

## Total Petroleum Hydrocarbons in Soil, Sediment and Waste

SpeedExtractor E-916:

Determination of Total Petroleum Hydrocarbons in Soil, Sediment, and Waste Samples using the SpeedExtractor E-916

Total petroleum hydrocarbons (TPH) are a family of several hundred chemical compounds that come from crude oil. This application note describes the extraction of TPH from spiked soil and certified sediment, and waste samples using the SpeedExtractor E-916. The determination of TPH was done by GC-FID. The results were in accordance with the certified values of the CRM samples.

### 1. Introduction

TPH is the general term used to describe a family of several hundred chemical compounds that come from crude oil. TPH are released from industry, commercial, or personal use. They are found in soil and water. Exposure to TPH such as gasoline or diesel fuel can affect human health. Therefore, TPH levels are monitored globally.

This application note describes the extraction of TPH from soil, sediment, and waste samples using the SpeedExtractor E-916. The determination of TPH was performed by GC-FID. This application meets the requirements of the U.S. EPA Method 3545A and the ISO 16703 and EN 14039 Standards [1,2].

### 2. Experimental

Instrumentation: SpeedExtractor E-916 with 40 mL cells

Samples: Pre-extracted soil sample, three certified reference materials (CRM): clay loam, sediment, and contaminated waste material (Figure 1).



Figure 1: Pre-extracted soil (1), clay loam (2), sediment (3), and waste material (4) samples

Approx. 10 g of sample was mixed with diatomaceous earth and filled into the extraction cell. Sample 1 was spiked with standard diesel. The samples were extracted fivefold according to the parameters described in Table 1.

Table 1: Extraction method of the SpeedExtractor E-916

Temperature	100°C
Pressure	100 bar
Solvent	n-hexane 50%, acetone 50%
Cells	40 mL
Vials	240 mL
Cycles	2
Heat-up	1 min
Hold	5 min
Discharge	2 min
Flush with solvent	1 min
Flush with gas	2 min

The extracts were washed with water and dried over sodium sulphate. Then approx. 8 mL of each extract was cleaned up on a column filled with florisil and sodium sulfate. Quantification of TPH was performed by GC-FID.

### 3. Results

The extraction method was validated by the use of spiked soil (Table 2). It gave a good recovery of 90.8% with an rsd of 3.3%, which meets the acceptance criteria of U.S. EPA 8015B [3]. In order to measure the divergence between the values obtained with the SpeedExtractor E-916 and the certified values, the z-score was calculated for each sample [4]. In each case, z was found to be < 2, which means that the mean values do not differ significantly from the certified values.

Table 2: TPH contents [mg/kg], reference values [mg/kg], and z-score

	Spiked Soil	Clay Loam	Sediment	Waste Material
Mean value (n=5)	1634	522	2182	2813
rsd	3.3	7.5	4.4	2.4
Reference value (SD <sub>CRM</sub> )	1800	661 (132)	1820 (192)	3010 (342)
Recovery [%]	90.8			
z score		1.1	1.9	0.58

### 4. Conclusion

The results show that the SpeedExtractor E-916 provided good recoveries and reliable results for the extraction of TPH from solid matrices. In addition, the SpeedExtractor E-916 only uses 70 mL of solvent per sample, and the extraction time is only 30 min for 6 positions.

### 5. Acknowledgement

We sincerely thank Bachema AG, Schlieren, Switzerland for the analytical work.

### 6. References

- [1] US EPA 3545A "Pressurized Fluid Extraction (PFE)"
- [2] ISO 16703 / EN 14039 "Determination of content of hydrocarbon in the range C10 to C40 by gas chromatography"
- [3] US EPA 8015B "Total Petroleum Hydrocarbons (TPH) as Gasoline and Diesel"
- [4] ISO/IEC 43-1: 1997 "Proficiency testing by interlaboratory comparisons"

For more details see Application Note 019/2009