

Extraction of PCDD and PCDF in large-volume soil sample using Microwave-Assisted Solvent Extraction

Milestone Application Note for Microwave Extraction - 08



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 dioxins following US EPA Method 3546.

Summary

Polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) are highly toxic organic pollutants, even at very low level. PCDD and PCDF are not manufactured deliberately but are the by-products of the combustion of chemical waste, chemical and pesticide manufacturing, pulp and paper bleaching processes etc. This application note represents a guideline of microwave-assisted solvent extraction in the extraction of PCDD and PCDF from soil sample. 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. The use of disposables prevent the risk of any memory effect. 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 – hexane (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

Dioxins (PCDD and PCDF) analyses of the soil extract were performed according to the following method working with polar and non-polar columns.

Polar columns: Injection was through a split-splitless injector in a GC-MS equipped with 50 m x 0.20 mm i.d. capillary column (5% methylphenylsiloxane, 0.25 µm). The injector was maintained at 280 °C. Interface temperature GC/MS: 300°C.

The detector worked with EI (28 - 34 eV).

Non-polar columns: Injection was through a split-splitless injector in a GC-MS equipped with 50 m x 0.22 mm i.d. capillary column (cyanopropyl siloxane, 0.25 µm). The injector was maintained at 240 °C. Interface temperature GC/MS: 240°C.

The detector worked with EI (28 - 34 eV).

Analytical results

Results from extractions of soil sample are shown in table below.

Recovery for all compounds are in the range 70-120% of the recovery obtained with the traditional Soxhlet extraction.

The results demonstrate the efficiency and the reproducibility of the Ethos X compared to the traditional Soxhlet extraction even in large-sample volume. Ethos X provides extracts with the lowest solvent usage and significant time compared to all the other extraction techniques.

Recovery of PCDD and PCDF from soil sample (10g) – Ethos X compared to Soxhlet (n=4).

Analyte	Soxhlet (ng/kg)	Ethos X (Recovery % of Soxhlet)	RSD (%)
2,3,7,8-TCDD	9.62	91	3.4
1,2,3,7,8-PeCDD	257	84	2.8
1,2,3,4,7,8-HxCDD	692	86	3.1

1,2,3,6,7,8-HxCDD	6003	107	2.1
1,2,3,7,8,9-HxCDD	2761	96	1.9
1,2,3,4,6,7,8-HpCDD	29474	121	2.3
OCDD	28220	113	2.8
2,3,7,8-TCDF	1168	109	1.6
1,2,3,7,8-PcDF	706	86	2.7
2,3,4,7,8-PcDF	1175	66	3.5
1,2,3,4,7,8-HxCDF	2528	94	2.6
1,2,3,6,7,8-HxCDF	541	87	1.9
2,3,4,6,7,8-HxCDF	321	102	3.8
1,2,3,4,6,7,8-HpCDF	1348	96	3.6
1,2,3,4,7,8,9-HpCDF	342	75	2.2
OCDF	596	75	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 soil samples. The use of contactless temperature control ensures high reproducibility and full recovery of PCDD and PCDF. Ethos X meets the requirements for dioxins analysis as described in US EPA 3546.

*Subject to change without notice.
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