Overcome the memory effect in Dioxins extraction with Ethos X powered by fastEX-24.

This paper shows the use of Milestone Ethos X with fastEX-24 technology for extraction of dioxins from environmental matrices, with emphasis to one of the most common challenge of this application: the carryover effect. The unique design of the fastEX-24 rotor with disposable glass vials allows to perform easy and efficient dioxins extraction, and of other organic pollutants, avoiding any memory effect. The fastEX24 simplify the routine pollutants extractions process and provide superior productivity at lower costs.

Introduction

Dioxin is one of the most toxic chemicals known, a serious public health threat. According to the US Environmental Protection Agency (EPA) reports, dioxin and dioxin-like chemicals have been associated to adverse health effects in the population. Dioxin is formed as an unintentional by-product of many industrial processes involving chlorine such as waste incineration, chemical and pesticide manufacturing and pulp and paper bleaching. Dioxin is a general term that describes a group of hundreds of chemicals that are highly persistent in the environment. Dioxins are classify as polychorinated dibenzo-p-dioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxin-like PCBs (DLPCB).

The analysis of this class of pollutants is fundamental for the environmental and human health protections, but due to the persistent nature of that compounds, the sample preparation and analysis is challenging for many extraction techniques. Many solutions are available for the extraction of dioxin such as soxhlet, automated soxhlet, sonication, pressurized liquid extraction and closed microwave vessels. A common and very critical issue in all sample preparation techniques is not only the dioxins extraction, but the subsequent memory effect/ carryover in the extraction cells. The stability of the dioxins requires long and tedious cleaning procedures of the extraction vessels. Ethos X equipped with the unique fastEX24 rotor is specifically designed to overcome the memory effect and cleaning by using disposable glass vials as reaction vessels, which are placed into a pressure reactor. This approach leads to ensure great analytical blanks and completely avoid carryover between runs.

Milestone's new Ethos X benchtop microwave extraction system offers the ability to extract up to 24 samples simultaneously in 40 minutes. The Ethos X with the fastEX 24 rotor is fully compliant with US EPA 3546 (100-115 °C and 50-150 psi). In addition, disposable glass vials can accommodate sample up to 30 grams of sample if needed, thereby improving the limit of quantitation (LOQ) for analysis. This exceeds by far both the throughput and sample size capabilities of all the other automated and not automated techniques, such as pressurized fluid extraction.

Experimental

Instrument

- Milestone Ethos X microwave system equipped with fastEX-24 extraction rotor
- 100 mL disposable glass vials (PN GB00122)
- Gas chromatograph with Mass Spectrometer detector (GC-MS)
- Analytical balance
- Vials for collection of extracts
- Glass funnels for filtration
- Glass fiber filters

Standard and reagents

Pesticide grade or grade solvents and chemicals must be used in all tests. The solvent mixture used was 1:1 acetone –hexane according to the US EPA 3546 method.
Sodium sulfate anhydrous, silica gel and glass wool or paper filter were used in the work up procedure. According to the analytical method surrogate and internal standard could be used.

Sample information

The sandy soil standard reference material BCR®-529 was used for the recovery study on PCDD and PCDF (1).

Analytical Procedure

Samples were weighed directly into the 100-mL extraction disposable glass vials. Solvent and in case, an aliquot of the surrogate solution were added to the glass vials than were closed. According to the moisture content, the best suitable built-in method were choose. The extraction procedure so described follows the detailed method provided by U.S. EPA SW-846 Method 3546.

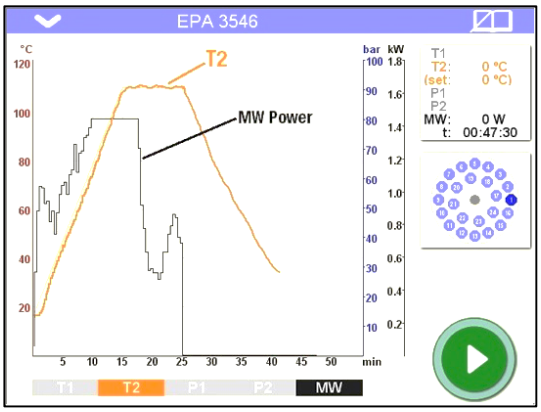
Table 1. Suggested solvent volumes according to the used sample amounts

Sample amount (g)	Solvent mixture (mL)
Up to 10	25
10-20	35
20-30	50

Table 3. Microwave Program

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

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



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.

Quantification

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

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).

Results and Discussion

Beside the recovery study on PCDD and PCDF, this work was aimed to proof the efficacy of the disposable glass vial to avoid any carryover between runs.

The results reported in table Table 2 shown recovery of all the molecules in the range of 80-120% with great reproducibility (RSD%). Table 3 shown the value of the blanks of the following run with new disposable glass vials. The data proof that the fastEX24 designed with disposable glass vial, fully overcome memory effect/ carryover from the entire vessel (all the blanks were below 0.065 µg/kg), ensuring accurate and precise results.

The results demonstrate the efficiency of the Ethos X as sample preparation method for the determination of contaminants. Ethos X provides extracts with the lowest solvent usage and significant lower time compared to all the other extraction techniques, avoiding any memory effect.

Table 2. Recovery (n=4) of PCDD and PCDF from sandy soil standard reference material BCR®-529 (2g).

Analyte	Certified value (µg/kg)	Ethos X (µg/kg)	Recovery (%)	RSD (%)
2,3,7,8-TCDD	4500±0.6	4236	94	3.4
1,2,3,7,8-PeCDD	440±0.05	515	117	2.8
1,2,3,4,7,8-HxCDD	1220±0.21	1298	106	3.1
1,2,3,6,7,8-HxCDD	5400±0.9	4610	85	2.1
1,2,3,7,8,9-HxCDD	3000±0.4	2522	84	1.9
2,3,7,8-TCDF	78±0.013	75	96	2.7
1,2,3,7,8-PeCDF	145±0.028	116	80	3.5
2,3,4,7,8-PeCDF	360±0.07	329	91	2.6
1,2,3,4,7,8-HxCDF	3400±0.5	3402	100	1.9
1,2,3,6,7,8-HxCDF	1090±0.15	1082	99	3.8
1,2,3,7,8,9-HxCDF	22±0.010	18	82	3.6
2,3,4,6,7,8-HxCDF	370±0.05	445	120	2.2

Table 3. Blank values after PCDD and PCDF extraction (from table 2). New disposable glass vials were used in order to proof the absence of memory effect.

Analyte	Blank value (µg/kg))
2,3,7,8-TCDD	0.0185
1,2,3,7,8-PeCDD	0.02122
1,2,3,4,7,8-HxCDD	0.00312
1,2,3,6,7,8-HxCDD	0.02198
1,2,3,7,8,9-HxCDD	0.06412
2,3,7,8-TCDF	0.02524
1,2,3,7,8-PeCDF	0.01939
2,3,4,7,8-PeCDF	0.01889
1,2,3,4,7,8-HxCDF	0.03112
1,2,3,6,7,8-HxCDF	0.00633

1,2,3,7,8,9-HxCDF	0.04043
2,3,4,6,7,8-HxCDF	0.05186

Conclusions

The ETHOS X enables simultaneous dioxins extraction of up to 24 samples (from weighing to filtration steps) in less than 1 hour, offering superior productivity of any other technology in the market. In addition, the use of disposable glass vials fully solve a constant issue in Dioxins determination by avoiding tedious cleaning, memory effect and carryover.
The use of contactless temperature control ensures high reproducibility and full recovery of the target analytes in full compliance with EPA 3546.
Moreover, the above procedure it applies to a wide variety of samples, ensuring reliable extraction also on difficult samples such as solid waste and other environmental samples. The ETHOS X with all its unique features fully addresses the need of environmental laboratories in terms of productivity, ease of use, running costs, and extraction quality.

References

(1) https://www.labmix24.com/files/info/22705.pdf