

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

Milestone Application Note for Microwave Extraction - 05



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 Total Petroleum Hydrocarbons (TPH) 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 TPH 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 – 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

TPHs analyses of the solid waste extract were performed according to the UNI EN 16703 method. Injection was through on column injector in a GC-FID equipped with Select Mineral Oil 15 m × 320 µm i.d. (film 0.1 µm) columns. The injector was maintained at 320 °C. The injection was 1 µL with 2 mL/min flow rate. The oven was hold at 70 °C for 2 min, from, 70-320 °C at 30 °C/min. The FID detector were programmed at flow rates of 400 mL/min air and 30 mL/min H₂, make up 30 mL/min He.

For additional information please contact application@milestonesrl.com

Analytical results

Results from extractions of solid waste are shown in table below. The table show the recovery of TPH with Ethos X compared to the reference Soxhlet method. The recovery was very high and perfectly comparable 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 technique.

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

Analyte	Soxhlet (mg/kg)	Ethos X (Recovery % of Soxhlet)	RSD (%)
TPH	11354 ± 122	111	5.2

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 TPH. Ethos X meets the requirements for TPH analysis as described in US EPA 3546.

Subject to change without notice.

