

## Extraction of PBDEs from Sediment Samples

Extraction system E-914:

Determination of PBDEs in Sediment Samples using the SpeedExtractor E-914

Polybrominated diphenyl ethers (PBDEs) are flame retardants present in many domestic items. They are monitored worldwide because of health concerns. In this application note, PBDE congeners were extracted from two sediment samples (certified sample and sediment core from Lake Maggiore) according to EPA 3545A [1] using the SpeedExtractor E-914 and determined by GC-MS/GC-ECD. The results were comparable to the certified values and showed high reproducibility and recovery.

### 1. Introduction

PBDEs are brominated flame retardants (Figure 1). They are monitored worldwide because of health concerns. Bay and estuary sediments store toxic compounds and are therefore very appropriate sites to evaluate the contamination level of PBDEs in the environment.

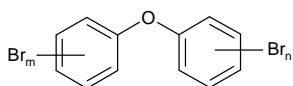


Figure 1: Chemical structure of PBDEs

This application note describes the extraction of PBDEs from two sediment samples using the SpeedExtractor E-914: one certified sediment sample BROCO-02 (RIVO, The Netherlands), one real sample from lake Maggiore, Italy. The PBDE congeners were determined by GC-MS/GC-ECD.

### 2. Experimental

Instrumentation: SpeedExtractor E-914 with 40 mL cells, Trace 2000 GC / PolarisQ ion-trap mass spectrometer (Thermo), Trace GC Ultra / ECD-40 detector (Thermo).

Sample: Certified reference material (CRM) BROCO-02 provided by the Research Institute for Fisheries (RIVO), The Netherlands; and sediment sample collected from Pallanza Bay, Lake Maggiore, Italy.

The extraction of both samples was performed four-fold using the parameters given in Table 1.

Table 1: Extraction method of the SpeedExtractor E-914

Temperature	100°C
Pressure	100 bar
Solvent	<i>n</i> -hexane 75%, acetone 25%
Cells	40 mL
Vials	240 mL
Cycles	1
Heat-up	3 min
Hold	13 min
Discharge	2 min
Flush with solvent	1 min
Flush with gas	2 min

The extract was concentrated and cleaned on a multi-layer silica gel/florisil column. The determination of tri- to

hepta-BDEs was performed by GC-MS. The congener BDE-209 was determined by GC-ECD.

### 3. Results

The mean values obtained for BROCO-02 are displayed in Table 2. They are comparable to the certified values. The calculated RSDs are generally below 20%. The recoveries of the <sup>13</sup>C-labeled internal standards fall in the range of 69-97% (data shown in Application Note 010/2009).

Table 2: Mean values (n=4) for eleven PBDE congeners

PBDE	SpeedExtractor E-914		Certified values
	Mean [ng/g]	RSD [%]	Mean [ng/g]
BDE-28/33	0.52	23	0.63
BDE-47	10.7	8	10.1
BDE-49	2.63	8	2.75
BDE-66	0.18	11	0.29
BDE-85	0.68	10	0.66
BDE-99	12.9	12	14.2
BDE-100	3.03	13	3.04
BDE-153	1.24	7	1.93
BDE-154	1.46	3	1.71
BDE-183	0.39	15	0.45
BDE-209	1'207	8	1'164

The mean concentrations obtained for the sediment sample from Pallanza Bay are displayed in Table 3. They range from 0.43 to 3.6 ng/g with RSDs below 15%.

Table 3: Mean values (n=4) for 4 PBDE congeners

PBDE	Mean [ng/g]	RSD [%]
BDE-47	0.43	14
BDE-99	0.43	10
BDE-100	0.16	13
BDE-209	3.60	14

### 4. Conclusion

The extraction of PBDEs with the SpeedExtractor E-914 delivered reliable and reproducible results. This extraction method requires an overall time of 26 min and consumes approx. 30 ml of solvent *per* position.

### 5. Acknowledgement

We sincerely thank the CNR Water Research Institute in Italy, for their help in developing of this application note.

### 6. References

[1] U.S. Environmental Protection Agency. Method 3545A, Pressurized Fluid Extraction (PFE)

For more detailed information refer to Application Note 010/2009