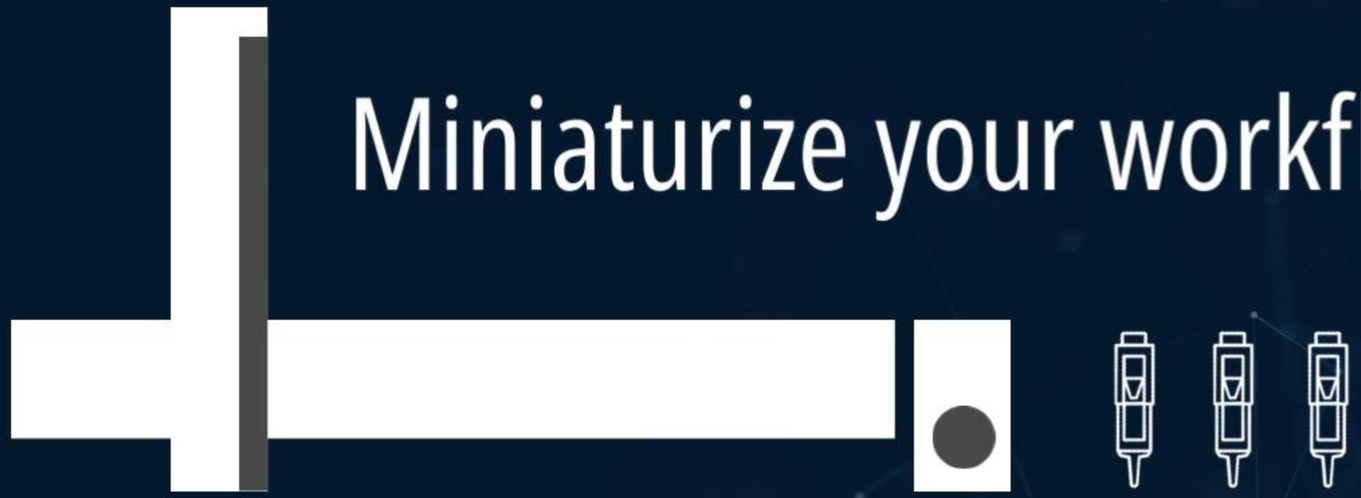
The septumless µSPE mini-cartridge design eliminates the seal leakage issues of other vendors

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

Filter
Sorbent
Filter

bed masses can be increased to up to 100 mg

Miniaturize your workflow



Applying µSPE in your automated QuEChERS clean-up

Keywords: Micro-SPE, GCMS, LCMS, QuEChERS, Automation

PAL SYSTEM

Take Home Message

PFAS Research and Regulation

- Health risks are recognized but more research needed
- Limits and regulations are still evolving

PFAS Analysis

- Workflows need to address broad range of sample types
- Focus on sample preparation and streamlining
- Automation reduces variation and interferences as well as noise

Online SPE

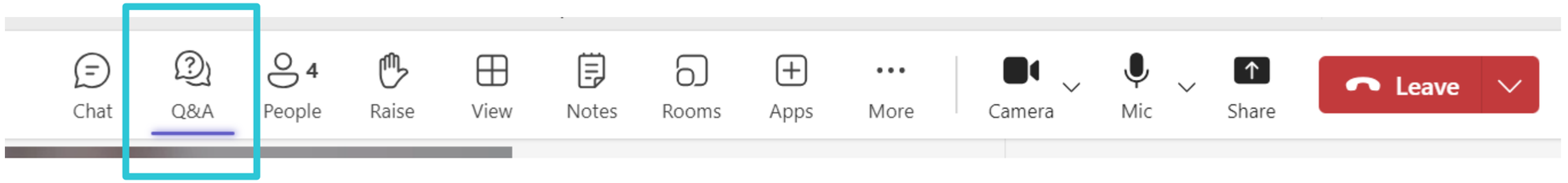
- SPE most used across sample types
- Increasing throughput and reducing risks of contamination

µSPE

- WAX cartridge is planned and will be available soon
- µSPE scripts and App Notes are in the content hub

Q & A

Ask your question in the Q & A Window



Ask your question in the **Q & A window** right now

Use the form linked in the pinned message

Send us an email **info@palsystem.com**


PAL SYSTEM
Ingenious sample handling

Global PFAS Regulations

Method	Matrix Tested	No. of Analytes	Sample Prep Procedure
EPA 537	Drinking water	14	SPE
EPA 537.1	Drinking water	18	SPE
EPA 533	Drinking water	29	SPE
EPA 1633 (draft)	wastewater, surface water, groundwater, soil, biosolids, sediment, landfill leachate, and fish tissue	40	SPE
EPA 8327 (draft)	Surface water, ground water, wastewater influent and effluent	24	Dilute & shoot
ASTM D7979	Surface water, ground water, wastewater influent and effluent	21	Dilute & shoot
ASTM D7968	Soil and solids	21	Organic extraction with MeOH
ISO/DIS 21675 (draft)	Drinking water, sea water, fresh water, wastewater	30	SPE

And more..
EPA 1621
EU Directive 2020/2184
ISO 25101:2009
ISO 21675:2019
DIN 38407-42:2011
Istisan 19/07

Regulations are still evolving:

Number of analytes ⇧

Concentration limits ⇩

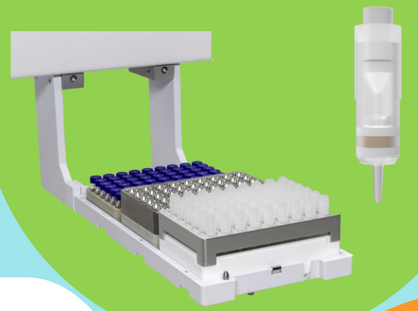
PAL Toolbox for Sample Prep Automation for PFAS Analysis

PAL SYSTEM
Ingenious sample handling

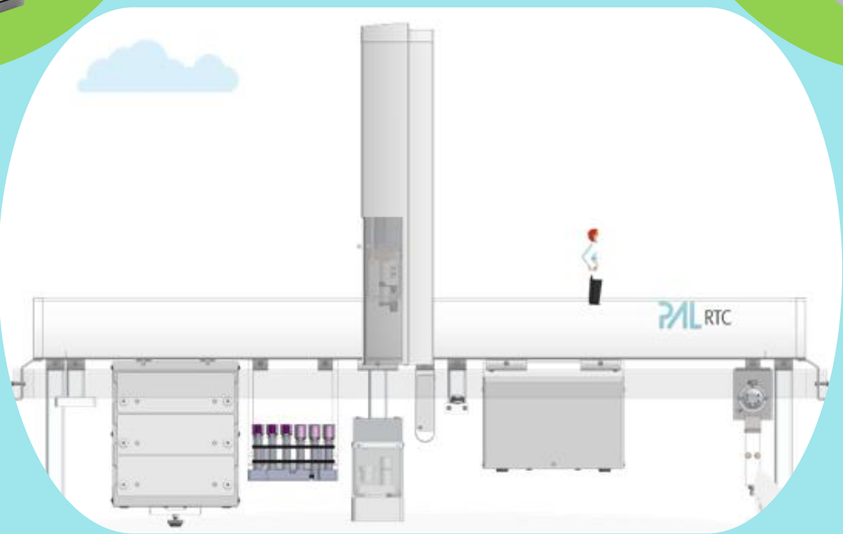
Online SPE

LLE; SLE

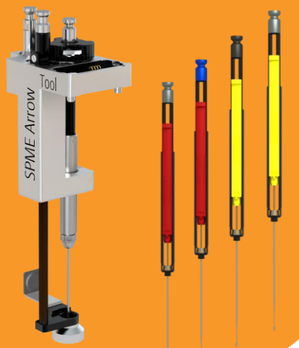
µSPE; QuEChERS;
Filtration



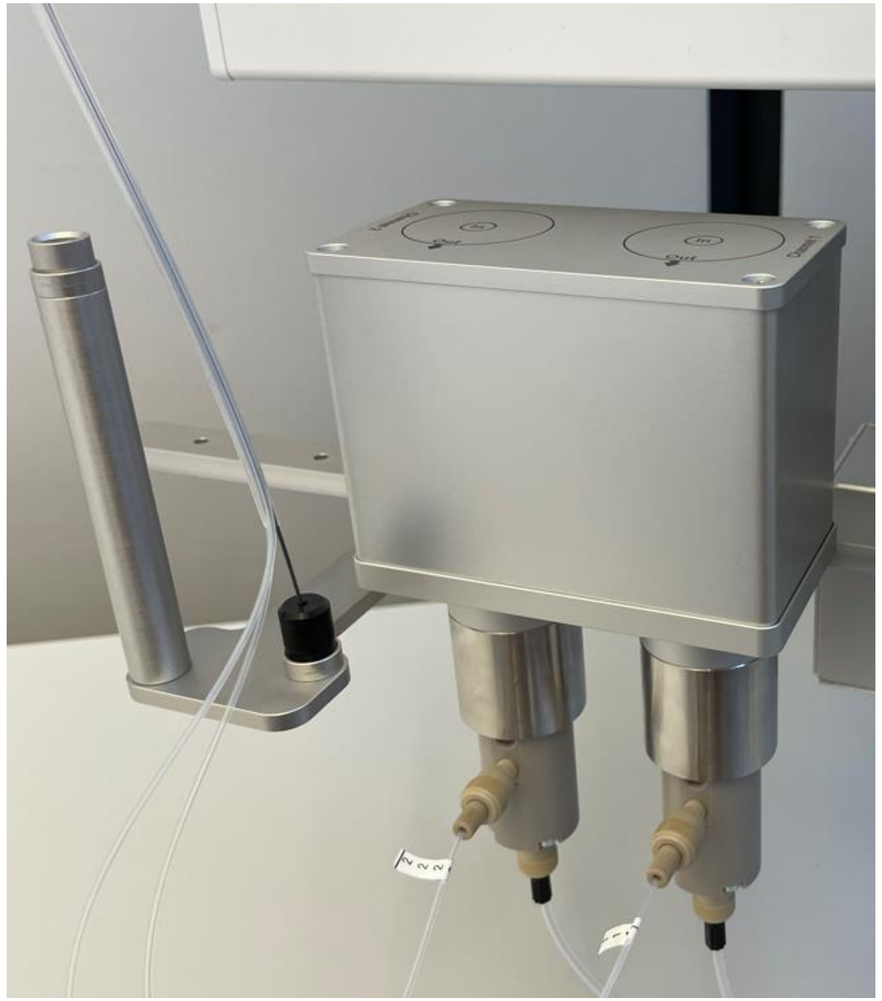
Dilution;
Addition of standards



SPME



2-Channel Volumetric Pump



Features

- Precise and reproducible flow rate
- Less wash solvent needed
- Robustness leading to long lifetime
- Low maintenance

Specifications

- Pump flow rate: 1-250 µL/s
- Pressure rating: 50 bar
- Service intervals (seal replacement of pump): >280k samples

Applications

- Pump for LCMS Tool
- Syringe pump
- Online SPE

Requires FW 4.12 or higher

Reduce Background Contamination

With PFAS-Free Tubings and Kits

PFAS-free Tubings

Tubing for connection of LC/MS Tool

Solvent inlet kit incl. tubing, filter and cap

Tubing for connection of Dilutor Tool

Tubing for HF-Fast Wash module

Tubing for HF-Fast Wash and Fast Wash module

Sample loop

Wash Cup

Upgrade Kits for:

- LCMS tool
- Dilutor
- Fast Wash Station
- HF-Fast Wash Station
- Wash Cup

(Components are also available seprately)

Reduce Background Contamination - Continued

With PFAS-free Consumables

To be released:

PFAS-free Diluter Syringes
- 5ml and 10ml
- PE plunger and insert

PFAS-free Injection Syringes
- 100ul, 1ml and 5ml

uSPE cartridge with WAX sorbent

PFAS-free vials and caps
- 2ml / 8ml / 18ml
- High-recovery PP vials
- PFAS-free caps (with septa)

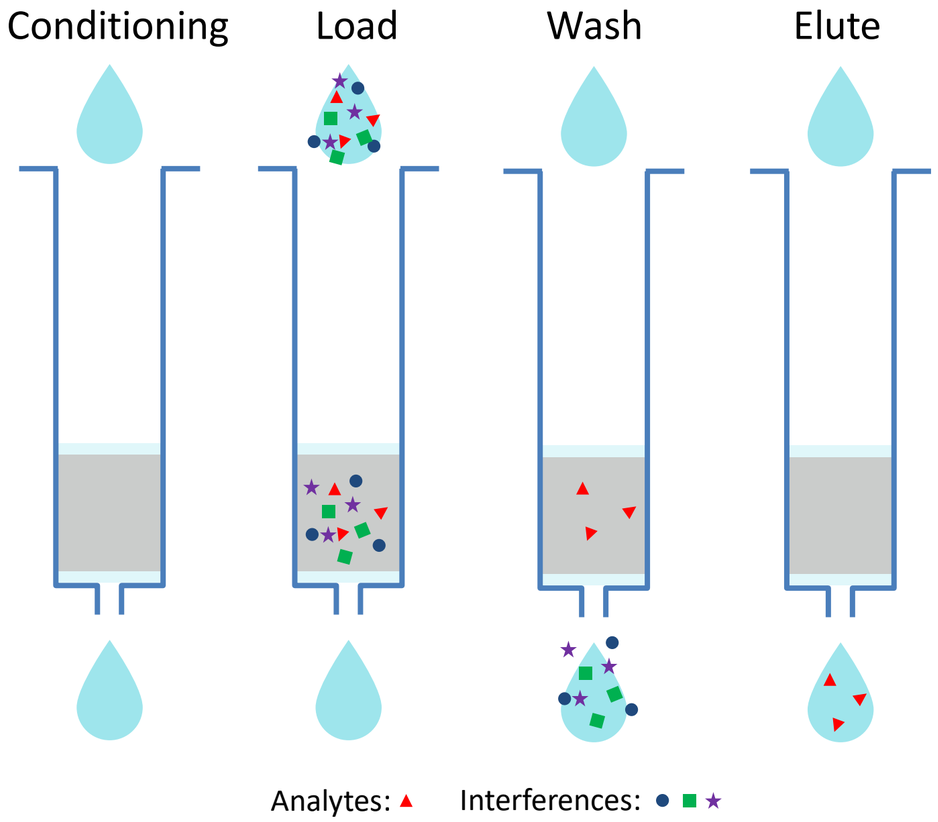
Dilutor syringes

Injection syringes

uSPE cartridges

Magnetic vial caps with PFAS-free septa (silicon with polyimide foil), pre-slitted

High recovery PP vials with conical bottom, with magnetic caps for vial transport


Conditioning
Load
Wash
Elute
Analytes:
Interferences:

LC/MS Setup with Volumetric Pump & Wash Cup

PAL SYSTEM
Ingenious sample handling

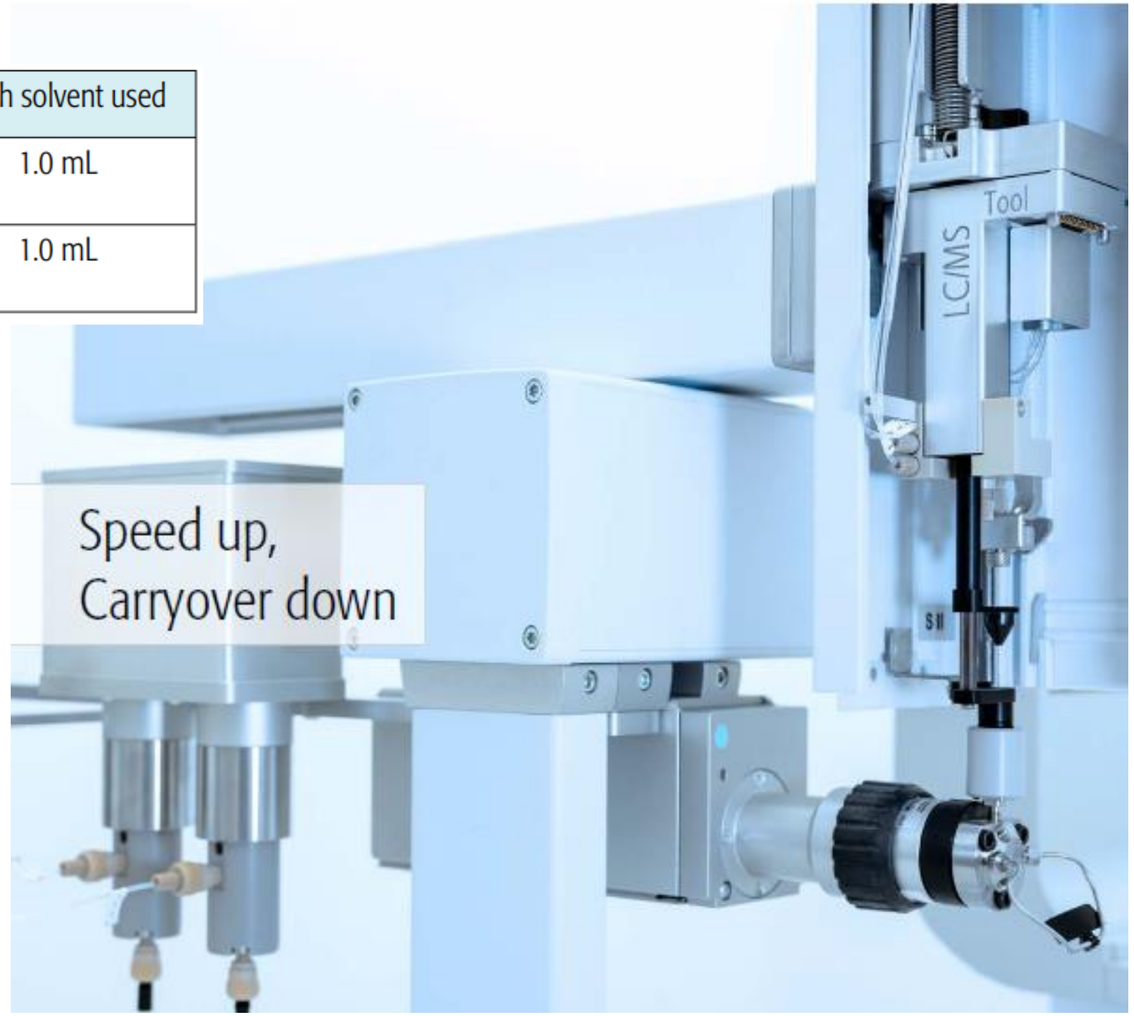
High throughput at low cost per sample:

LCMS script	Carry-over [ppm]	Cycle time [s][1]	Start to stop time [s] [2]	Wash solvent used
High-Throughput*	26	48	46	1.0 mL
High-Throughput DHR*	26	24	45	1.0 mL

* with LC/MS Pump, Wash Cup without backflush pump

1 Time definition: batch start to stop time divided by n samples

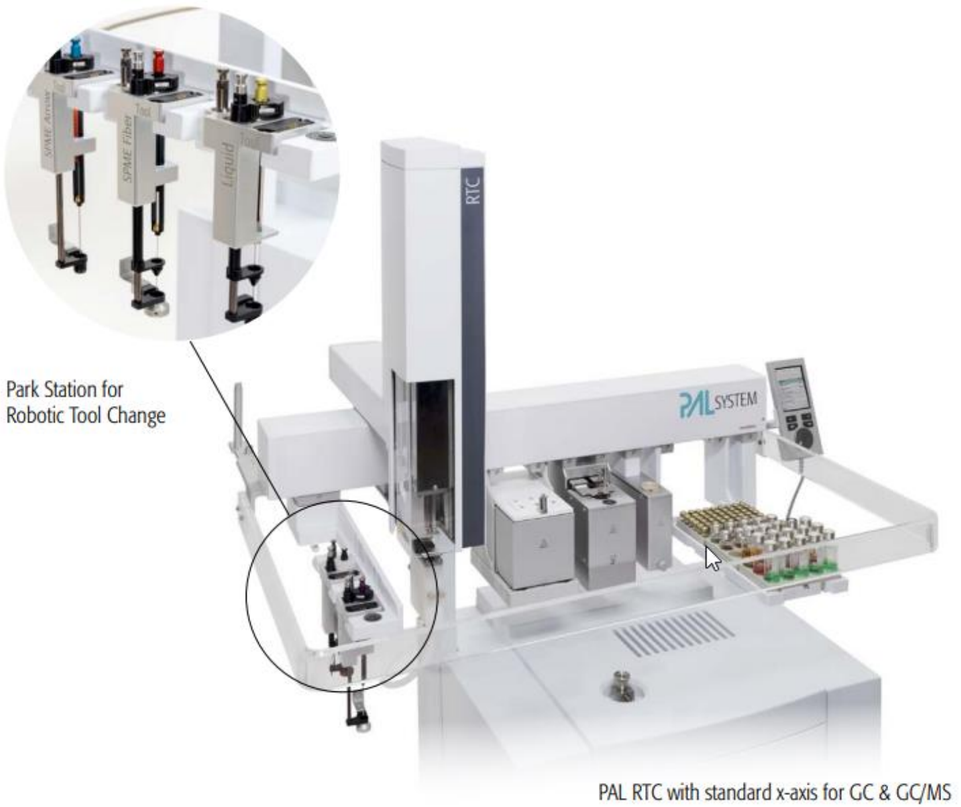
2 time definition: single sample start to stop time



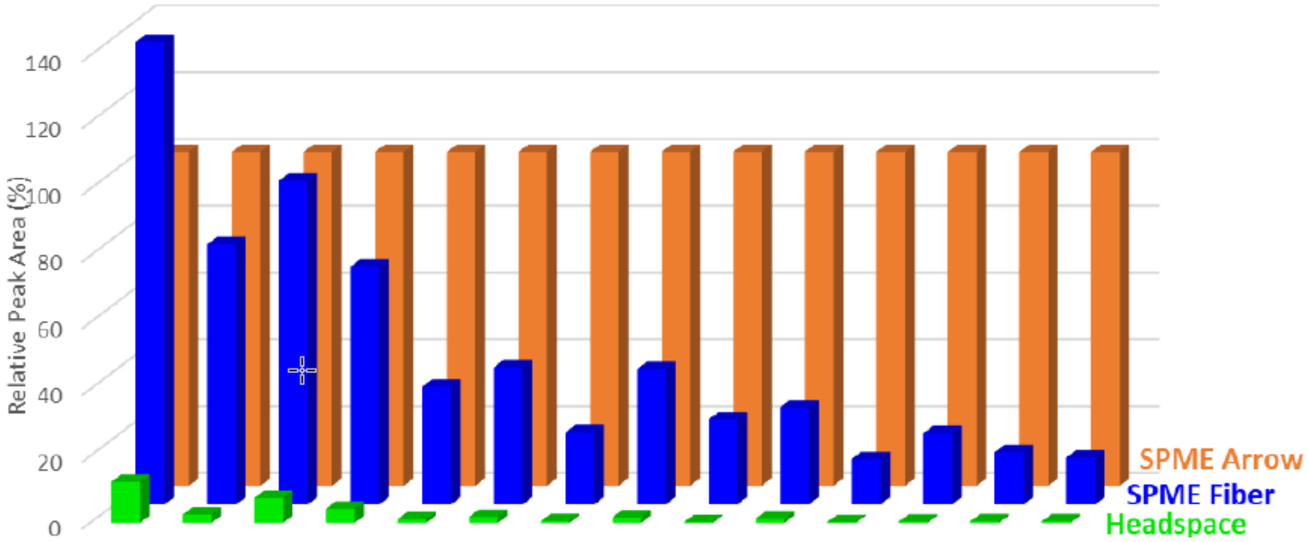
Low service cost leads to low cost per sample

Low cost per sample: Operating cost is a key criterion for commercial labs. The reliable PAL System offers low maintenance because of the robust design . With a service interval of 280’000 samples the service cost/sample can be as low as 0.05 US $.

Analysis of Volatile PFAS with HS-SPME GC/MS



PAL RTC with standard x-axis for GC & GC/MS



- Show difference SPME, SPME Arrow