

Analysis of **55 PFAS from Aqueous samples** Using Automated FREESTYLE XANA PFAS TableTop System and EluCLEAR[®] PFAS SPE Cartridges (European Standard EN 17892)

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Key Features

- Excellent recovery rates and low standard deviations for 55 PFAS analytes according to European Norm
- 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, 21822, 21823 (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

Other Relevant LCTech Application Notes and Product Information

<https://www.lctech.de/application-notes>

<https://www.lctech.de/en/products/pfas-workflow>



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



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

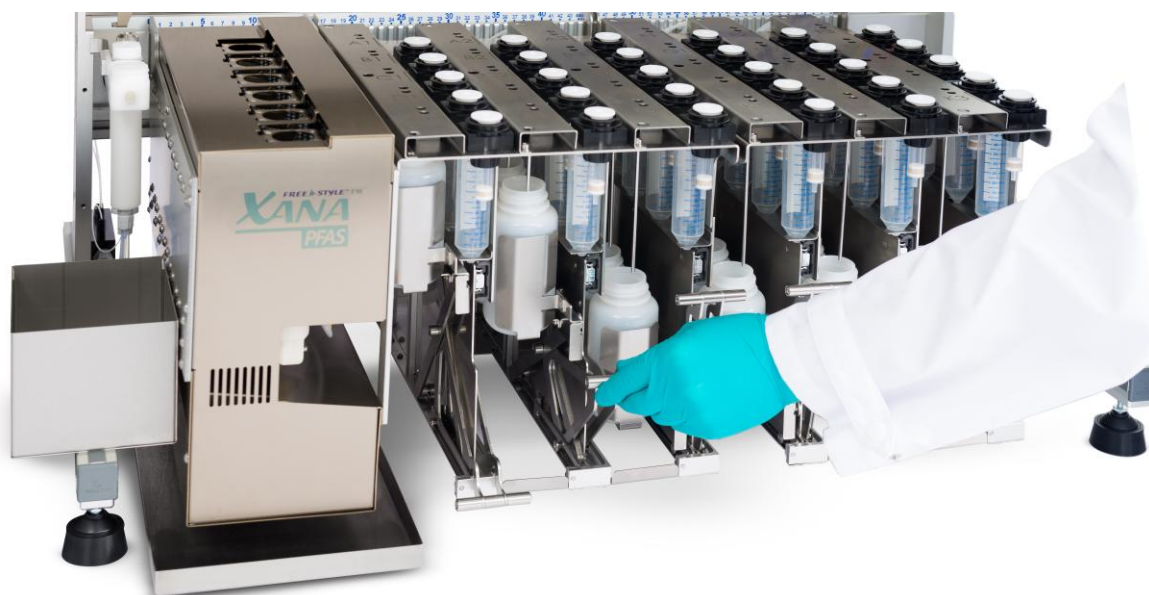


Figure 3. Loading the bottles of the FREESTYLE XANA PFAS TableTop



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. European standard method^[1] 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 amount can also be taken) of reagent water is taken in 250 mL PE bottles (In case of other aqueous samples like tap water, acetic acid should be added to achieve a pH in range of 4-7). 55 Native PFAS and 24 isotope dilution standard were spiked as mentioned in table below.

Table 1. 55 Native PFAS and 24 isotope dilution standards

Compounds	ng
Labeled compounds	1.64– 33.3
11Cl-PF3OUdS, 9Cl-PF3ONS, ADONA, HFPO-DA, NFDHA, PFEESA, PFMB, PFMPA, PFPeA	8
PFBA, 4:2FTS, 6:2FTS, 8:2FTS	16
N-MeFOSE, N-EtFOSE	40
5:3 FTCA, 7:3 FTCA	80
FBSA-I, PFECHS, FHXSA-I, P37DMOA, FOUEA, 6:2 diPAP, 8:2 diPAP	6.67
PFHxDA, PFODA	3.33
L-PFUdS, L-PFTrDS	2
6:2 PAP, 8:2 PAP, PFDPA	13.33
All other PFAS*	4

*All PFAS are visible in Fig 7 below



The method was designed to comply with the SPE procedure described in EN 17892 method.

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:	ON		
Volume:	10 ml	Dispensing Speed:	20 ml / min
Suction Speed:	20 ml / min	Waiting time:	0 min
			Port : W8 0,1% NH4OH MeOH
Conditioning 2:	ON		
Volume:	10 ml	Dispensing Speed:	20 ml / min
Suction Speed:	20 ml / min	Waiting time:	0 min
			Port : W4 MeOH
Conditioning 3:	ON		
Volume:	10 ml	Dispensing Speed:	20 ml / min
Suction Speed:	20 ml / min	Waiting time:	0 min
			Port : W10 H2O
Conditioning 4:	OFF		
Load 1:	ON	Typ:	empty
Number of bottles:	1	Transfer Speed	20 ml / min
without rinsing of vial			
Washing 1:	OFF		
Washing 2:	OFF		
Drying 1:	ON	stay on actual position	
Time:	5 min		
PFAS RinsElution	ON		
1x Volume:	4 ml	Dispensing Speed:	3 ml / min
Suction Speed:	15 ml / min	Waiting time:	0.33 min
			Port : 1 MeOH
RinseElute 2:			
1x Volume:	7 ml	Dispensing Speed:	3 ml / min
Suction Speed:	15 ml / min	Waiting time:	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 500 µL - 1 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 μ m, 2.1 x 150 mm																		
Delay column	Acclaim RSLC, PolarAdvantage II, 3.0 mm x 33 mm, 3 μ m																		
Column temperature	40 °C																		
Injection volume	5 μ L																		
Mobile Phase	A) H ₂ O B) 2 mM ammonium acetate MeOH with 2 % H ₂ O and 0.1 % acetic acid																		
Gradient Flow rate	0.4 mL/min																		
Gradient	<table> <tr> <th>Time (min)</th><th>% B</th></tr> <tr><td>0</td><td>10</td></tr> <tr><td>1</td><td>100</td></tr> <tr><td>4</td><td>50</td></tr> <tr><td>14</td><td>100</td></tr> <tr><td>17</td><td>100</td></tr> <tr><td>18</td><td>10</td></tr> <tr><td>20</td><td>10</td></tr> <tr><td>22</td><td>10</td></tr> </table>	Time (min)	% B	0	10	1	100	4	50	14	100	17	100	18	10	20	10	22	10
Time (min)	% B																		
0	10																		
1	100																		
4	50																		
14	100																		
17	100																		
18	10																		
20	10																		
22	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

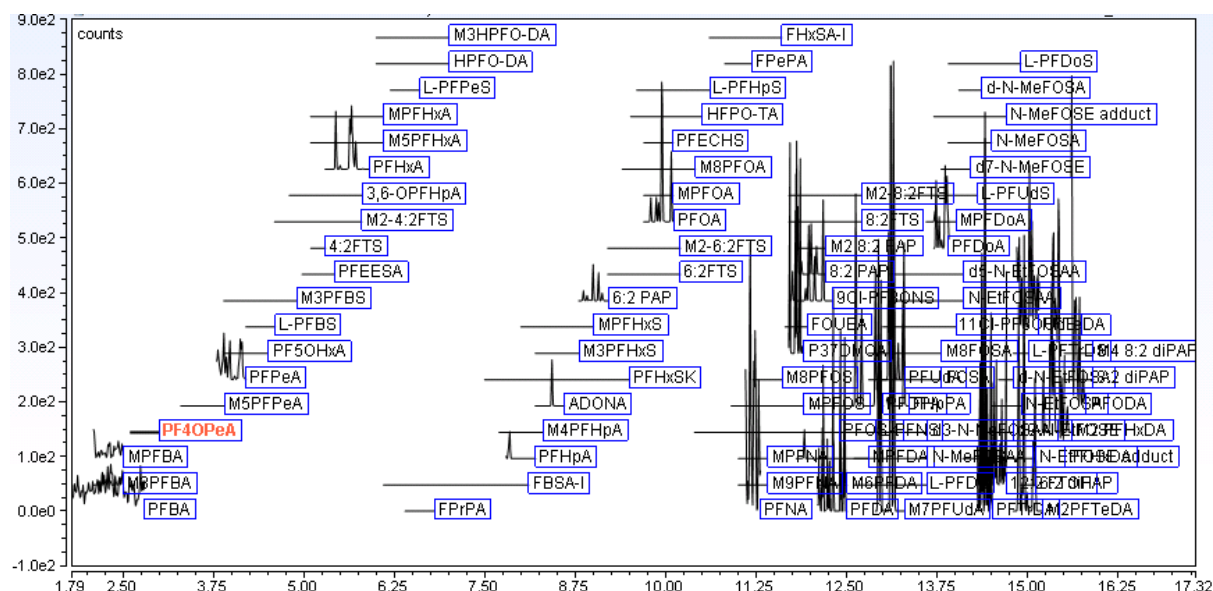


Figure 4. Chromatogram demonstrating EluCLEAN® PFAS WAX cartridges are free of blind value when screened for 55 PFAS analytes

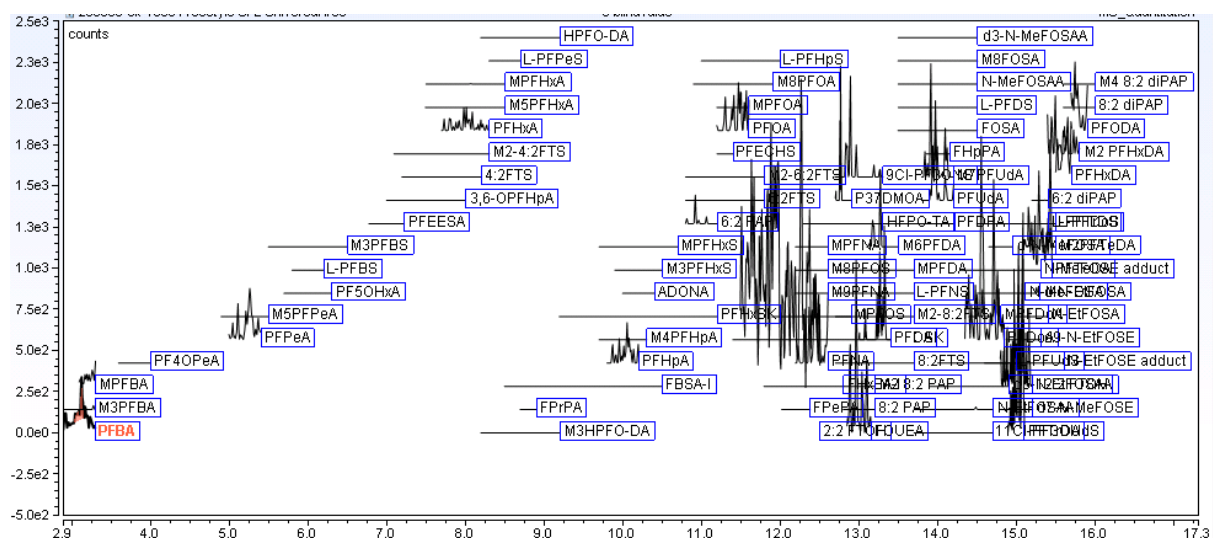


Figure 5. Chromatogram demonstrating FREESTYLE XANA PFAS –TableTop is free of blind value when screened for 55 PFAS analytes

3.2 Recovery of 55 PFAS

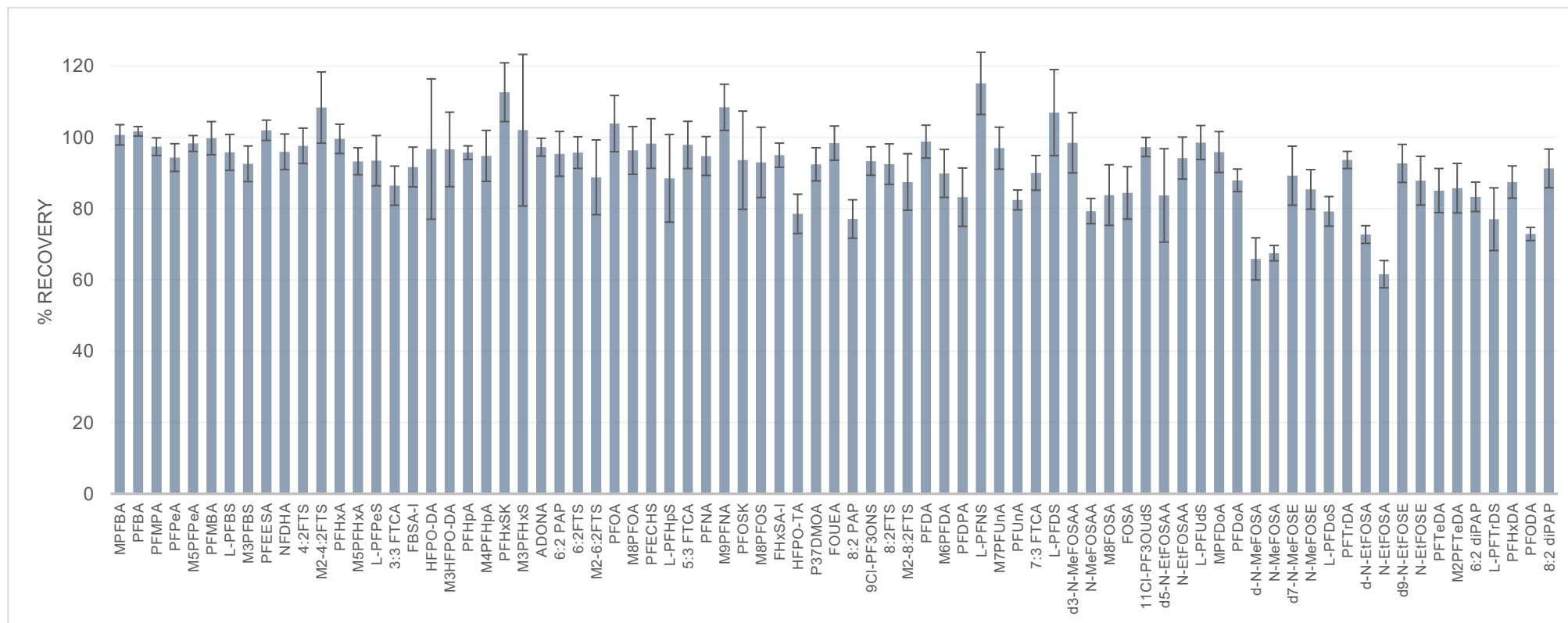


Figure 6. Recovery rates of 55 PFAS from reagent water extracted with EluCLEAR® PFAS WAX SPE cartridge (n=4, spiked concentration=1.64-80 ng in 250 mL reagent water) using FREESTYLE XANA PFAS TableTop automated SPE



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.

5 mL/min



100 mins / 3 samples

10 mL/min



70 mins / 3 samples

20 mL/min



55 mins / 3 samples



4. Conclusion

- ✓ Both cartridge and automated system have no detectable PFAS background contamination.
- ✓ EluCLEAR® 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

**FREESTYLE™ XANA PFAS
Tabletop**



EluCLEAR® PFAS
SPE columns



D-EVA Concentration



**LCMS/MS
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: