



Development of suitable methods for PFAS sample preparation in different matrices

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Dr. Suman Kharel

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Per- and polyfluoroalkyl substances (PFAS)

- Family of more than 6000 chemicals
- Persistent in environment: known as „Forever chemicals“
- Excellent water repellent (strong C-F bond)
- Ubiquitous (household, industry, automotive, medical, etc)
- Harmful (endocrine disruptors, carcinogens, affecting the immune system)



<https://paustenbachandassociates.com/services/pfoa-and-pfos/>

Challenges in PFAS Sample Preparation



Contamination

Parts of devices e.g. pump seals, tubing

Consumable, glassware, vials

Solvents

Gloves, clothing

Cosmetics

Laboratory air (e.g. air condition filters)

Low detections levels and background issues, sample throughput

Suitable and efficient SPE cartridges / matrix

Large sample volume

Instrument sensitivity

Suitable evaporation methods

Dedicated devices

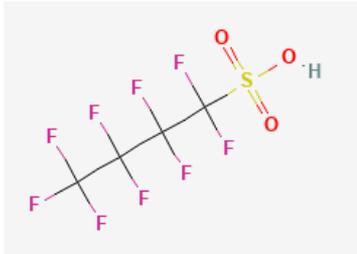
Challenges in PFAS Sample Preparation



Extended number of PFAS analytes, different matrices

Short chain

- PFBA
- PFBS



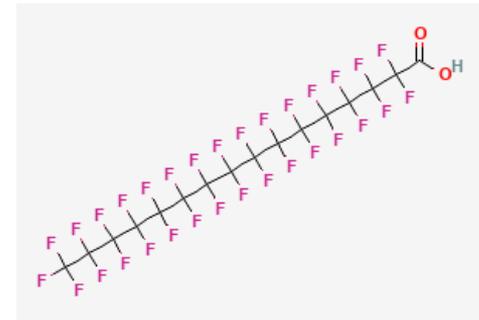
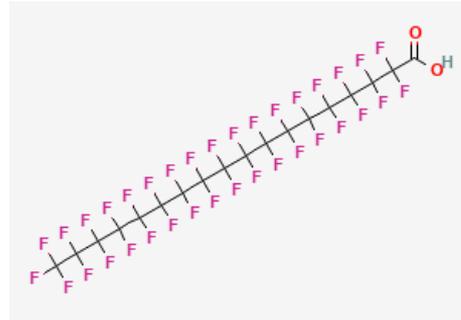
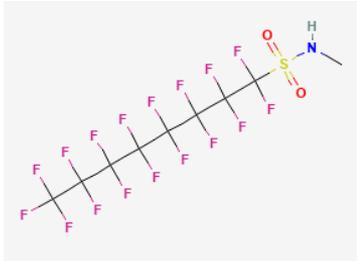
Longer chain

- > C12 carboxylic acids, sulfonic acids
- PFH_xDA (C16)
- PFODA (C18)

Suitable and efficient SPE cartridges

Volatile

- FOSA
- N-MeFOSA
- N-MeFOSE



PFAS Workflow for *Aqueous Samples*



FREESTYLE™ XANA PFAS

For clean-up of up to 4 L



or

FREESTYLE™ XANA PFAS TableTop

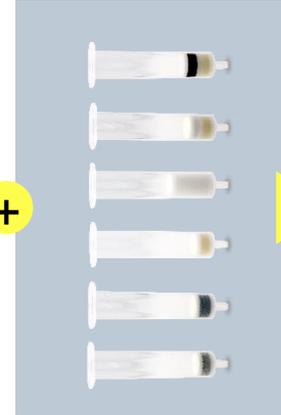
For clean-up of up to 250 mL



+

EluCLEAR® PFAS

SPE columns



D-EVA Concentration

For the sensor controlled evaporation to a few µL



PFAS Sample Prep: Think LCTech!
Excellence meets Experience



PFAS Workflow for *Solid Samples*

X-TRACTION® PFAS (PFE*) or MIX-TRACTION

For Extraction



D-EVA Concentration

For the sensor controlled
evaporation of extracts



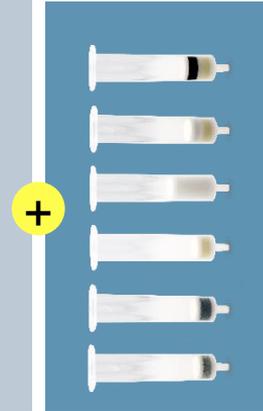
FREESTYLE™ SPE PFAS

For clean-up up to 50 mL
(e.g. Dual SPE)



EluCLEAN® PFAS

SPE columns



D-EVA Concentration

For the sensor controlled
evaporation to a few µL



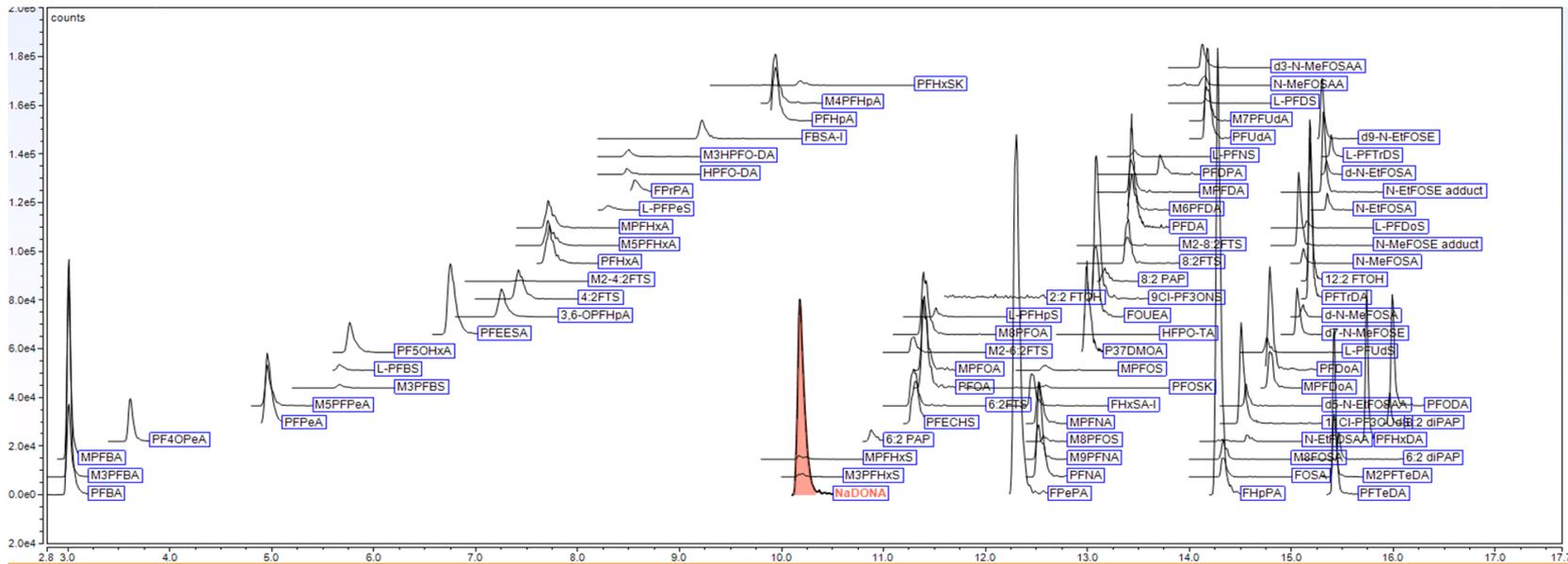
* Pressurized Fluid Extraction

PFAS Sample Prep: Think LCTech!
Excellence meets Experience

Regulation: Extended number of PFAS (53 analytes)



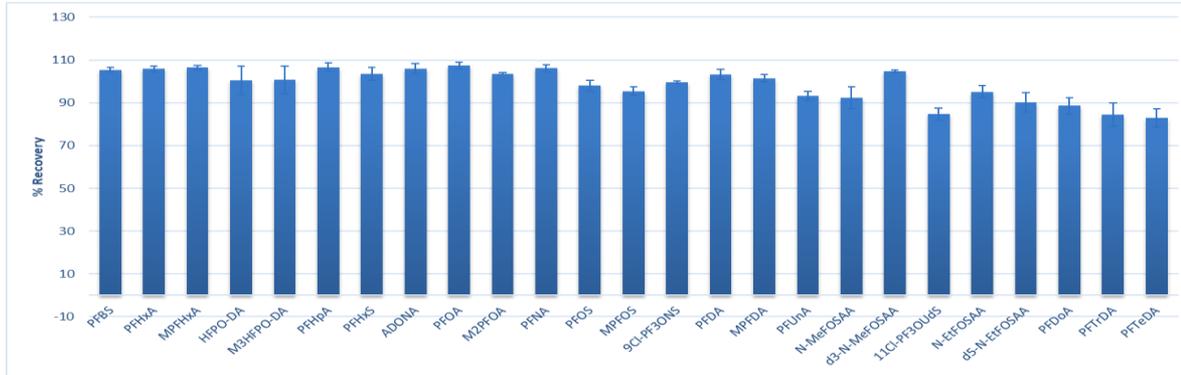
REGULATIONS	EPA 537.1	EPA 533	EPA 3rd DRAFT 1633	DoD/DOE QSM 5.3	DIN 38407-42 DIN 38414-14	ISO 21675-2019
MATRIX	Drinking Water	Drinking Water	Non-potable water, solids, biota	Non-potable water, solids, biota	Drinking water, ground water, surface water, treated wastewater	Drinking water, natural water, waste-water (< 2 g/L SPM)
COMPOUNDS	18	25	40	25	10	14





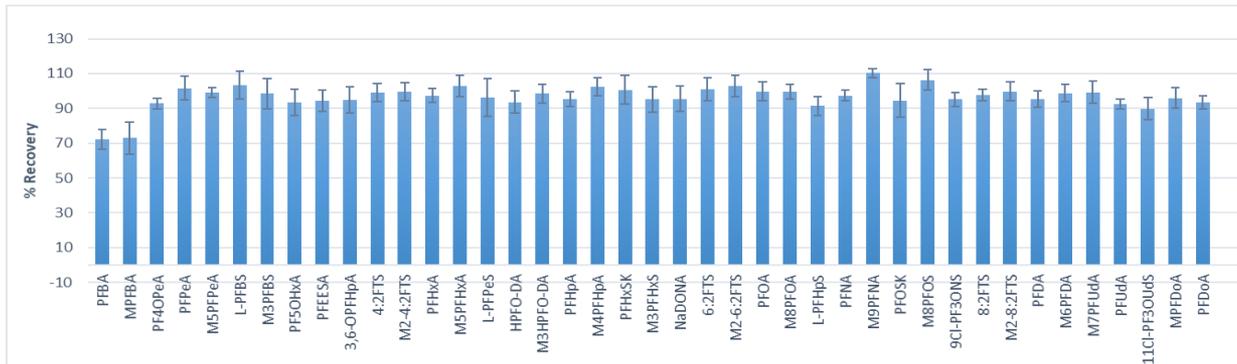
EluCLEAN® PFAS – WAX and SDVB

US EPA method 537.1 and US EPA method 533 – Tap Water



Tap water

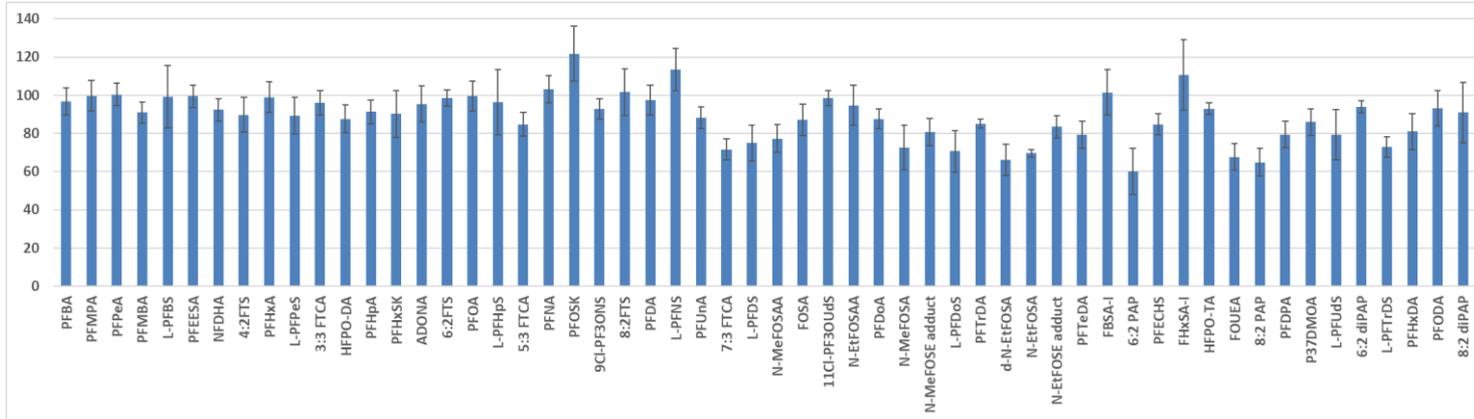
Recovery rates of 18 PFAS (listed in **US EPA method 537.1**) + 7 surrogates from drinking water with EluCLEAN® SDVB columns (n = 4, c = 40 ng/L)



Tap water

Recovery rates of 25 PFAS (listed in **US EPA method 533**) from drinking water with EluCLEAN® PFAS - WAX 500 mg columns (n = 4, c = 20 ng/L)

Tap Water with extended number of PFAS (53 analytes) with 1633 draft method

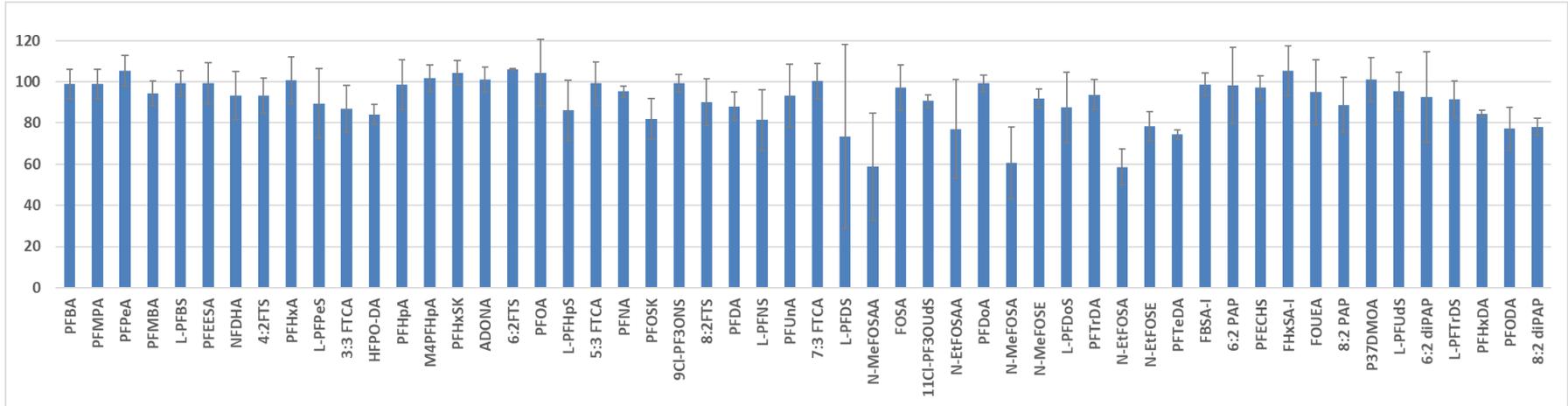


50 mL tap water spiked with the following absolut amounts, n=3

- 11CI-PF3OUdS, 9CI-PF3ONS , ADONA, HPFO-DA, NFDHA, PFEESA, PFMBA, PFMPA, PFPeA: each 2 ng
- PFBA, 4:2FTS, 6:2FTS, 8:2FTS: each 8 ng
- N-MeFOSE, N-EtFOSE: each 20 ng
- 5:3 FTCA, 7:3 FTCA: each 40 ng
- FBSA-I, PFECHS, FHXSA-I, P37DMOA, FOUEA, 6:2 diPAP, 8:2 diPAP : each 3.35 ng
- PFHxDA, PFODA: each 1.667 ng
- L-PFUdS, L-PFTrDS: each 6.667 ng
- 6:2 PAP, 8:2 PAP, PFDPa : each 16.67 ng
- All other PFAS: each 0.325 ng



Tap Water with extended number of PFAS (53 analytes), 1633 draft method



50 mL tap water spiked with the following absolute amounts, n=3

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Proposed PFAS National Primary Drinking Water Regulation



On March 14, 2023, the Environmental Protection Agency (EPA) announced the proposed National Primary Drinking Water Regulation (NPDWR) for six PFAS, including perfluorooctanoic acid (PFOA), perfluorooctane sulfonic acid (PFOS), perfluorononanoic acid (PFNA), hexafluoropropylene oxide dimer acid (HFPO-DA, commonly known as GenX Chemicals), perfluorohexane sulfonic acid (PFHxS), and perfluorobutane sulfonic acid (PFBS). The proposed PFAS NPDWR mandates testing of drinking water and environmental samples for PFAS to minimize the risk of health problems.

LC Tech: SPE COLUMNS for PFAS ANALYSIS

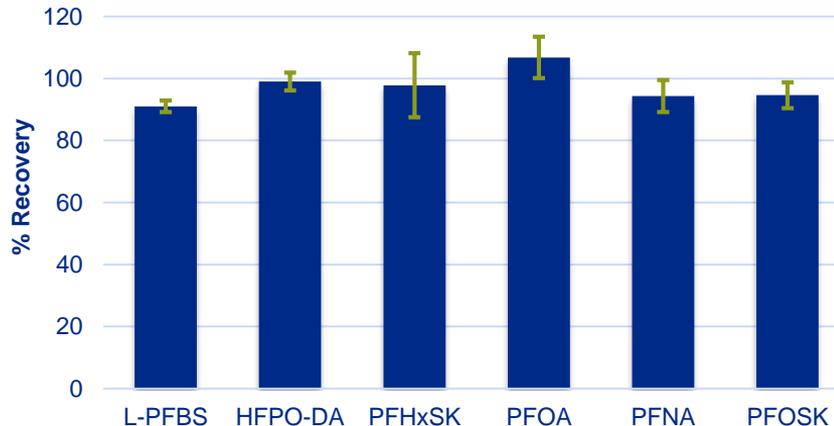




EPA's Proposed Action for the PFAS NPDWR

Analyte	MCL	PQL
PFOA	4 ng/L	4 ng/L
PFOS	4 ng/L	4 ng/L
PFNA	HI 1.0	4 ng/L
PFHxS		3 ng/L
PFBS		3 ng/L
HFPO-DA (GenX)		5 ng/L

n = 4, concentration: 500 mL drinking water spiked with an absolute amount of 1 ng each (HPFO-DA: 2 ng) → 2 ng/L (HPFO-DA: 4 ng/L)

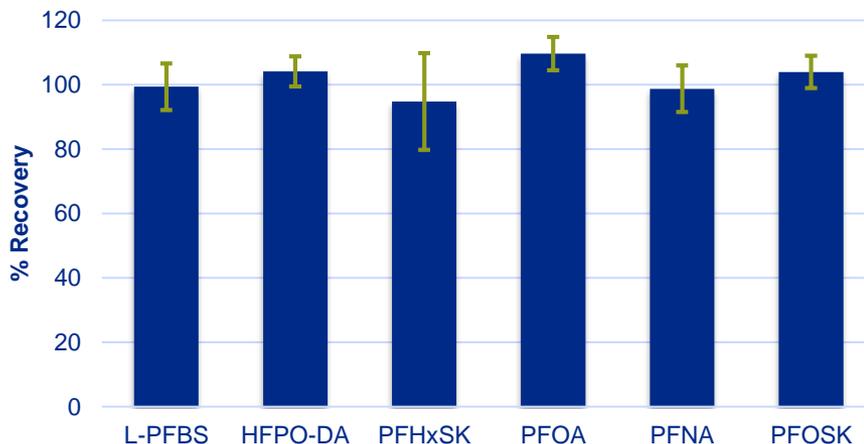




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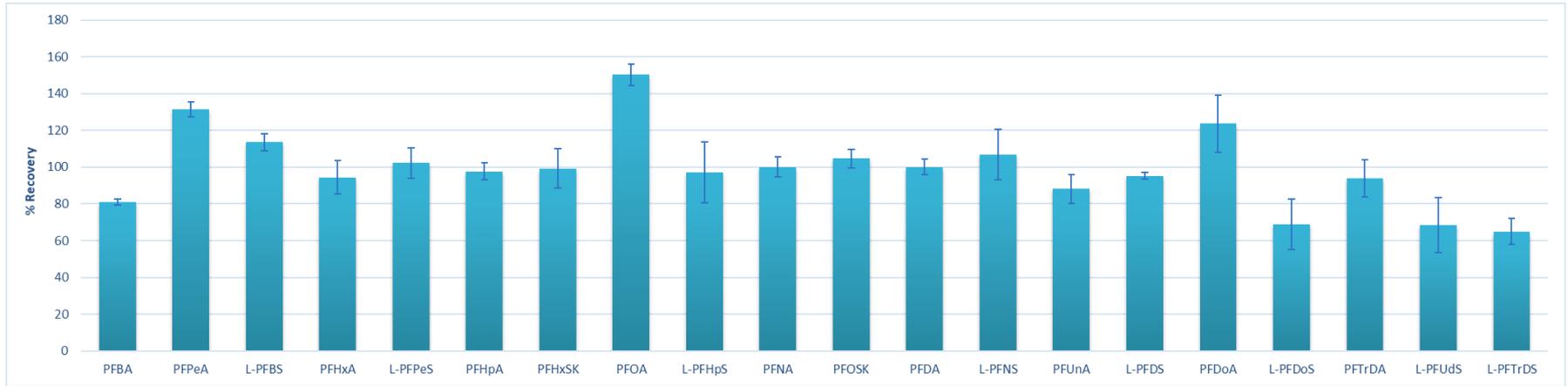
n = 4, concentration: 1000 mL drinking water spiked with an absolute amount of 0.4 ng each (HPFO-DA: 0.8 ng) → 0.4 ng/L (HPFO-DA: 0.8 ng/L)



EluCLEAN® PFAS – WAX 200 mg



New EU-DWD: The limit value is 100 ng/l (0.1 µg/l) for the sum of the twenty



n = 4, concentration: 1000 mL drinking water spiked with an absolut amount of 0.4 ng each (PFBA: 1.6 ng, L-PFUdS, L-PFTrDS 1.34 ng each) → 0.4 ng/L (PFBA: 1.6 ng/L, L-PFUdS, L-PFTrDS 1.34 ng/L)



Regulation (EC) No 1881/2006 as regards maximum levels of perfluoroalkyl substances in certain foodstuffs

The newly introduced maximum levels for EFSA's four priority PFAS applied from 1 January 2023. This is valid for certain foodstuffs placed on the market from the mentioned date. The four PFAS are perfluorooctanoic acid (PFOA), perfluorooctane sulfonic acid (PFOS), perfluorononanoic acid (PFNA) and perfluorohexane sulfonic acid (PFHxS).

LC Tech: SPE COLUMNS for PFAS ANALYSIS

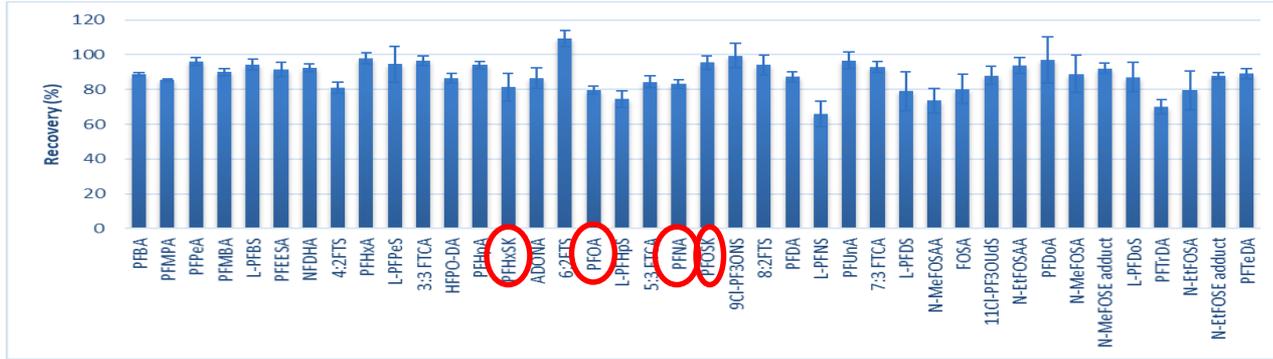


Analyte	MCL wet weight in egg	MCL wet weight in minced meat
PFOA	0.30 µg/kg	0.80 µg/kg
PFOS	1.0 µg/kg	0.30 µg/kg
PFNA	0.70 µg/kg	0.20 µg/kg
PFHxS	0.30 µg/kg	0.20 µg/kg
Sum of the four	1.7 µg/kg	1.3 µg/kg



EluCLEAN® PFAS – Universal

Food matrices



Minced meat

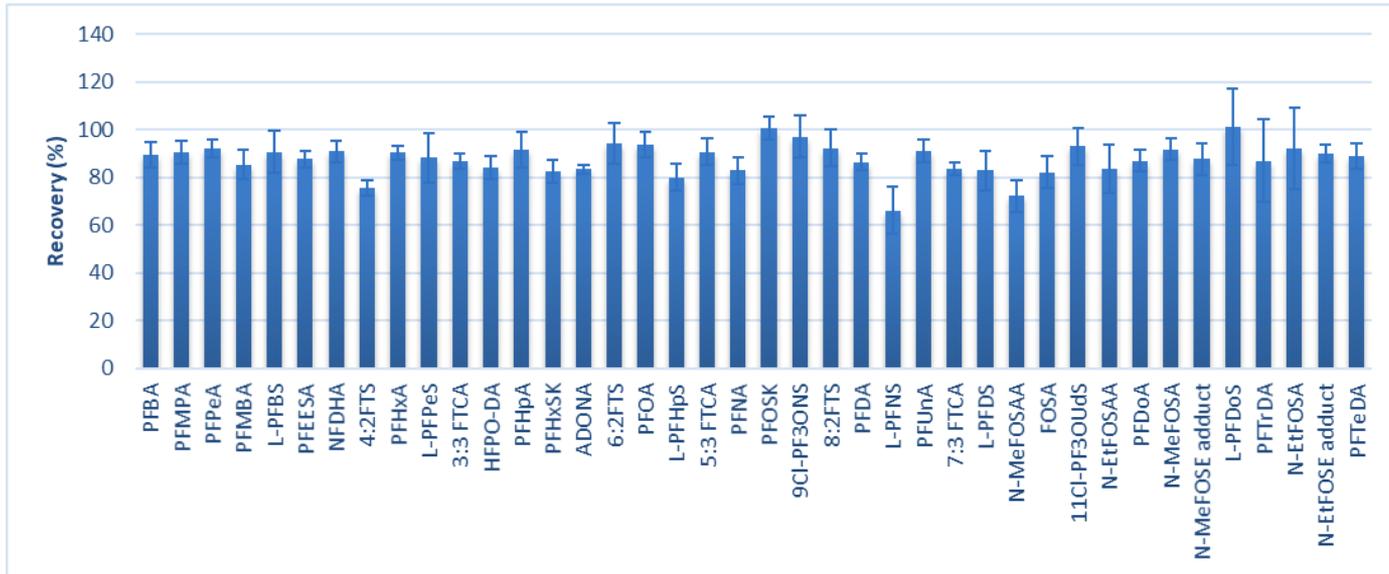
Recovery rates of 40 PFAS from minced meat (beef/pork) with EluCLEAN® PFAS - Universal columns (n = 4, 0.1 - 8 µg/kg (0.4 µg/kg each for PFOS, PFOA, PFNA, PFHxS))



Egg yolk

Recovery rates of 40 PFAS from egg yolk with EluCLEAN® PFAS - Universal columns (n = 4, 0.1 - 8 µg/kg (0.4 µg/kg each for PFOS, PFOA, PFNA, PFHxS))

Example Applications:

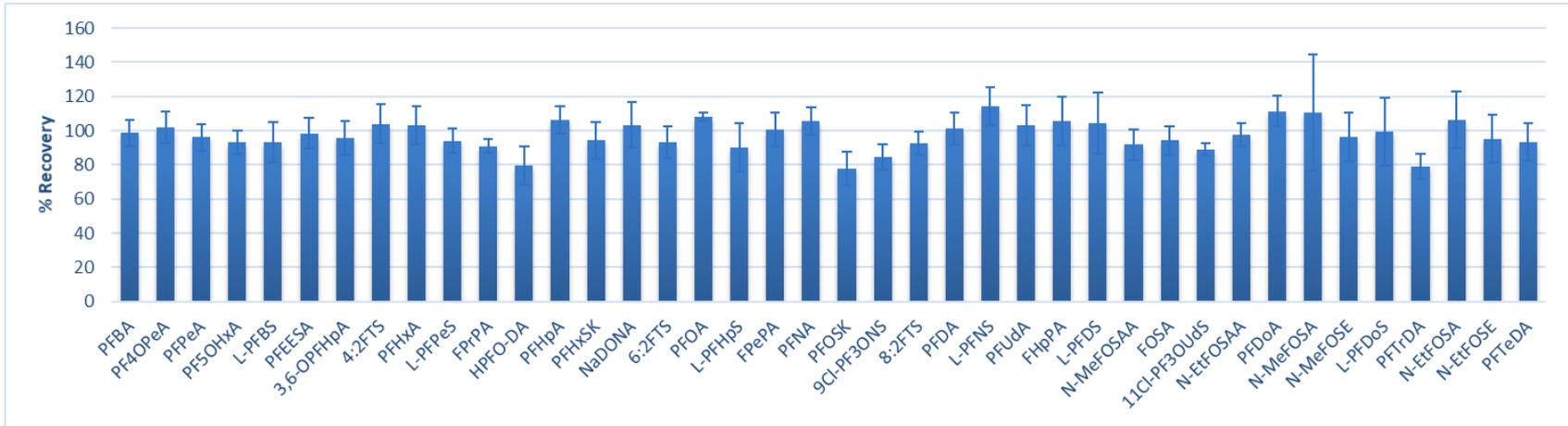


Carrots

Recovery rates of 40 PFAS from carrots with EluCLEAN® PFAS - Universal HP columns (n = 4, 0.1 - 8 µg/kg (0.4 µg/kg each for PFOS, PFOA, PFNA, PFHxS))

EluCLEAN® PFAS – WAX / GCB

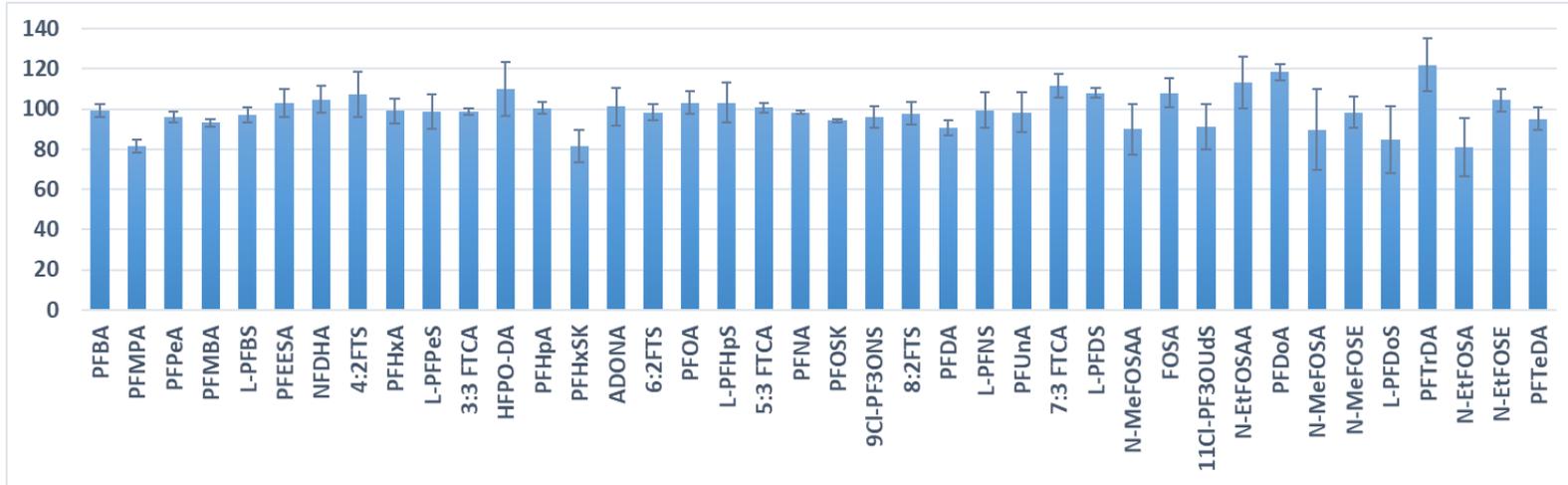
US EPA draft method 1633 - Soil



Recovery rates of 40 PFAS (listed in US EPA method 1633 2nd draft) from soil with EluCLEAN® PFAS WAX/GCB columns (n = 4, c = 0.5 - 40 µg/kg)

EluCLEAN® PFAS – Universal

US EPA draft method 1633 - Soil

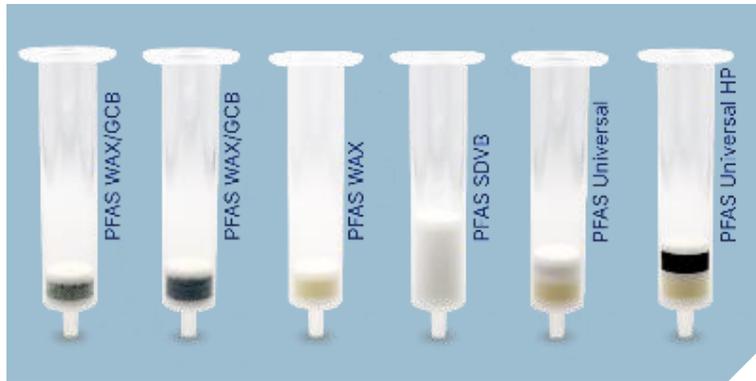


Recovery rates of 40 PFAS (listed in US EPA method 1633 2nd draft) from soil with EluCLEAN® PFAS WAX/GCB columns (n = 4, c = 0.5 - 40 µg/kg)

Conclusion



Using suitable workflow (e.g. SPE Cartridges and instruments) - can be successfully used for all kind of matrices, are compliant to standards and show very good recoveries and low standard deviations for a wide range of PFAS



LCTechs EluCLEAN PFAS SPE cartridges

Acknowledgement:
Dr. Thomas Gersthagen
Dr. Uwe Aulwurm
Mr. Sebastian Wierer
all at LCTech GmbH

[Hervé Ozouf](#)
Serlabo Technologies

PFAS Sample Prep: Think LCTech!
Excellence meets Experience

Thank You for your Attention!
Any Questions?



LCTech GmbH

Daimlerstraße 4

84419 Obertaufkirchen

Deutschland

info@LCTech.de

www.LCTech.de

www.LCTech-online.com