

PCBs and Phthalates in Waste Polymer Samples

SpeedExtractor E-916:

Determination of Polychlorinated Biphenyls (PCBs) and Phthalates in Waste Polymer Samples

Polychlorinated biphenyls (PCBs) and phthalates are polymer additives present in a myriad of industrial and domestic applications. Due to health concerns PCB and phthalate levels are monitored worldwide. Here PCBs and phthalates were extracted from two waste polymer samples intended for mechanical recycling using the SpeedExtractor E-916 and an ASE[®] system. The PCB and phthalate congeners were determined by GC-MS. Data show that the SpeedExtractor E-916 delivered extractions equivalent to those obtained with the ASE[®].

1. Introduction

PCBs and phthalates are polymer additives present in a myriad of industrial and commercial applications. Because of health concerns, many countries banned the production of PCBs and restricted the use of phthalates. Today, these chemicals are monitored worldwide.

This application note describes the extraction of PCBs and phthalates from two waste polymer samples intended for mechanical recycling using the SpeedExtractor E-916. This application meets requirements of U.S. EPA Method 3545A [1].

2. Experimental

Two polymer samples were extracted applying the SpeedExtractor and a commonly used ASE[®] method [2].

Instrumentation: SpeedExtractor E-916 with 20 mL cells, GC-MS QP 5000 (Shimadzu) for PCBs, GC-MS TSQ 7000 (Thermo) for phthalates, Ultra Centrifugal Mill (Retsch).

Sample: Two waste polymer samples ground and sieved to < 0.7 mm.

About 0.6 g of ground polymer was mixed with pumice stone and filled into the extraction cell. After addition of ¹³C-labeled internal PCB standards and 2 deuterated internal phthalate standards the samples were extracted in duplicate using the parameters given in Table 1. The extraction process required 45 min and approx. 40 mL of solvent were consumed per position.

Table 1: Extraction method of the SpeedExtractor E-916.

Temperature	80°C
Pressure	100 bar
Solvent	n-hexane 90 %, 2-propanol 10 %
Cells	20 mL
Vials	240 mL
Cycles	3
Heat-up	1 min
Hold	5 min
Discharge	2 min
Flush with solvent	1 min
Flush with gas	0.4

The extracts were cleaned up on a chromatography column containing acid and basic modified silica. PCBs and phthalates were determined by GC-MS.

3. Results

The mean concentrations of 7 PCB and 3 phthalate congeners are displayed in Tables 2 and 3. The data obtained by SpeedExtractor E-916 and ASE[®] are comparable.

Table 2: Concentrations (ppb) and RSDs (% in brackets) for PCBs, (n=2).

PCBs	Sample 1		Sample 2	
	E-916	ASE [®]	E-916	ASE [®]
2,4,4'-TriCB	6'192 (3)	5'436 (3)	1'214 (1)	1'051 (1)
2,2',5,5'-TetraCB	1'172 (1)	1'063 (1)	241 (2)	230 (2)
2,2',4,5,5'-PentaCB	222 (1)	217 (4)	110 (1)	115 (3)
2,2',3,4,4',5'-HexaCB	98.0 (1)	112.8 (19)	90.3 (2)	95.6 (4)
2,2',4,4',5,5'-HexaCB	79.4 (6)	95.2 (22)	82.7 (10)	85.3 (3)
2,2',3,4,4',5,5'-HeptaCB	18.5 (1)	21.7 (34)	21.1 (5)	19.7 (4)
DecaCB	0.5 (120)	0.4 (27)	0.6 (16)	0.5 (20)

Table 3: Concentrations (ppm) and RSDs (%) for phthalates, (n=2).

PCBs	Sample 1		Sample 2	
	E-916	ASE [®]	E-916	ASE [®]
DiBP Diisobutylphthalate	8.56 (22)	7.84 (5)	21.4 (1)	22.1 (1)
DBP Dibutylphthalate	5.72 (10)	5.82 (7)	22.7 (3)	21.4 (4)
DEHP Diethylhexylphthalate	1'257 (1)	1'272(20)	1'278 (13)	1'180 (4)

4. Conclusion

The results show that the SpeedExtractor E-916 delivers extractions equivalent to those obtained with the ASE[®] system and demonstrate that this extraction procedure is perfectly suitable for the extraction of PCBs and phthalates in polymer samples.

5. Acknowledgement

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

5. References

- [1] U.S. Environmental Protection Agency. Method 3545A, Pressurized Fluid Extraction (PFE).
- [2] Cleres *et al.*, "Determination of PCBs and Phthalates in Waste Polymer Samples Intended for Mechanical Recycling", 29th International Symposium on Halogenated Persistent Organic Pollutants, 2009.

For more details see Application Note 011/2009