

Extraction of PCBs from solid waste using Microwave-Assisted Solvent Extraction

Milestone Application Note for Microwave Extraction - 06



This app note will discuss the use of Ethos X Microwave Extraction system utilizing fastEX24 rotor with contact-less temperature sensor and disposable glass vials to treat solid waste for recovery study of polychlorinated biphenyl (PCB) following US EPA Method 3546.

Summary

Solid waste, due to its heterogeneous composition, is a harsh sample to extract for several extraction technologies. Ethos X thanks to the batch, high throughput fastEX24 rotor makes the sample preparation of solid waste easier and reliable. Ethos X is the right solution for this kind of sample since its ability to work with heterogeneous material without any clogging or additional cleaning and maintenance steps.

This application note represents a guideline of microwave-assisted solvent extraction in the extraction of PCBs from solid waste. Milestone Ethos X equipped with fastEX24 rotor benchtop microwave extraction system, fully compliant with US EPA 3546, was used for this study. FastEX 24 rotor works with 145 mL Weflon vessels with disposable glass vials of 100 mL. Thanks to the Weflon construction and the contactless temperature control in all positions, FastEX 24 rotor allows a simultaneous extraction of 24 samples ensuring a perfect temperature uniformity. Built-in methods and app notes provide an unmatched ease of use and low running costs.



Method details

Samples, wet or dried and ground, were weighed directly into the 100-mL extraction glass vials. An aliquot of the surrogate solution were added to the samples just prior to solvent addition. The glass vials were then closed into vessels and the microwave extraction program were started. The samples were extracted using the conditions mentioned above. The extraction procedure so described follows the detailed method provided by U.S. EPA SW-846 Method 3546.

Sample weight (g)	1:1 acetone – CH ₂ Cl ₂ (mL)
Up to 10	25
10 -20	35
20-30	50

After the extraction, samples were filtered on glass fiber filters and sodium sulfate anhydrous and the vials were rinsed with additional solvent aliquots. Extracts and rinsates were collected together.

Microwave program

Step	Time (min)	T2 (°C)	Power (W)
1	00:15:00	110°C	up to 1600 W*
2	00:10:00	110°C	up to 1600 W*

*The power applied depends on the moisture content. Dedicated methods are pre-loaded in the ETHOS X software according to the moisture content.



Quantification

PCBs analyses of the soil extract were performed according to the following method. Injection was through a splitless injector in a GC-MS equipped with VF-17-MS 30 m × 0.25 mm i.d. capillary columns with 5 m guard column. The injector was maintained at 280 °C. The injection was 2µL at 2mL/min flow rate. The oven was hold at 80°C for 2 min, from 80-300°C at 20°C/min than hold for 29 min at 300°C. The detector worked with electron impact chemical ionization mass spectrometer.

Analytical results

Results from extractions of solid waste are shown in table below. The table show the recovery and the RSD (%) for PCBs, content of that matrix. Recovery for all compounds are in the range 70-120% of the recovery obtained with the traditional Soxhlet extraction. The results demonstrate the efficiency of the Ethos X compared to the traditional Soxhlet extraction. Ethos X provides extracts with the lowest solvent usage and significant time compared to all the other extraction techniques.

Recovery of PCBs from solid waste sample (1g) – Ethos X compared to Soxhlet (n=4).

Analyte	Soxhlet (mg/kg)	Ethos X (Recovery % of Soxhlet)	RSD (%)
PCB 28	4.09	88	5.2
PCB 52	3.70	88	4.8
PCB 95	2.46	79	6.2
PCB 99	1.40	73	3.1
PCB 101	3.18	72	2.6
PCB 105	1.22	90	6.4
PCB 114	0.07	85	7.3
PCB 118	2.68	79	2.0
PCB 123	0.07	114	5.6

PCB 126	0.16	118	4.2
PCB 128	0.55	82	3.4
PCB 138	1.79	80	8.3
PCB 146	0.25	116	6.2
PCB 151	0.17	105	7.4
PCB 153	1.46	90	6.1
PCB 156	0.29	110	7.9
PCB 157	0.10	100	6.5
PCB 169	0.45	104	3.1
PCB 170	0.41	78	2.2
PCB 180	0.36	81	7.7
PCB 183	0.20	80	2.3
PCB 187	0.35	100	5.3
PCB 189	0.21	114	4.7
PCB 77+149	2.37	71	4.9
PCB 81+110	7.03	76	6.1

General precautions

Always use hand, eye and body protection when operating with the microwave system.

Conclusion

The ETHOS X enables simultaneous solvent extraction of up to 24 solid waste samples. The use of contactless temperature control ensures high reproducibility and full recovery of PCBs. Ethos X meets the requirements for PCBs analysis as described in US EPA 3546.

*Subject to change without notice.
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