

Extraction of PCBs from sandy loam soil using Microwave-Assisted Solvent Extraction

Milestone Application Note for Microwave Extraction - 01



This app note describes the use of Ethos X Microwave Extraction system utilizing fastEX24 rotor with contact-less temperature sensor and disposable glass vials to extract polychlorinated biphenyl (PCB) from certified soils during a recovery study following US EPA Method 3546.

Summary

Polychlorinated biphenyls (PCBs) are strictly regulated on the state and federal levels. Responsible parties must determine the concentration and extent of contamination to make appropriate decisions regarding remediation of PCBs contaminated soils. This application note represents a guideline of microwave-assisted solvent extraction in the extraction of PCBs from sandy loam soil standard reference material LGC6115. 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 x 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 sandy loam soil are shown in table below. The tables show the recovery and the RSD (%) for PCBs, content of that matrix. Recovery for all compounds are above 70% of the certified standard reference material.

The results demonstrate the efficiency of the Ethos X as sample preparation method for GC analysis. Ethos X provides extracts with the lowest solvent usage and significant time compared to all the other extraction technique.

PCBs recovery (n=4) from 1g sandy loam soil standard reference material (LGC6115)

PCB Cogenener	Certified value (mg/kg)	Ethos X (mg/kg)	Rec. (%)	RSD (%)
PCB 101	93	74	80	1.75
PCB 118	116	86	74	4.94
PCB 138	16	14	88	0.2
PCB 153	19	17	89	3.2
PCB 180	9.6	10	104	2.6

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 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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