Development of Suitable Methods for *PFAS Sample Preparation* in Different Matrices

Gersthagen T.[1] , Köpf A.[1], Wierer S.[1] and Kharel S.[1]
[1] LCTech GmbH, Daimlerstrasse 4, 84419 Obertaufkirchen, Germany, gersthagen@LCTech.de, www.LCTech.de

1. Introduction

Per- and polyfluorinated alkyl substances (PFAS) get into the environment during their manufacturing process and further also during their use and disposal. Analyzing these PFAS compounds is challenging and dedicated lab equipment helps to avoid blind values and standardizes the processes in sample preparation. Different types of PFAS and different matrices have different needs for extraction, enrichment and clean-up. Another problem is the volatility of certain compounds [1].

Currently, there are existing SPE cartridge solutions for extractions of PFAS from drinking water. However, solid samples (especially soil samples) have often the ability to strongly bind neutral and long chain PFAS. An appropriate method for extraction of these PFAS from solids and for the critical evaporation step has to be established. Further, more demanding sample matrices, for instance, environmental and food/feed matrices are handled with a more elaborate dual SPE or a combination of SPE and dispersive clean-up to remove matrix intereferences. In spite of the tedious process, all of them lack the ability to enrich some specific PFAS in a sufficient way e.g. neutral sulfonamides or long chain PFAS. The challenge is to develop a SPE solution that is not cost intensive and at the same time effective for the clean-up and enrichment for all types of PFAS analytes.

2. Materials and Methods

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

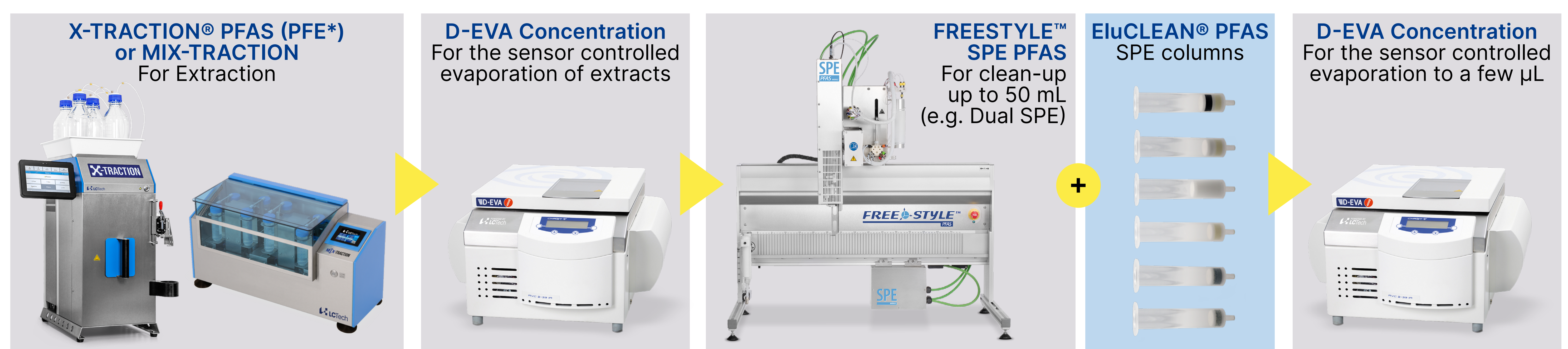**D-EVA Concentration** For the sensor controlled evaporation to a few µL

Figure 1: Workflow for solid samples (first two steps are not needed for aqueous samples)   * Pressurized Fluid Extraction

3. Results and Discussion

3.1. Solid-Liquid Extraction

Ideally a blind value free PFE-system should be used for extraction. The PFE-System shows best recoveries for long chain and neutral sulfonamide PFAS.
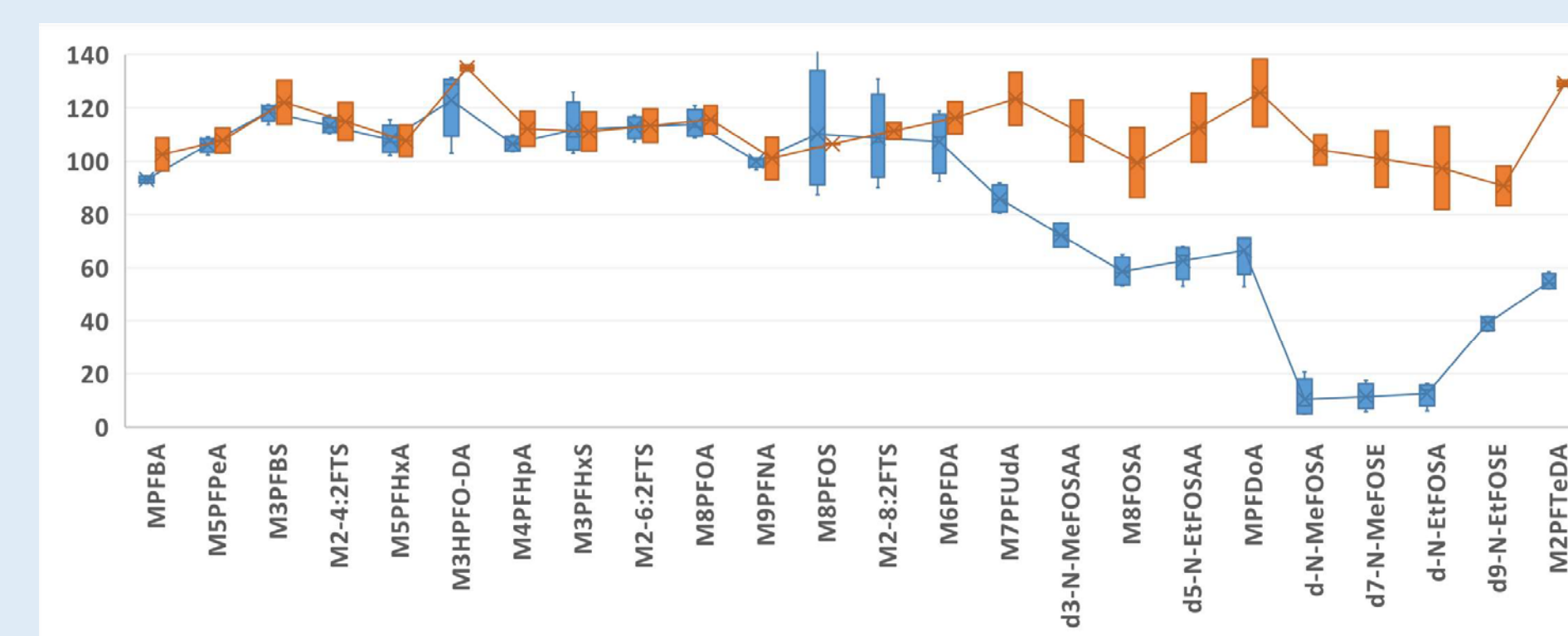

Figure 2: Comparison of recovery rates in % of PFAS compounds in spiked farmland soil with different extraction methods, n = 3 (orange: X-TRACTION (PFE-System), blue: shaker)

3.2. Solid-Phase Extraction

For solid-phase extraction a new dual-layer column was developed. This column shows excellent recovery rates for a wide range of PFAS analytes in various matrices.
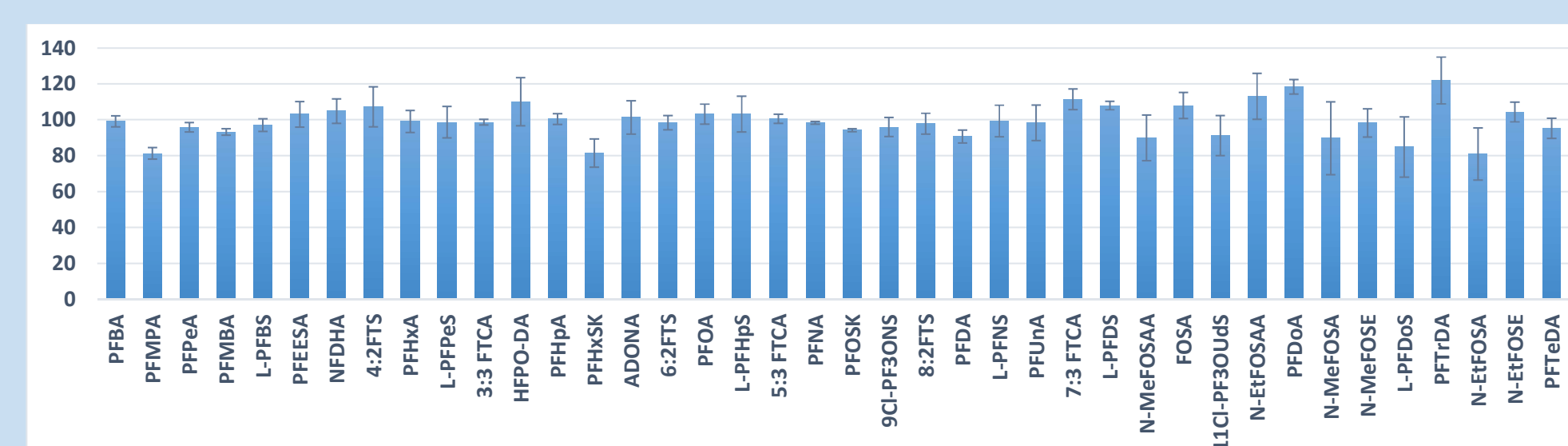

Figure 3: Recovery rates in % of 40 PFAS analytes in farm land soil using EluCLEAN® PFAS – Universal columns on automated FREESTYLE PFAS system, n = 4, conc. 0.5 - 40 µg/kg
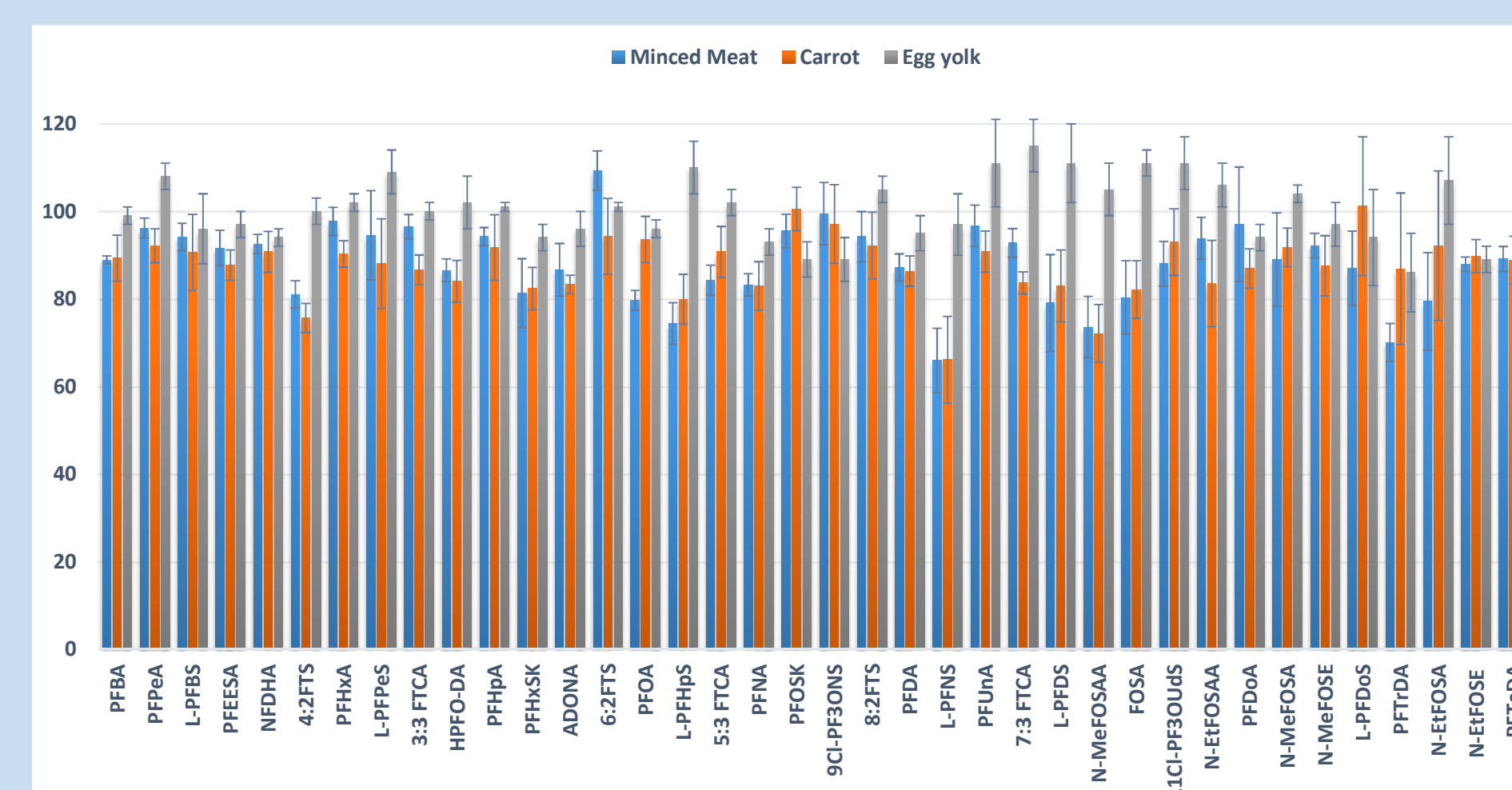

Figure 4: Recovery rates in % of 40 PFAS analytes in different food matrices using EluCLEAN® PFAS – Universal (and HP for carrot) columns, n = 4, conc. 0.1 - 8 µg/kg
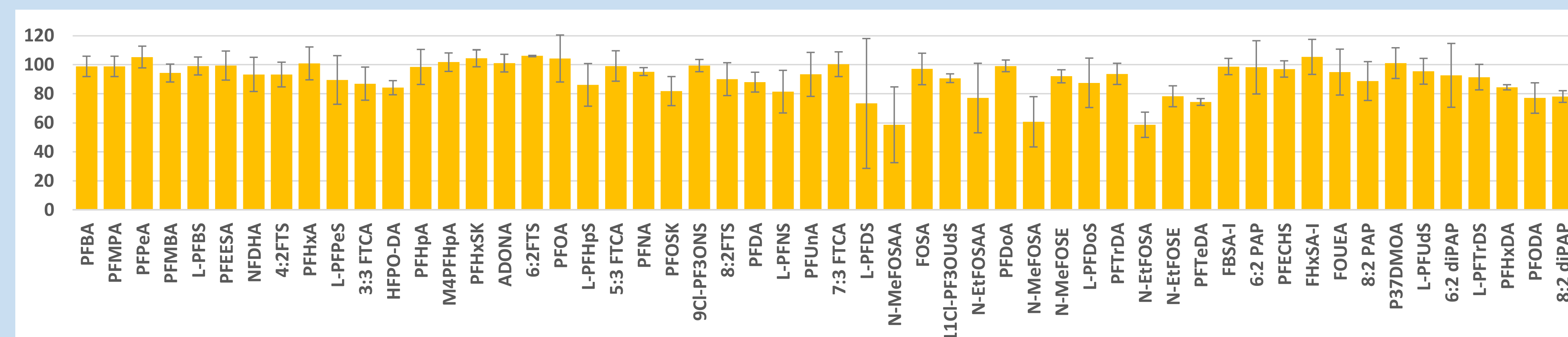

Figure 5: Recovery rates in % of 52 PFAS analytes in drinking water using EluCLEAN® PFAS – Universal, n =3, conc. 0.325 - 40 ng in 50 mL tap water

3.3. Sample Concentration

An evaporation method where no volatile PFAS compounds were lost has been successfully developed using a vacuum centrifuge in combination with a cold trap. Optimal final volume lies between 600 - 1000 µL.
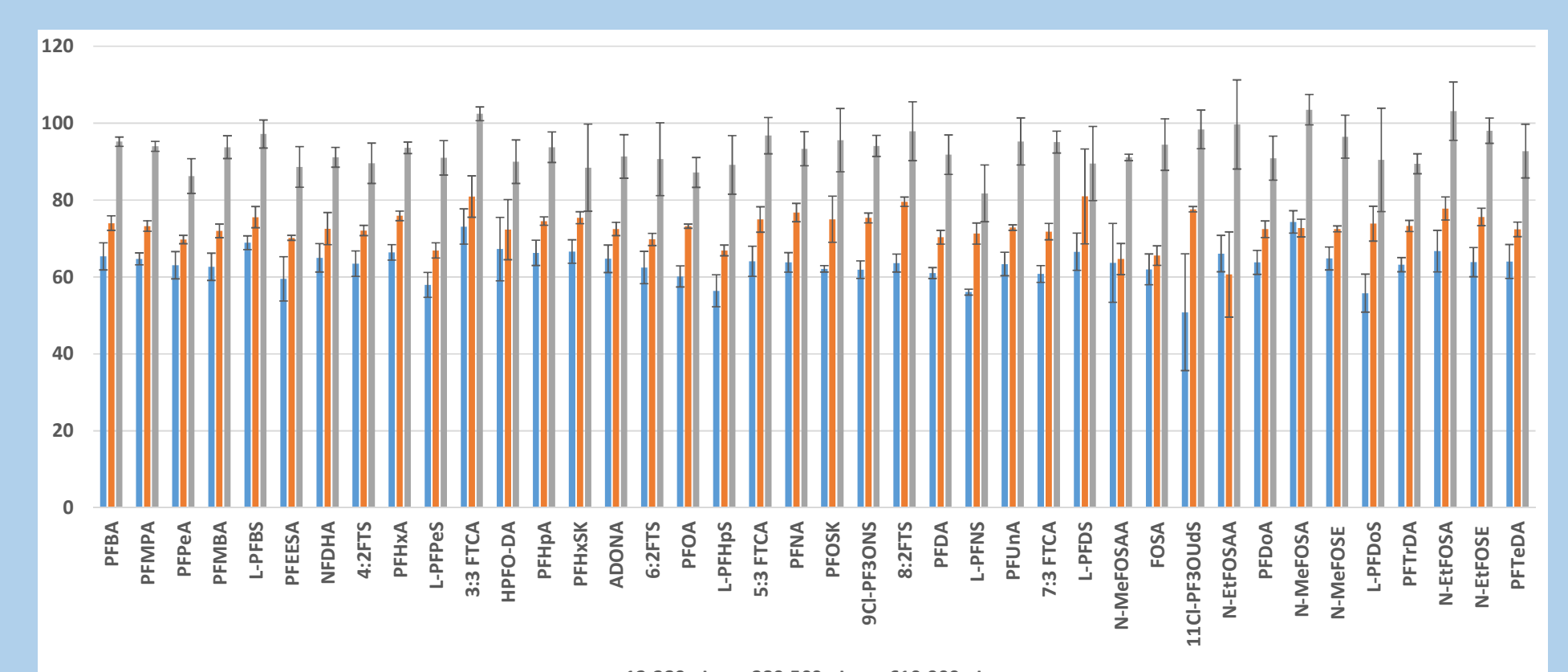

Figure 6: Comparison of Recovery rates of 40 PFAS analytes for different final volumes, n = 3 for each volume range, conc.

4. Conclusions

The presented workflow shows a streamlined sample preparation process for PFAS analysis which can be used for different kinds of PFAS including neutral sulfonamides and long chain analytes in various matrices. A blind value free automated system was integrated to perform solid-phase extraction experiments. Further, SPE cartridges with a superior performance for enrichment and clean-up of PFAS from water, soil and food matrices were developed.

5. References

[1] C.M.A., Eichler, J.C. Little. 2020. A framework to model exposure to per- and polyfluoroalkyl substances in indoor environments. Environ. Sci.: Processes Impacts, 2020,22, 500-511