New Options for Solvent Extraction of Polychlorinated Biphenyls from Environmental Matrices

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Introduction

Polychlorinated Biphenyls (PCBs) are man-made compounds that were formerly used in a variety of products. Even though the use of PCBs is now banned due to their toxicity, they can still be found in the environment. PCBs are regulated by the Environmental Protection Agency (EPA); a recent rule change allows EPA methods 3545A Pressurized Fluid Extraction and 3546 Microwave Extraction to be used for the extraction of PCBs from a variety of environmental matrices to meet the requirements in 40 CFR 761.[1] In this study, the EDGE® automated solvent extraction system as well as the MARS 6™ Extraction microwave system, following EPA method 3545A and 3546, respectively, are both shown to effectively extract PCBs from soil with exceptional percent recoveries and reproducibility.

Materials and Methods

1. Weigh 15 g Ottawa sand into Q-Cup® or MARSXpress™ Plus vessel.
2. Spike with 50 µL of PCB Congener mix, Method 8082A (100 µL/mL)* for pre-spiked samples.
3. Extract using EDGE or MARS 6 Extraction.
4. Spike with 50 µL of PCB Congener mix, Method 8082A (100 µL/mL)* for post-spiked samples.
5. Evaporate extract to below 1 mL using heat and N_2 flow.
6. Add 50 µL of 2,4,5,6-Tetrachloro-m-xylene Internal Standard, (200 µg/mL)*.
7. Reconstitute in hexane to 1 mL for analysis.

MARS 6 Extraction Procedure

1. Add 30 mL of (1:1) hexane/acetone to MARSXpress Plus vessel containing sample.
2. Vortex MARSXpress Plus vessel for 30 seconds and place in turntable.
3. Place turntable (with up to 24 MARSXpress Plus vessels) in MARS 6 Extraction system.
4. Run One Touch™ EPA 3546 method:
   Temperature: 115 °C
   Ramp Time: 15:00 (mm:ss)
   Hold Time: 15:00 (mm:ss)
5. Decant extract and rinse MARSXpress Plus vessel with 30 mL of (1:1) hexane/acetone and add to decant.
6. Filter extract.
7. Filtered extracts are ready for post-spike addition or concentration.

*available from Restek

Materials and Methods (Cont.)

EDGE Extraction Procedure

EDGE will perform 12 automated solvent extractions in sequence.

1. Place Q-Cups with samples ready for extraction in rack and rack into the EDGE system.
2. Load the EDGE method below and press start.
   EDGE Method (EPA 3545A):
   Q-Disc®: S1
   Cycles: 4
   Extraction Solvent: (1:1) hexane/acetone
   Top Add: 15 mL
   Temperature: 115 °C
   Hold Time: 01:00 (mm:ss)
3. Remove sample and/or rack. The collected extracts are cool and filtered, ready for post-spike addition or concentration.

Results

Table 1. Extraction of PCBs from Ottawa Sand

	EDGE		MARS 6 Extraction	
Compound	**% Recovery**	**% RSD**	**% Recovery**	**% RSD**
PCB 1	80.05	14.73	99.35	10.28
PCB 5	95.59	0.75	102.60	7.74
PCB 18	96.99	1.28	102.77	5.64
PCB 31	93.47	2.14	102.45	5.66
PCB 44	94.52	2.29	103.42	3.55
PCB 52	94.13	2.30	103.65	3.94
PCB 66	93.58	3.15	103.77	2.81
PCB 87	94.92	2.94	103.40	1.81
PCB 101	94.86	2.70	103.61	2.89
PCB 110	93.23	3.26	103.31	2.22
PCB 138	96.52	2.89	103.32	4.33
PCB 141	95.21	3.17	108.71	11.23
PCB 151	95.21	2.67	103.28	1.66
PCB 153	96.49	2.74	102.53	3.42
PCB 170	93.57	4.36	100.22	5.32
PCB 180	94.98	4.44	101.95	5.08
PCB 183	95.09	3.90	101.62	4.28
PCB 187	96.00	3.57	102.55	3.92
PCB 206	91.20	9.07	98.69	3.85

All extractions were run in triplicate and % recovery was determined by comparing pre- and post-spiked samples.

Analysis

- System - Agilent 8860 GC with 5977 MSD and 7693 auto sampler
- Column – Agilent HP-5MS UI (30 m x 0.25 mm x 0.25 µm)
- Calibration curve – 1, 2.5, 5, 10, 50, 100 ppm
- Injection Temp – 280 °C
- Carrier Gas – He
- Column Flow – 1.2 mL/min
- Inject Mode – Splitless
- Injection Volume – 1 mL
- Oven Program:

Temperature (°C)	Rate (°C/min)	Hold (mm:ss)	Time (min)
40			0
230	20		9.5
260	5	01:30	17
310	20	03:00	22.5

Conclusions

Both the EDGE, following EPA 3545A, and the MARS 6 Extraction, following EPA 3546, can effectively extract PCBs from soil with exceptional recoveries and reproducibility. These systems give labs the flexibility to use extraction methods that are rapid, simple, and efficient. They not only reduce time of extraction but also reduce solvent usage as well, making both the EDGE (sequential) and MARS 6 Extraction systems (high throughput) great options for any laboratory performing PCB analysis.

References

[1] Environmental Protection Agency. Alternate PCB Extraction Methods and Amendments to PCB Cleanup and Disposal Regulations. 88 FR 59662. 40 CFR 761. Effective February 26, 2024. https://www.federalregister.gov/documents/2023/08/29/2023-17708/alternate-pcb-extraction-methods-and-amendments-to-pcb-cleanup-and-disposal-regulations (Accessed June 18, 2024).

For more information on PCBs and their regulation by the EPA, visit https://www.epa.gov/pcbs.