



## **SPE Application Note for Total Petroleum Hydrocarbons from Water Samples**

This method was developed for the extraction of total petroleum hydrocarbons (or silica gel treated-hexane extractable material (SGT-HEM) as described in EPA method 1664) from water samples. The analytes are determined by gravimetric analysis.

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### **EXTRACTION PROCEDURE**

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**ISOLUTE® SPE Column:** TPH 1 g/6 mL Part # 752-0100-C

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**Pre-treatment:** To the water sample (1 L) add methanol (10 mL) and adjust to pH 2 with 6 M hydrochloric acid.

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**Solvation:** Condition the column with methanol (10 mL) at a flow rate of 5 mL/min.

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**Equilibration:** Rinse the column with deionized water adjusted to pH 2 with HCl (10 mL) at a flow rate of 5 mL/min.

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**Sample application:** Apply the sample to the column at a flow rate of up to 30 mL/min. Rinse the sample bottle well with acetone (10 mL) ensuring that the sides come into good contact with the acetone, and dilute the rinse solvent with 40 mL of deionized water. Mix thoroughly, and adjust to pH 2 before loading onto the SPE column. Repeat this step until the rinse solvent is clear.

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**Interference elution:** Dry the column for 30 minutes on a VacMaster (full vacuum) or with gas (nitrogen or carbon dioxide) at 4 L/min. When dry, the column will no longer feel cool to the touch.

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**Analyte elution:** Elute the TPH fraction with hexane (2 x 4 mL, with a soak step between each aliquot) at 5 mL/min into a tared vial. Remove the hexane under a gentle stream of nitrogen. The evaporation time may be reduced by placing the vial in a heating block at 35 C. Once the hexane is removed, weigh the collection vial to the nearest 0.1 mg.

Repeat the evaporation for 1 minute and re-weigh.

Repeat this step until the weight lost is <1 mg.

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**Structure** Various non-polar hydrocarbons.



**Structural considerations**

This method is suitable for the non-polar petroleum hydrocarbons (SGT-HEM)

**Matrix considerations**

The matrix is polar, and the analytes are extracted by a non-polar extraction mechanism.

**Analytical method**

Gravimetric analysis using an analytical balance having precision to 0.1 mg.

**Reagents**

**General comments**

1. This method and column can be used for 'Oil and Grease' type determinations, where the concentration of polar components is very low. In this situation, the hexane elution step should be followed by an elution (into a second tared vial) using 2 x 4 mL THF/hexane (1:1, v/v).

Combination of these two fractions will give a determination of Total Oil and Grease, as described in EPA method 1664.

However, for higher concentrations of polar components, we would recommend reference to application note IST1005 (Extraction of Oil and Grease from water samples) which utilizes a column specifically optimized for EPA method 1664 type extractions.

2. Due to the nature of the analytes, the bottle washing steps after sample loading are very important, as analytes do stick to the walls of the sample bottle. For this reason, sample splitting is not recommended for TPH or Oil and Grease samples.

3. Previous # IST3002

ISOLUTE column part numbers represent the product configuration of choice for use with a vacuum sample processing station. For 96-well and alternative column configurations compatible with any SPE automation system, please contact Biotage.

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