Extraction of TPH from sediment sample using Microwave-Assisted Solvent Extraction

Milestone Application Note for Microwave Extraction - 03

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 extract Total Petroleum Hydrocarbons (TPH) from certified soils during a recovery study following US EPA Method 3546.

Summary

TPH is a term used to describe a large family of several hundred chemical compounds that originally come from crude oil. Because there are so many different chemicals in crude oil and in other petroleum products, it is useful to measure the total amount of TPH at a site. This application note represents a guideline of microwave-assisted solvent extraction in the extraction of TPH from sediment standard reference material BAM-U015b. 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 soil 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 1µL with 2mL/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 H_2, make up 30mL/min He.

Analytical results

Results from extractions of sandy loam soil are shown in table below. The tables show the recovery and the RSD (%) for TPH, content of that matrix. Recovery for all compounds are above 90% 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.

Semivolatile organics, TPH recovery (n=4) from 1g certified standard reference mineral oil contaminated sediment sample (BAM-U015b).

Analyte	Certified value (mg/kg)	Ethos X (mg/kg)	Recovery (%)	RSD (%)
TPH	920 ± 100	841.8	91.5	2.4

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.
For additional information please contact application@milestonesrl.com