

Application Note

009/2009



SpeedExtractor E-916

**Extraction of Sediment using the
SpeedExtractor E-916 for the Determination of
Polychlorinated Biphenyls (PCB)**

Extraction of Sediment using the SpeedExtractor E-916 for the determination of Polychlorinated Biphenyls (PCBs)

PCBs were used as coolants and dielectric fluid in transformers, as flame retardants, hydraulic fluids and additives in plastics and for many other applications until the 1980s. They are toxic, persistent and bioaccumulate in terrestrial and aquatic biosystems and due to atmospheric effects they are ubiquitous in the environment. Sediment samples from a round robin program (SETOC 2008/4, Wepal, Wageningen University) were extracted according to EPA 3545A [1] with the Buchi SpeedExtractor E-916 and analyzed by GC-MS. The results correspond to the values found by the round robin testing and show high recovery and low variation.

Introduction

Polychlorinated Biphenyls (PCB) are a class of organic compounds whose biphenyl skeleton is substituted with 1-10 chlorine atoms. To simplify description they are usually specified with a number from 1 to 209.

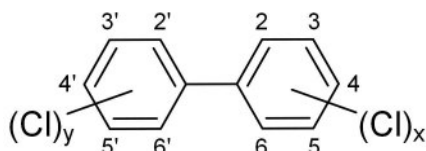


Figure 1: Formula of Polychlorinated Biphenyls

Until the 1980s PCBs were used as coolants and dielectric fluid in transformers, as flame retardants, hydraulic fluids and additives in plastics and for many other applications. Today, production and use are banned nearly worldwide.

Experimental

Instrumentation: SpeedExtractor E-916 with 20 ml cells, GC-MS Varian 3000

Samples: 3 different samples from the SETOC 2008/4 round robin, Wepal, Wageningen University

Approx. 3 g of dry sample was mixed with 10 g of sand and filled into the extraction cell. After addition of the internal standard solution the samples were extracted using the parameters shown in Table 1. A four-fold extraction was performed. The collected extracts were analyzed by GC-MS.

Table 1: Extraction method PCB

Temperature	100 °C
Pressure	120 bar
Solvent	Acetone 50%, Hexane 50%
Cells	20 ml
Vials	240 ml
Cycles	2
Heat-up	1 min
Hold	10 min
Discharge	2 min
Flush with solvent	1 min
Flush with gas	2 min

Results

The results correspond to the values found by the round robin testing and show good recovery and low variation.

Table 2: Values for PCB in µg/kg, SpeedExtractor: n=4, SETOC round robin: n≥33, RSD in brackets

	Sample 1		Sample 2		Sample 3	
	E-916	SETOC	E-916	SETOC	E-916	SETOC
PCB 28	6.3 (16)	7.1 (12)	8.0 (24)	5.4 (17)	25.1 (25)	20.6 (27)
PCB 52	8.0 (14)	6.7 (35)	3.2 (40)	4.1 (21)	29.5 (8)	30.6 (23)
PCB 101	10.3 (29)	9.2 (21)	11.2 (27)	6.3 (23)	50.4 (13)	47.9 (19)
PCB 118	6.5 (5)	6.0 (15)	6.0 (26)	4.4 (17)	34.5 (7)	29.7 (30)
PCB 153	12.1 (16)	12.6 (10)	12.6 (14)	9.6 (25)	79.9 (4)	74.2 (23)
PCB 138	10.7 (21)	10.9 (16)	9.5 (17)	8.4 (28)	77.6 (3)	74.2 (26)
PCB 180	5.1 (26)	6.0 (28)	6.2 (26)	4.9 (20)	43.9 (26)	45.3 (23)

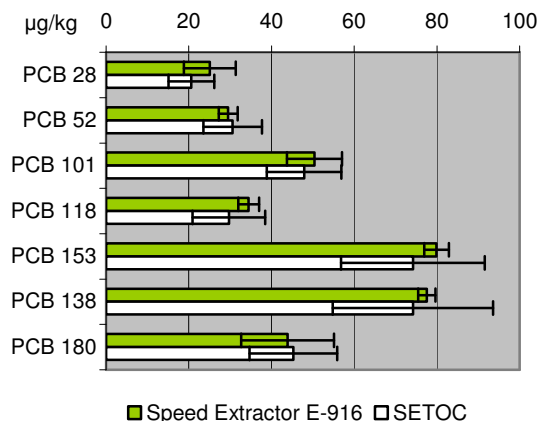


Figure 2: Values for Sample 3 of SETOC 2008/4

Conclusion

Extraction of sediments with the SpeedExtractor E916 is a reliable and powerful method for the extraction of PCBs in sediment.

References

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

SpeedExtractor E-916 operation manual

For more detailed information refer to Application note 009/2009

1 Introduction

Polychlorinated Biphenyles (PCB) are a class of organic compounds based on biphenyl substituted with 1-10 chlorine atoms. There are 209 different congeners. To simplify description they are usually specified with a number from 1 to 209.

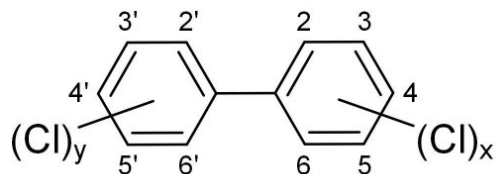


Figure 1: Formula of Polychlorinated Biphenyles

Till the 1980 PCBs were used as coolants and dielectric fluid in transformers, as flame retardants, hydraulic fluids, additives in plastics and for many other applications. They are toxic, persistent and bioaccumulate in terrestrial and aquatic biosystems and are ubiquitous in the environment. Production and use is nowadays banned nearly worldwide.

This application note describes the extraction and determination of 7 PCB in dried sediment samples origin from a round robin program (SETOC 2008/4, Wepal, Wageningen University) according to EPA 3545A [1]. The extraction and quantification was done by Labor Veritas Zurich, an ISO 17025 accredited laboratory.

2 Equipment

- SpeedExtractor E-916, with 20 ml cells
- Analytical balance
- Pipette, Rainin Pos—D 100-1000 μ l
- Volumetric flask 20 ml
- Varian 3000, GC-MS (details see chapter 5.4)

3 Chemicals and Materials

- *n*-Hexane, analytical grade, Fluka (52765)
- Acetone, analytical grade, Scharlau (AC0311005P)
- Acetonitrile, analytical grade, Fluka (66740)
- EPA 525, 525.1 Internal Standard Mix, Supelco (4-8242)

4 Samples

Three samples from the international round robin SETOC 2008/4, Wepal by Wageningen University.

The SETOC round robin is an international round robin organised by Wepal of Wageningen University. The participating laboratories receive dried sediment samples of a fraction < 0.5 mm every three months. They analyse the samples according to their own procedures and for those compounds they are interested in. The results of the determinations are collected and processed at Wageningen University and published every three months under code names [2]. The main goal of the round robin is to give the individual laboratory a tool to compare their results with other laboratories and give feedback about their performance.

5 Procedure

The extraction and analysis of PCB in sediment includes the following steps:

- Preparation of sample
- Preparation of cells
- Addition of internal standard
- Extraction with Speed Extractor E-916
- Quantification by GC-MS

5.1 Preparation of internal standard solution

Add 1 ml of EPA 525, 525.1 Internal Standard Mix into a 20 ml volumetric flask and fill up to volume with acetonitrile.

5.2 Preparation of the cells

- Give approx. 8 g sand into the cells
- Weigh in approx. 3 g of sample using an analytical balance.
- Mix the sample with approx. 10 g of sand.
- Fill in the mixture into the 20 ml cells using a funnel
- Fill up any void volume to 10 mm to the upper filter with sand
- Add 0.6 ml internal standard solution on top of the sand

NOTE: Add the internal standard directly prior to inserting the cells into the heating block.

5.3 Extraction with SpeedExtractor E-916

Carry out the extraction, using the parameters of Table 1. A fourfold extraction of the samples was done.

Table 1: Extraction method for Speed-Extractor E-916

Parameter	Value
Temperature	100 °C
Pressure	120 bar
Solvent	Acetone 50% Hexane 50%
Cells	20 ml
Vials	240 ml
Cycles	2
Heat-up	1 min
Hold	10 min
Discharge	2 min
Flush with solvent	1 min
Flush with gas	2 min

5.4 Quantification by GC-MS

The PCB were quantified with a Varian GC-MS by Labor Veritas, Zurich.

Table 2: Quantification by GC-MS

Type of GC-MS	Varian 3000
Injector	Lauber
Column	Rtx-5MS (Restek); 15 m x 0.25 mm x 0.1 µm
Carrier Gas	Hydrogen at 5 psi
Oven Program	40 °C (hold 1 min); 40 °C-310 °C at 1 0°C/min
Detector	MS (Magnum MAT Finnigan); scan range: 100-400 m/z

6 Results

The extraction by SpeedExtractor E-916 provides comparable results to the results of the SETOC round robin (Table 3 and Figure 2 to 4). In the majority of cases the recovery achieved with the SpeedExtractor is better than the average of the results achieved by the laboratories participating in the round robin. In addition, low variation between the four extractions of the same sample was found.

The total extraction time was about 40 min and approx. 50 ml of solvent per cell was used.

Table 3: SETOC 2008/4, Results of the fourfold extraction by SpeedExtractor E-916 and the SETOC Round Robin

	Sample 1				Sample 2				Sample 3			
	E-916		SETOC		E-916		SETOC		E-916		SETOC	
	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
	µg/kg	%	µg/kg	%	µg/kg	%	µg/kg	%	µg/kg	%	µg/kg	%
PCB 28	6.3	16	7.1	12	8.0	24	5.4	17	25.1	25	20.6	27
PCB 52	8.0	14	6.7	35	3.2	40	4.1	21	29.5	8	30.6	23
PCB 101	10.3	29	9.2	21	11.2	27	6.3	23	50.4	13	47.9	19
PCB 118	6.5	5	6	15	6.0	26	4.4	17	34.5	7	29.7	30
PCB 153	12.1	16	12.6	10	12.6	14	9.6	25	79.9	4	74.2	23
PCB 138	10.7	21	10.9	16	9.5	17	8.4	28	77.6	3	74.2	26
PCB 180	5.1	26	6.0	28	6.2	26	4.9	20	43.9	26	45.3	23

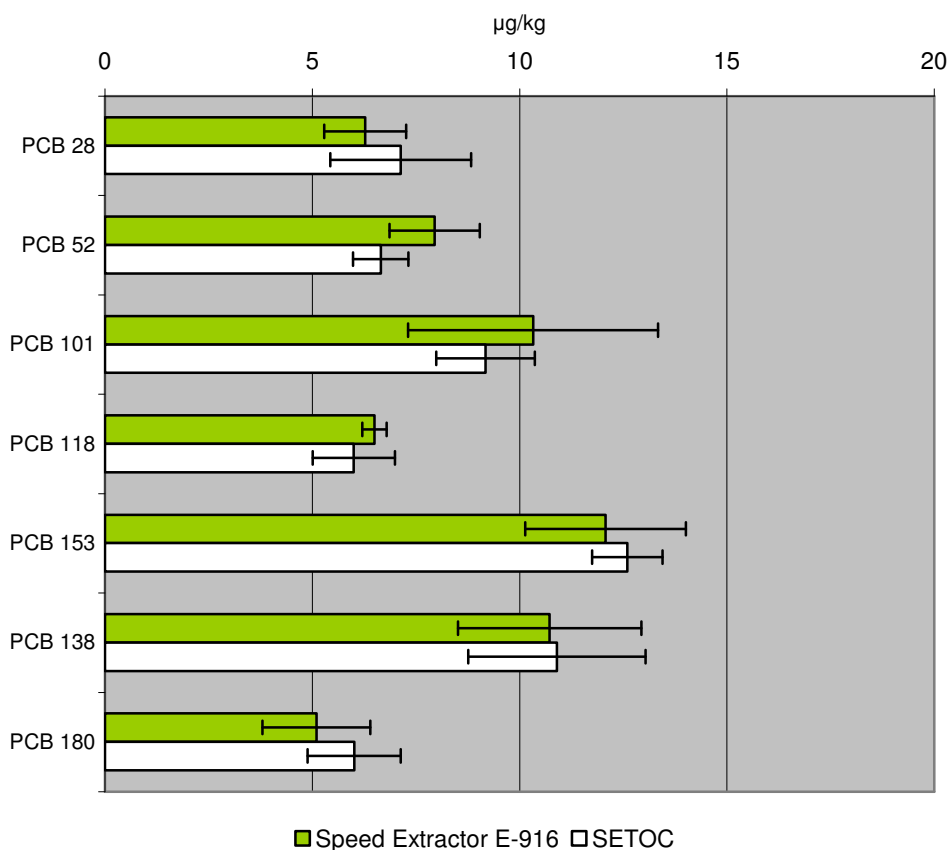


Figure 2: SETOC 2008/4, Sample 1

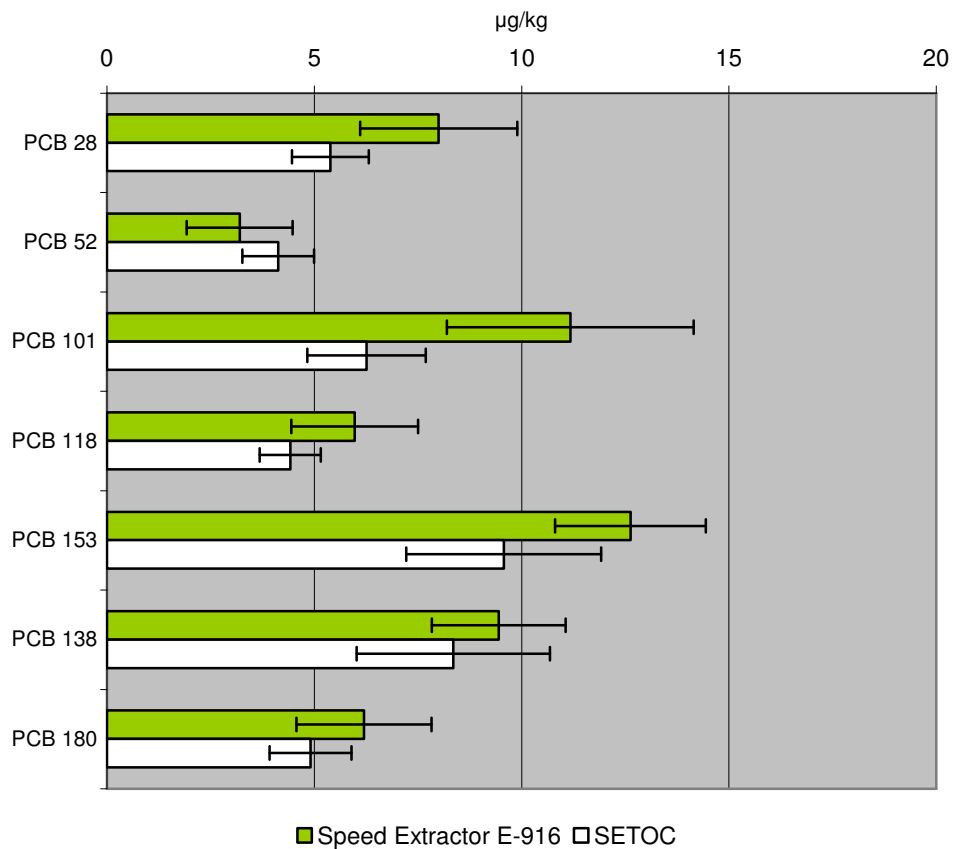


Figure 3: SETOC 2008/4, Sample 2

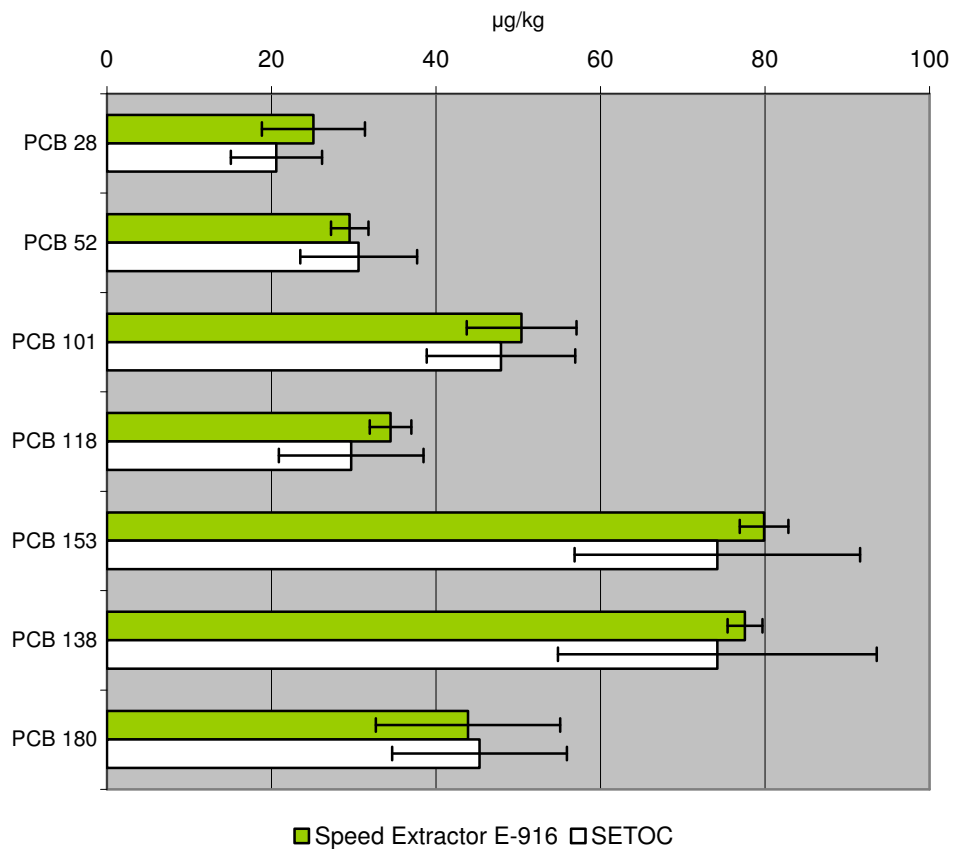


Figure 4: SETOC 2008/4, Sample 3

7 Conclusion

The method presented in this application note demonstrates that the extraction by SpeedExtractor E-916 is a fast and reliable way to extract PCB in sediment. The extraction with the SpeedExtractor E-916 is equivalent to the different established methods as Soxhlet, ultrasonic extraction and ASE used in the SETOC round robin.

8 Acknowledgements

We greatly acknowledge Labor Veritas Zurich, Mr. Peter Egli for his support for the development of this application note.

9 References

- [1] U.S. Environmental Protection Agency. Method 3545A, Pressurized Fluid Extraction (PFE)
- [2] SETOC Round Robin, www.wepal.nl/website/products/SETOC.htm
Operation manual SpeedExtractor E-916

