Streamlining the Sample Preparation for *PFAS Analysis* According *US-EPA* Methods

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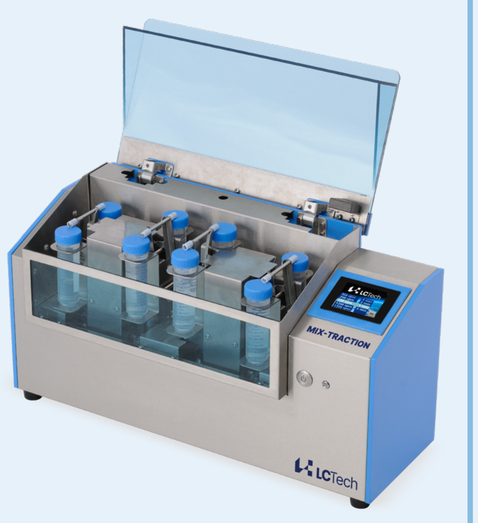
Introduction

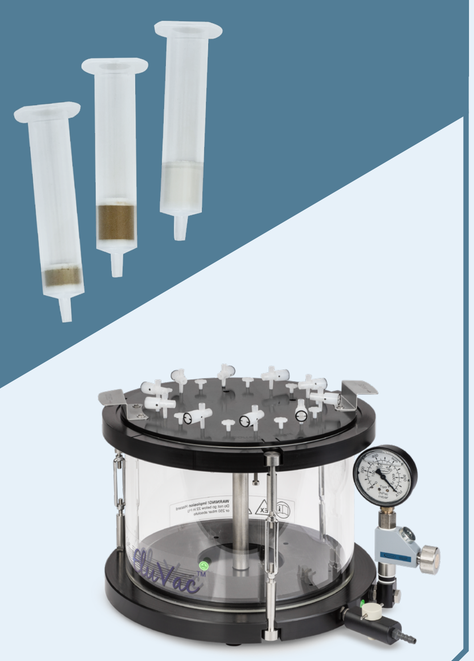
Analyzing PFAS compounds is challenging. The analytical interest in these compounds has rapidly increased in the last few years. The US EPA (e.g. US EPA methods 537.1, 533 and 1633),US DoD and european authorities have already published several methods for PFAS analysis in drinking water and environmental matrices. The analysis of PFAS from food and feed is also an emerging topic. The EURL has just recently published a guidance document for the analysis of PFAS in food and feed.[1] Dedicated lab equipment helps to avoid blind values and standardizes the processes in sample preparation. Laboratory equipment for shaking like the MIX-Traction will help to save extraction time. An automated SPE-System like the FREESTYLE can save time and costs and delivers reliable and reproducible SPE results. The vaccum centrifuge D-EVA allows parallel concentration of a large number of samples. A cruicial part is using suitable SPE columns. Currently, there are single cartridge SPE solutions for drinking water. The aim is to reduce costs per cartridge. More demanding sample matrices, for instance, environmental and food/feed matrices are handled with more elaborate dual SPE or a combination of SPE and dispersive clean-up. The challenge is to develop a SPE solution that is less cost intensive and at the same time as effective as a dual SPE or a combination of SPE and dispersive clean-up.

Material and Methods

Extraction



SPE/Clean-up



Sample concentration

PFAS in Food and Feed with automated dual SPE method on the FREESTYLE robotic system[1]

Example method can be found in the ANNEX of the EURL POPS Guidance Document on Analytical Parameters for the Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Food and Feed[1].

Results: PFAS in Drinking Water and Soil

LCTech ELUCLEAN PFAS-Drinking Water I SPE column

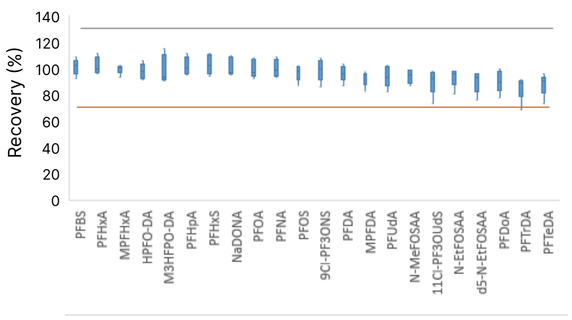


Figure 1: Recovery of PFAS in Drinking Water for US EPA 537.1 including surrogates. SPE were performed following US EPA 537.1 method protocol (n=5). 18 PFAS analytes and 4 mass labelled PFAS surrogates standards were spiked in 250 mL drinking water (concentration of PFAS analytes in water = 40 ng/mL and 160 ng/mL for d5-N-EtFOSAA).

LCTech ELUCLEAN PFAS-Drinking Water I SPE column

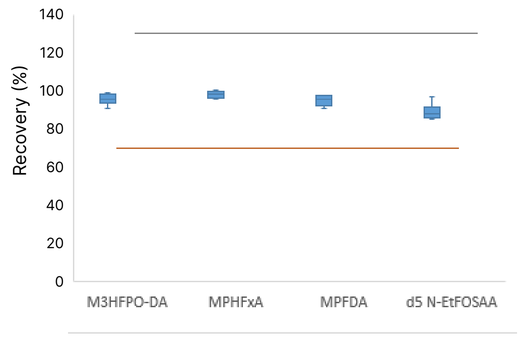


Figure 2: Recovery of 4 mass labelled surrogates PFAS in Drinking Water for US EPA 537.1 with FREESTYLE robotic system. SPE were performed following US EPA 531.1 method protocol (n=5) in FREESTYLE automated system. Concentration in 250 mL drinking water = 40 ng/mL (160 ng/mL for d5-N-EtFOSAA).

LCTech ELUCLEAN PFAS-Drinking Water II SPE column

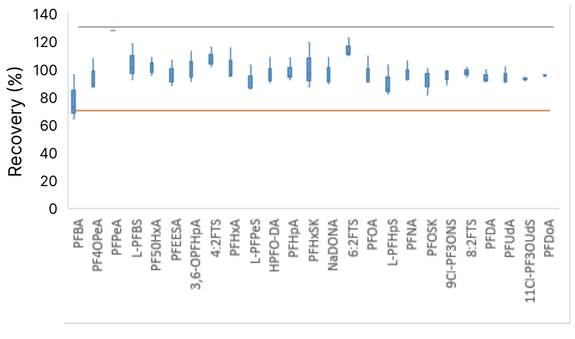


Figure 3: Recovery of PFAS in Drinking Water for US EPA 533. SPE was performed following US EPA 533 method protocol (n=3, *n=1). 25 PFAS were spiked in 250 mL drinking water for extraction (Concentration in water = 20ng/L).

LCTech ELUCLEAN PFAS-Environmental I SPE column

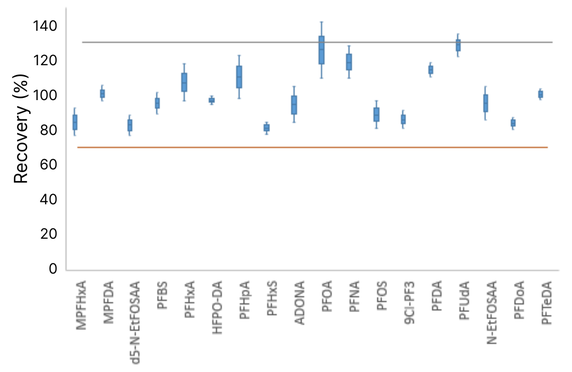


Figure 4: Recovery of selected PFAS in soil for US EPA 1633. Extraction was performed via MIX-TRACTION and SPE was performed following US EPA 1633 method protocol (n=2, without glass wool). Soil was spiked after the extraction step before SPE with a resulting final concentration of 8 ng/40 mL (32ng/40 mL for d5-N-EtFOSAA).

Conclusion

The presented workflow shows a streamlined sample preparation process with excellent recovery rates for the PFAS analysis with new low cost polymeric SPE cartridges for drinking water in compliance with US EPA methods 537.1 and 533. Further, a single cartridge SPE solution (instead of a dual SPE) shows recovery rates within the acceptance criteria for selected PFAS analytes in soil in compliance with US EPA 1633. The new introduced shaker MIX-Traction allows parallel extraction of multiple samples and is therefore time and cost saving.

The use of D-EVA avoids the loss of neutral PFAS. The automated FREESTYLE system is proven to perform without blind values and delivers excellent recovery rates with low standard deviations and thus ideally suited for automated PFAS SPE. The FREESTYLE robotic system is officially mentioned for the automated solid-phase extraction process in food and feed in EURL guidance.[1]

Future Prospects

Our work will continue and in the near future we will launch more new single SPE-cartridge solutions which can be used for PFAS analysis in compliance with US EPA 1633 and DoD 5.1/5.3 methods for different matrices. Additionally, a specially designed SPE column with improved clean-up for different matrices like soil, waste water and food/feed matrices will be introduced.

References

[1] EURL for halogenated POPs in feed and food (2022): Guidance Document on Analytical Parameters for the Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Food and Feed, version 1.2 of 11 May 2022. ANNEX Version 1.0, 6.4.3 Module 2C – Solid-phase extraction (SPE), automated. Available online under: https://eurl-pops.eu/core-working-groups#_pfas

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