

Extraction of Dioxins and Furans from Soil

Extraction system E-916:

Determination of Dioxins and Furans in Soil using the SpeedExtractor E-916

PCDDs and PCDFs were extracted from a soil sample with the SpeedExtractor E-916 and an ASE[®] 200 system (Thermo Scientific Dionex) according to the U.S. EPA Method 3545A [1]. Dioxin and furan congeners were determined by GC-HRMS. Data showed that the SpeedExtractor E-916 delivered extractions equivalent to those obtained with the ASE[®] 200.

1. Introduction

Polychlorinated dibenzo dioxins (PCDDs) and polychlorinated dibenzo furans (PCDFs) are persistent organic pollutants (POPs) banned by the Stockholm Convention and they are therefore monitored worldwide. They are unwanted by-products of combustion released by chemical manufactures and municipal and industrial waste incinerators.

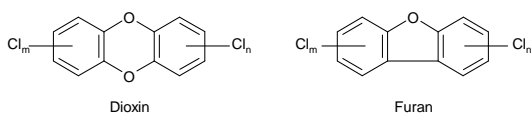


Figure 1: Chemical structure of dioxins and furans.

This application note describes the procedure used to extract PCDDs and PCDFs from a soil sample using the SpeedExtractor E-916 in accordance with the U.S. EPA Method 3545A [1].

2. Experimental

The soil sample was extracted in parallel using the SpeedExtractor E-916 and an established ASE[®] method [2].

Instrumentation: SpeedExtractor E-916 with 10 mL cells, Rotavapor[®], HP 5890 high resolution GC coupled with a MAT-90 Finnigan mass spectrometer (Thermo).

Sample: Approx. 1 g of soil sample was mixed with sand and filled into the extraction cells. After addition of the ¹³C-labeled PCDD/PCDF internal standards a three-fold extraction was performed using the parameters given in Table 1.

Table 1: Extraction method of the SpeedExtractor E-916

Temperature	130°C
Pressure	100 bar
Solvent	Toluene
Cells	10 mL
Vials	240 mL
Cycles	4
Heat-up	1 min
Hold	5 / 10 / 10 / 10 min
Discharge	2 min
Flush with solvent	2 min
Flush with gas	2 min

The extracts were cleaned-up by flash chromatography on silica gel and alumina and the PCDDs and PCDFs were analyzed by GC-HRMS using isotopic dilution method.

3. Results

The analytical results based on triplicate determination with the SpeedExtractor E-916 and replicate determination with the ASE[®] 200 are displayed in Table 2. The data are comparable. Relative standard deviations (RSDs) ranged from 2 to 29%. The relative standard deviation of the WHO-TEQ was calculated to be 6% for the SpeedExtractor E-916.

Table 2: Mean values and RSDs for dioxins and furans

	SpeedExtractor E-916		ASE [®] 200	
	Content [pg/g]	RSD [%]	Content [pg/g]	RSD [%]
WHO-TEQ	476	6	424	4
OCDD	2'003	10	1'509	17
OCDF	2'006	2	1'191	23
1,2,3,4,6,7,8-HpCDD	672	5	519	7
1,2,3,4,7,8,9-HpCDF	529	5	484	15
1,2,3,4,6,7,8-HpCDF	2'807	11	2'068	13
1,2,3,7,8,9-HxCDD	33	9	39	3
1,2,3,6,7,8-HxCDD	58	5	54	3
1,2,3,4,7,8-HxCDD	35	14	32	12
1,2,3,7,8,9-HxCDF	190	10	201	1
2,3,4,6,7,8-HxCDF	276	21	208	2
1,2,3,6,7,8-HxCDF	311	14	265	15
1,2,3,4,7,8-HxCDF	657	9	557	9
1,2,3,7,8-PeCDD	47	4	51	5
2,3,4,7,8-PeCDF	370	15	325	18
1,2,3,7,8-PeCDF	504	2	439	6
2,3,7,8-TCDD	7	29	7	5
2,3,7,8-TCDF	154	17	158	11

4. Conclusion

The SpeedExtractor E-916 delivers extractions equivalent to those obtained with the ASE[®] 200 providing high recovery and high reproducibility.

5. Acknowledgments

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

5. References

- [1] U.S. EPA Method 3545A, Pressurized Fluid Extraction (PFE)
- [2] Cleres, S. et al., "Parallel Pressurized Solvent Extraction of PCDD/F, PBDE and PFC from Soil, Sludge and Sediment Samples" 29th International Symposium on Halogenated Persistent Organic Pollutants, 2009, Beijing, China

For more details see Application Note 012/2009