Rapid, Efficient, and High-Throughput Extraction of PFAS from Soil

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Introduction

There is increasing concern of per- and polyfluoroalkyl substances (PFAS) in our environment as a whole, due to their persistent nature. More and more regulation regarding PFAS is being implemented. Having a harmonized method to accurately determine the PFAS content in soil, as well as other solid matrices, is important to this industry. Further, the number of samples and sample types that laboratories are analyzing is rapidly growing. The extraction of PFAS can be challenging, given the susceptibility to contamination and the low levels in which these compounds are present. Existing techniques are predominately manual methods that are not rapid, simple, or efficient. In this study, a high-throughput extraction system that uses microwave technology is explored as an alternative to traditional PFAS sample preparation techniques. This method offers efficient extraction of PFAS from solid matrices in a batch format in under an hour. Extracts were either subject to cleanup following EPA 1633 or diluted for analysis. Acceptable extraction recovery and reproducibility of PFAS soil Certified Reference Material (CRM) is presented for both post extraction workflows.

Materials & Instrumentation

- Extract in disposable centrifuge tubes
- CRM: ERA (a Waters™ Company) per- and polyfluoroalkyl substances in soil (item #604)
- Internal standard: Wellington Mass-Labelled PFAS Extraction Standard
- n=4 for all samples extracted

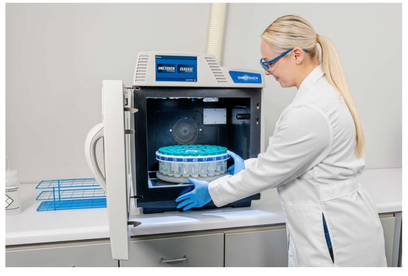

Figure 1. MARS 6 with 40-place Centrifuge Tube Turntable

Method

1. Weigh 5 g sample + 50 µL internal standard into centrifuge tube and allow to equilibrate for 30 minutes.
2. Add 20 mL 0.3% methanolic ammonium hydroxide to centrifuge tube and cap.
3. Vortex to mix.
4. Place in MARS 6™ turntable. Enter the following parameters in the “Method” screen: Temperature: 65 °C, Ramp Time: 15 minutes, Hold Time: 15 minutes. Press “Start”. The system will automatically perform the programmed method and allow the samples to cool upon completion.
5. Centrifuge sample and extract @ 2800 RPM for 10 minutes.
6. Remove an aliquot of extract for dilution analysis. Remaining extract is decanted for SPE cleanup following the cleanup procedure in EPA Method 1633.
7. Analysis on Waters Xevo TQ Absolute™.

Results

Table 1. Acceptable PT Performance for CRM Soil Extracts with or without SPE Cleanup

Compound	Certified Value ng/L	Min	Max	SPE Avg	Dilution Avg
ADONA	261	130	390	181	181
4:2 FTS	248	124	371	178	189
GenX	458	229	688	340	344
PFBA	573	286	856	410	406
PFDoDA	380	190	569	276	286
PFHpS	496	248	744	368	369
PFHxS	294	147	439	216	229
FOSA	156	78.1	233	121	133
PFOS	508	254	756	430	452
PFUnDA	208	105	311	156	157

Table 2. Percent Recoveries and Percent RSD of Extracted Soil CRM with or without SPE Cleanup

Compound	% Recovery with SPE	% RSD	% Recovery w/o SPE	% RSD
ADONA	69	19	69	19
4:2 FTS	72	19	76	25
GenX	74	19	75	19
PFBA	72	16	71	16
PFDoDA	73	19	75	14
PFHpS	74	17	74	16
PFHxS	74	19	78	15
FOSA	77	13	85	13
PFOS	85	18	89	17
PFUnDA	75	16	75	18

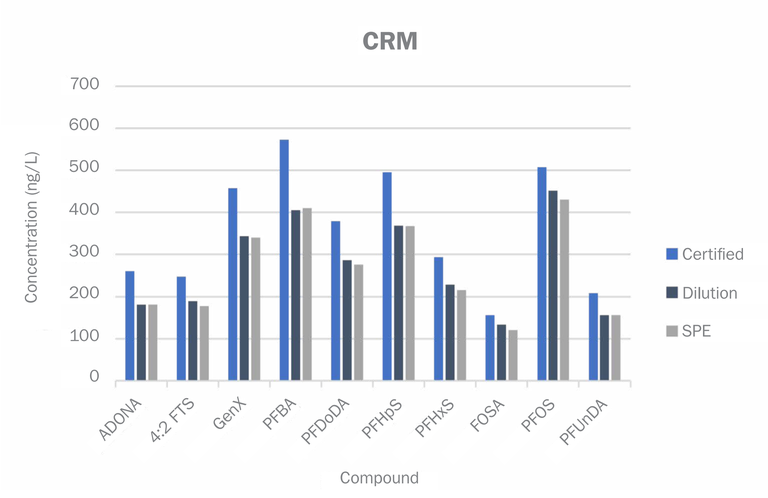

Figure 2. Recoveries for Extracted Soil CRM Compared to Certified Value

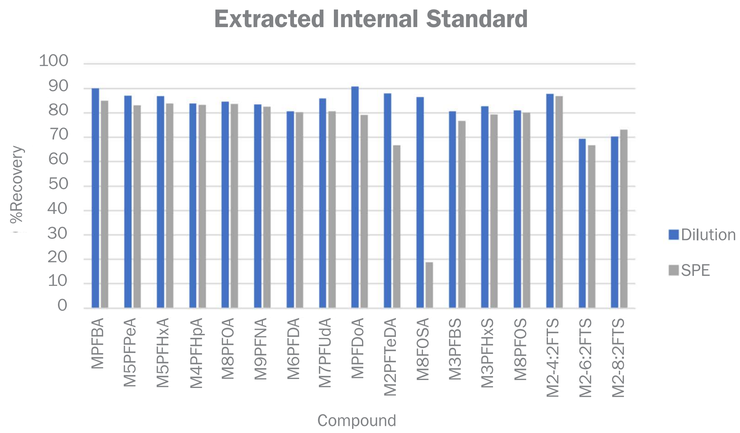

Figure 3. Recoveries of Extracted Internal Standard

Figure 4. Extracted Soil CRM

Conclusion

The MARS 6, equipped with a 40-place centrifuge turntable, was able to successfully extract PFAS from a soil CRM. The entire extraction process, for a batch of 40 samples, took under one hour, enabling a rapid and efficient high-throughput PFAS extraction from solid sample matrices. Furthermore, it was shown that comparable results were obtained with and without the use of SPE cleanup. This method, utilizing a batch microwave system, offers a simple solution for PFAS testing of solid matrices that can easily be integrated into any laboratory workflow.

Acknowledgments

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