Analysis of ***48 PFAS (UK DWI list) from aqueous samples*** Using Automated FREESTYLE XANA PFAS TableTop System and EluCLEAN® PFAS SPE Cartridges

Dr. Suman Kharel, Dr. Thomas Gersthagen, Sebastian Wierer, LCTech GmbH

Content

Key Features

- Excellent recovery rates and low standard deviations for 48 PFAS analytes
- No detectable PFAS background contamination from both systems and cartridges
- Reliable and robust automation with FREESTYLE XANA-PFAS TableTop
- Fully automated processing of up to 30 samples in one sequence
- Parallel processing of samples

LCTech Products

SPE cartridges

Part No.: 21821, 20831, 20841 (150 mg/6 mL) // 20871, 20872, 20873 (200 mg/6 mL)

EluCLEAN® PFAS – WAX

Sorbent: Weak Anion Exchanger, Mixed-Mode Polymeric Sorbent (WAX)

FREESTYLE XANA PFAS TableTop

Part No.: 20600	FREESTYLE XANA-PFAS TableTop (30 positions)
Part No.: 19372	SPE cartridge adapters for elution into 50 mL Falcon tubes (10 pcs.)
Part No.: 14923-PFAS	Caps for 6 mL SPE PFAS cartridges, reusable (10 pcs.)
Part No.: 13156	Solvent bottle rack, 6 x 1 L
Part No.: 12709	Overflow Sensor for Waste Level Control, with GL 45 Cap

Figure 1. FREESTYLE XANA PFAS TableTop with EluCLEAN® PFAS – SPE cartridges

Figure 2. Equipping the bottle rack of the FREESTYLE XANA PFAS TableTop

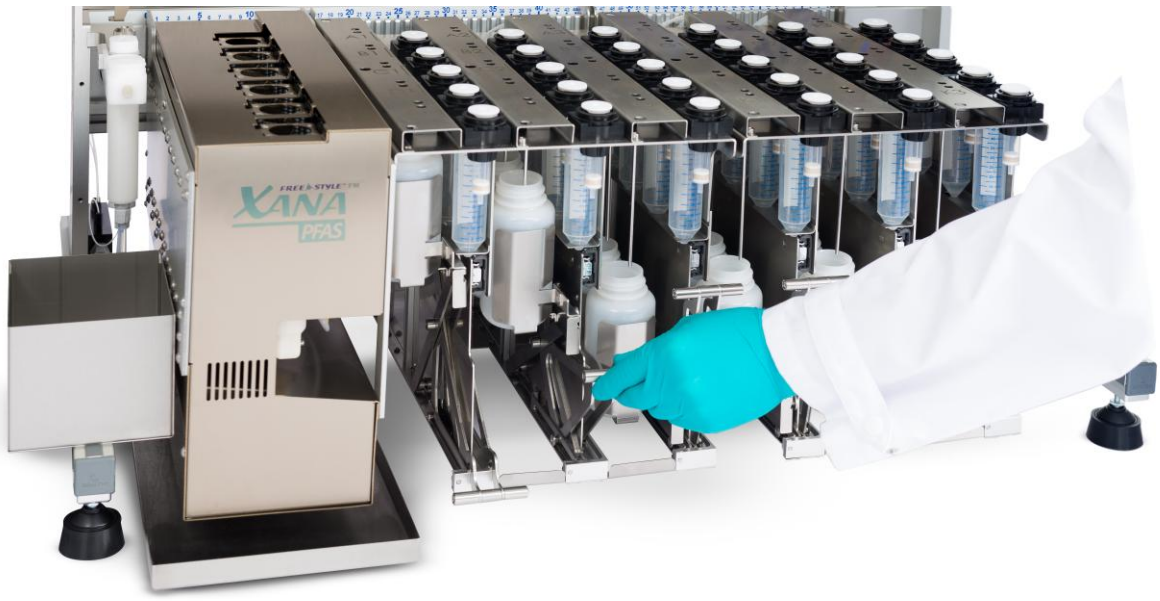


Figure 3. Loading the bottles of the FREESTYLE XANA PFAS TableTop

1. Introduction

Per- and polyfluorinated alkyl substances (PFAS) products have been in use for more than 60 years. They get into the environment during their manufacturing process, usages and disposal. Research has revealed the high toxicity of PFAS compounds and thus the resulting need to regulate the substances. Therefore the analytical interest in these compounds has rapidly increased in the last few years. The method described here is derived from european standard method[1] and require solid phase extraction (SPE) prior to liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis. This method apply SPE cartridges containing a weak anion exchanger, mixed-mode polymeric sorbent.

The following application note shows how aqueous samples can be prepared fully automated for LC-MS/MS analysis by applying SPE with the FREESTYLE XANA-PFAS TableTop robotic system. By the application of fully automated parallel sample preparation, multiple samples can be processed at the same time. Thus, a high sample throughput at low demand of personnel resources is obtained. The FREESTYLE XANA-PFAS TableTop robotic system is a solution especially for PFAS applications by avoiding fluorine-containing plastics such as PTFE and is thus solving the challenge: No contamination from the system were measurable.

The EluCLEAN® PFAS WAX SPE cartridge contains a weak anion exchanger and mixed-mode polymeric sorbent.The SPE cartridge shows excellent recovery rates in combination with low standard deviations and is therefore ideally suited for SPE of PFAS from aqueous matrices. The fully automated solution for SPE saves time and costs in PFAS analysis.

In this application note the EluCLEAN® PFAS WAX SPE cartridges were used in FREESTYLE XANA PFAS TableTop to extract PFAS analytes automatically from aqueous samples.

2. Experiments

250 mL (lower volume can also be used) of reagent water is taken in 250 mL PE bottles (In case of other aqueous samples like tap water, formic acid should be added to achieve a pH in range of 4-7). 48 Native PFAS and 24 labelled standard were spiked as mentioned in table below.

Table 1. 55 Native PFAS and 24 isotope dilution standards

Compounds	ng
Labelled compounds	**1.64– 33.3**
11Cl-PF3OUdS, 9Cl-PF3ONS, ADONA, HFPO-DA, NFDHA, PFEESA, PFMOBA PFMOPrA, PFPeA	**8**
PFBA, 4:2FTS, 8:2FTS	**16**
N-MeFOSE, N-EtFOSE	**40**
5:3 FTCA, 7:3 FTCA	**80**
FBSA-I, PFECHS, FHXSA-I	**6.67**
PFHxDA, PFODA	**3.33**
L-PFUdS, L-PFTrDS	**2**
All other PFAS*	**4**

*All PFAS are visible in Fig 7 below

Table 2. FREESTYLE XANA-PFAS TableTop method

LCTech FreeStyle - Report on Methods: WAT Type PFAS - EPA

Column:	LCTech_3ml.col		Extension cannula:	yes
Conditioning 1: Volume: Suction Speed:	ON 10 ml 20 ml / min	 Dispensing Speed: Waiting time:	 20 ml / min 0 min	 Port : W8 0,1% NH4OH MeOH
Conditioning 2: Volume: Suction Speed:	ON 10 ml 20 ml / min	 Dispensing Speed: Waiting time:	 20 ml / min 0 min	 Port : W4 MeOH
Conditioning 3: Volume: Suction Speed:	ON 10 ml 20 ml / min	 Dispensing Speed: Waiting time:	 20 ml / min 0 min	 Port : W10 H2O
Conditioning 4:	OFF			
Load 1: Number of bottles: without rinsing of vial	ON 1	Typ: Transfer Speed	empty 20 ml / min	
Washing 1:	OFF			
Washing 2:	OFF			
Drying 1: Time:	ON 5 min	stay on actual position		
PFAS RinsElution 1x Volume: Suction Speed:	ON 4 ml 15 ml / min	 Dispensing Speed: Waiting time:	 3 ml / min 0.33 min	 Port : 1 MeOH
RinseElute 2: 1x Volume: Suction Speed:	 7 ml 15 ml / min	 Dispensing Speed: Waiting time:	 3 ml / min 0.33 min	 Port : 8 0,1% NH4OH MeOH
PFAS Extra Cleaning: 1x				Port : W4 MeOH

5 µL of concentrated acetic acid and 10 µL NIS (MPFAC-HIF-IS) solution were added to each sample eluate and vortexed.

2.1 Evaporation/Concentration

All samples were evaporated to 1-2 mL using D-EVA Rotational Vacuum Concentrator (temperature: 45°C, vacuum: 20 mbar) and transferred into a 1.5 mL polypropylene vial and kept at 0 – 4 °C for LC-MS/MS analysis.

2.2 Instrumentation

Table 3. LC Parameters

Parameter	Value	
LC	Thermo Scientific Vanquish Flex UHPLC system	
Analytical column	Acclaim 120, C18, 3 µm, 2.1 x 150 mm	
Delay column	Acclaim RSLC, PolarAdvantage II, 3.0 mm x 33 mm, 3 µm	
Column temperature	40 °C	
Injection volume	5 µL	
Mobile Phase	A) H2O B) 2 mM ammonium acetate MeOH with 2 % H2O and 0.1 % acetic acid	
Gradient Flow rate	0.4 mL/min	
Gradient	Time (min) 0 1 4 14 17 18 20 22	% B 10 100 50 100 100 10 10 10

Table 4. MS Parameters

Parameter	Value
MS	TSQ Quantis (Thermo)
Polarity	Negative
Spray voltage	2500 V
Sheath Gas	50 Arb
Aux Gas	10
CID Gas	2 mTorr
Ion transfer tube temp	325 °C
Vaporizer Temp	300 °C
Q1 resolution	0.7 FWHM
Q3 resolution	1.2 FWHM
Cycle time	0.5 sec
Chromatographic peak width	6 sec

3. Results

3.1 Blind values of system and cartridges

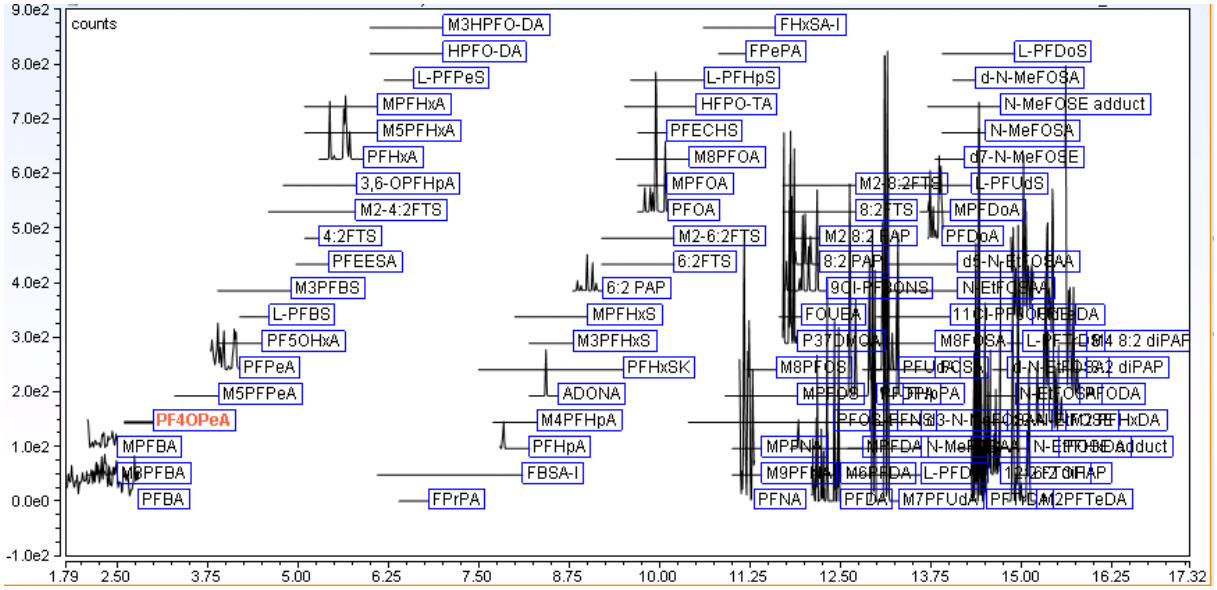


Figure 4. Chromatogram demonstrating EluCLEAN® PFAS WAX cartridges are free of blind value when screened for 48 PFAS analytes

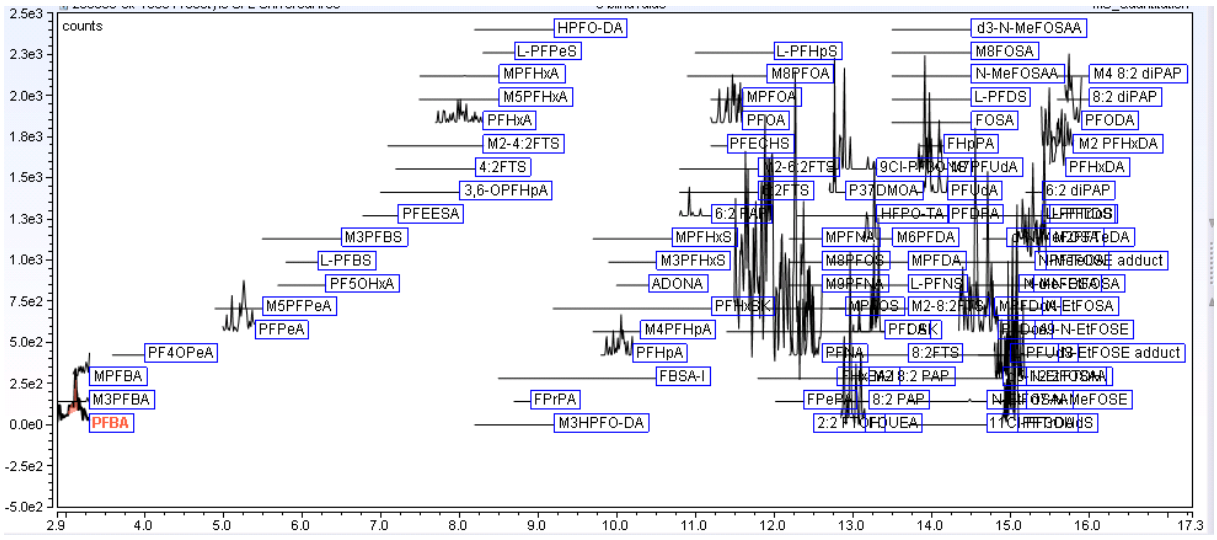


Figure 5. Chromatogram demonstrating FREESTYLE XANA PFAS –TableTop is free of blind value when screened for 48 PFAS analytes

3.2 Recovery of 48 PFAS

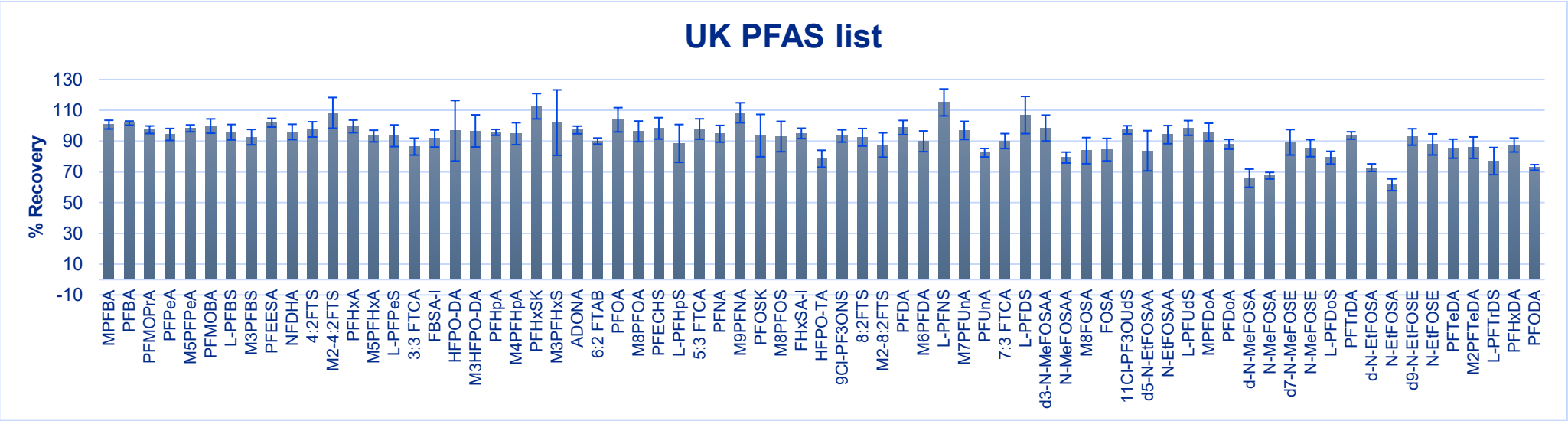


Figure 6. Net Recovery rates of 48 PFAS from reagent water extracted with EluCLEAN® PFAS WAX SPE cartridge (n=4, spiked concentration=1.64-80 ng in 250 mL reagent water) using FREESTYLE XANA PFAS TableTop automated SPE

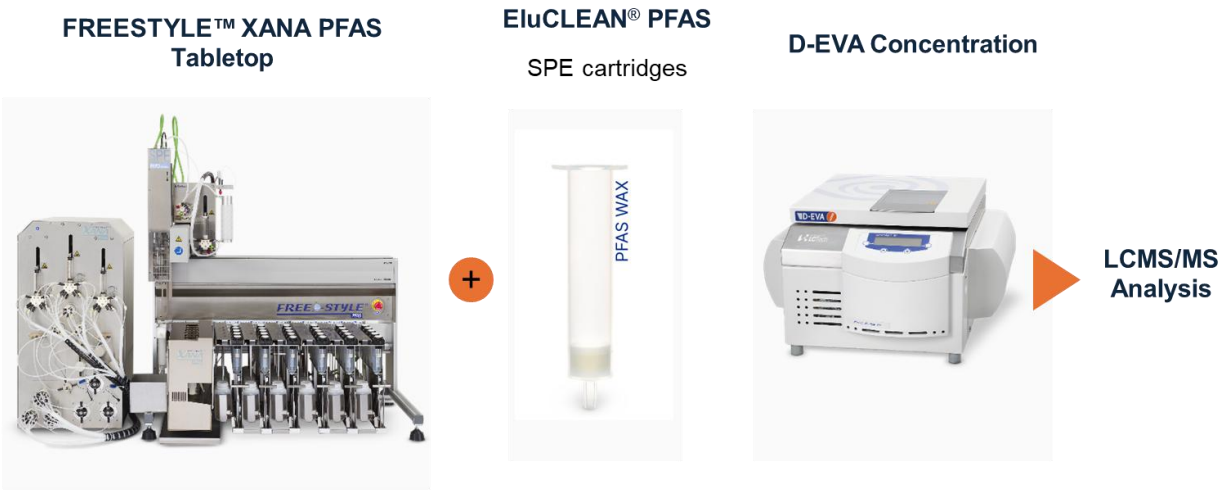
(Note: 6:2 FTAB recovery were derived from customer results as the analyte was not present in house during this measurement period)

3.3 Flowrate vs time

It is possible to speed up conditioning and loading step without compromising the performance. The equivalent results were obtained when the flow rate is increased to 2x and 4x during conditioning and loading step but keeping the flow rate constant for final elution. The sample processing time for large number of samples will be significantly reduced when using higher flow rates. Below is the example of flowrate vs time for processing of 3 samples in parallel.

4. Conclusion

- ✓ Both cartridge and automated system have no detectable PFAS background contamination.
- ✓ EluCLEAN® PFAS WAX combined with FREESTYLE XANA PFAS TableTop achieve recoveries between 70-130%
- ✓ Ideally suited to be used for the enrichment and clean-up of PFAS for all kind of aqueous matrices
- ✓ A fully automated sample preparation
- ✓ Time and cost saving alternative for every laboratory
- ✓ Reliable and robust analysis



5. References

[1] ISO/CEN. (2024). EN 17892:2024 – Water quality — Determination of selected per- and polyfluoroalkyl substances in drinking water — Method using LC-MS/MS. European Standard

Any Questions?
Do not hesitate to contact us:

LCTech

LCTech GmbH
Daimlerstr. 4

84419 Obertaufkirchen, Germany
Tel. +49 8082 2717-0

info@LCTech.de
www.LCTech.de