

Determination of PFCs in Soil and Sewage Sludge

SpeedExtractor E-916:

Determination of Perfluorinated Compounds (PFCs) in Soil and Sewage Sludge using the SpeedExtractor E-916

Perfluorinated Compounds (PFCs) are fluorocarbons used in many commercial and domestic applications. They are persistent pollutants and are therefore monitored worldwide. Twelve PFC congeners were extracted from soil and sewage sludge samples using the SpeedExtractor E-916 and an ASE® 200 system (Thermo Scientific Dionex). The PFC congeners were analyzed by HPLC-ESI-MS/MS. The data showed that the SpeedExtractor E-916 delivered extractions equivalent to those obtained with the ASE® 200

1. Introduction

PFCs are very stable chemicals and accumulate in the environment. Perfluorooctane sulfonate (PFOS), which belongs to the group of PFCs, was used as a fabric protector numerous stain repellent. PFOS was banned by the Stockholm Convention in 2009 [1].

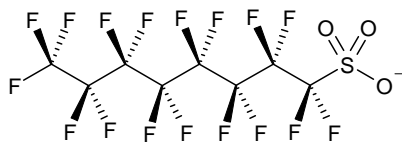


Figure 1: Chemical structure of PFOS

This application note describes the extraction of PFCs from soil and sewage sludge samples using the SpeedExtractor E-916.

2. Experimental

The soil and sludge samples were extracted using the SpeedExtractor E-916 method described below and an established ASE® 200 method [2].

Instrumentation: SpeedExtractor E-916 with 40 mL cells, HPLC Alliance 2695 (Waters) coupled with a Quattro-LC mass spectrometer (Waters).

Samples: 2 soil samples and 1 sewage sludge sample provided by the Fraunhofer Institute IVV, Germany.

Approx. 1 g of soil or sewage sludge sample was mixed with silica gel and filled into the extraction cells. ¹³C-labeled and ¹⁸O-labeled internal standards were added to the mixture and extracted according to the parameters given in Table 1.

Table 1: Extraction method of the SpeedExtractor E-916

Temperature	80°C
Pressure	100 bar
Solvent	Methanol
Cells	40 mL
Vials	240 mL
Cycles	4
Heat-up	1 min
Hold	5/20/20/20 min
Discharge	2 min
Flush with solvent	2 min
Flush with gas	3 min

The extracts were purified by SPE using Oasis WAX cartridges (Waters). The cleaned extracts were then subjected to HPLC-ESI-MS/MS analysis. Quantification was done by the isotope dilution method.

3. Results

The analytical results based on two extractions with the SpeedExtractor E-916 and one extraction with the ASE® 200 are displayed in Table 2. The results obtained with both instruments are very similar.

Table 2: Concentrations of PFCs in ppm; E-916 (n=2) and ASE (n=1)

		PFTeA	PFPeA	PFHxA	PFHpA	PFOA	PFNA
Soil 1	E-916	<0.5	<0.5	<0.5	<0.5	2.8	0.2
	ASE®	<0.5	<0.5	<0.5	<0.5	1.7	0.2
Soil 2	E-916	<0.5	<0.5	<0.5	<0.5	2.1	<0.5
	ASE®	<0.5	<0.5	<0.5	<0.5	1.5	<0.5
Sludge	E-916	<0.5	<0.5	2.2	1.7	17.7	0.6
	ASE®	<0.5	<0.5	2.5	1.0	15.5	0.9
		PFDA	PFUdA	PFDoA	PFHxA	PFOS	PFDS
Soil 1	E-916	0.2	1.2	<0.5	<0.5	0.4	0.5
	ASE®	0.2	<0.5	2.3	<0.5	0.4	<0.5
Soil 2	E-916	0.2	1.4	<0.5	<0.5	0.5	<0.5
	ASE®	0.2	<0.5	0.7	<0.5	0.6	<0.5
Sludge	E-916	8.0	3.2	9.5	<0.5	1065	29.3
	ASE®	7.4	2.6	4.8	<0.5	1043	24.8

4. Conclusion

Twelve PFC congeners were extracted from soil and sewage sludge samples with the SpeedExtractor E-916 and an ASE® 200 system. The results show that the SpeedExtractor delivered extractions equivalent to those obtained with the ASE® 200.

5 Acknowledgement

We sincerely thank the Fraunhofer Institute for Process Engineering and Packaging IVV, Freising, Germany.

6. References

- [1] Stockholm Convention on Persistent Organic Pollutants, COP4, 4-9 May 2009
- [2] Cleres, S.; Gruber, L.; Lastennet, G.; Schlummer, M.; Wolz, G. "Parallel Pressurized Solvent Extraction of PCDD/F, PBDE and PFC from Soil, Sludge and Sediment Samples" 29th International Symposium on Halogenated Persistent Organic Pollutants, Beijing, China, Dioxin 2009.

For more details see Application Note 013/2009