

Extraction of Sediment for the Determination of PCBs

SpeedExtractor E-916:

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 and additives in plastics and for many other applications until the 1980s. They are toxic, persistent and bioaccumulate in terrestial and aquatic biosystems and due to atmospheric effects they are ubiquitous in the environment. Sediment samples from a round robin program were extracted according to EPA 3545A with the BUCHI SpeedExtractor E-916 and analyzed by GC-MS. The results correspond to the values found by the round robin testing showing high recovery with low variation.

1. Introduction

Poychlorinated Biphenyles (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.

$$(CI)_y$$
 $\xrightarrow{5'}$ $\xrightarrow{6'}$ $\xrightarrow{6}$ $\xrightarrow{5}$ $(CI)_x$

Figure 1: Formula of Polychlorinated Biphenyles

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.

2. 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

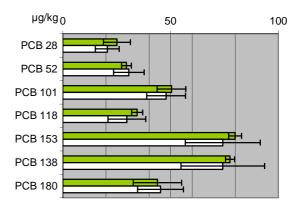
Temperature	100 °C		
Pressure	120 bar		
Solvent	Acetone 50%, Hexane 50%		
Cells	20 mL		
Vials	240 mL		
Cycles	2		
Heat-up	1 min		
Hold	10 min		
Discharge	2 min		
Flush with solvent	1 min		
Flush with gas	2 min		

3. Results

The determined PCB concentrations are shown in Table 2 and Figure 2. 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	7.1	8.0	5.4	25.1	20.6
	(16)	(12)	(24)	(17)	(25)	(27)
PCB 52	8.0	6.7	3.2	4.1	29.5	30.6
	(14)	(35)	(40)	(21)	(8)	(23)
PCB 101	10.3	9.2	11.2	6.3	50.4	47.9
	(29)	(21)	(27)	(23)	(13)	(19)
PCB 118	6.5	6	6.0	4.4	34.5	29.7
	(5)	(15)	(26)	(17)	(7)	(30)
PCB 153	12.1	12.6	12.6	9.6	79.9	74.2
	(16)	(10)	(14)	(25)	(4)	(23)
PCB 138	10.7	10.9	9.5	8.4	77.6	74.2
	(21)	(16)	(17)	(28)	(3)	(26)
PCB 180	5.1	6.0	6.2	4.9	43.9	45.3
	(26)	(28)	(26)	(20)	(26)	(23)



■Speed Extractor E-916 ■SETOC

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

4. Conclusion

Extraction of sediments with the SpeedExtractor E-916 is a reliable and powerful method for the determination of PCBs in sediment.

5. References

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

For more detailed information refer to Application note 009/2009