

# Automated Solid-Phase Extraction (SPE) of Total Petroleum Hydrocarbons

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## Introduction

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.

## Extraction Procedure

**\*ISOLUTE® SPE Column:** TPH 1 g / 6 mL

**Pre-treatment:** Add 10 mL of methanol to 1 L of sample. Acidify the sample with 6 M HCl to a pH ~2.

**Solvation:** Program the Thermo Scientific™ Dionex™ AutoTrace™ Solid Phase Extraction instrument to WASH SYRINGE with 5 mL of methanol, followed by a CONDITION COLUMN with 10 mL of methanol.

**Equilibration:** Program the Dionex AutoTrace instrument to WASH SYRINGE with 10 mL of distilled deionised water pH~2, followed by a CONDITION COLUMN with 10 mL of distilled deionised water pH~2.

**Sample application:** Program the Dionex AutoTrace instrument to LOAD 1100 mL SAMPLE. Following the sample load step, include a PAUSE AND ALERT step.

**Interference elution:** During pause step, remove sample line from sample bottle. Manually rinse sample bottle with 10 mL of acetone, swirl well to cover sides of bottle. Dilute acetone in bottle with 40 mL of distilled deionised water, pH~2. Return sample line to bottle, and press CONT. Program the Dionex AutoTrace to LOAD 60 mL SAMPLE. Following this step, include a PAUSE AND ALERT step. Repeat this step if necessary until the rinse solvent appears clear. Press CONT. Dry cartridge assembly for 30 min.

**Analyte elution:** Rinse the Dionex AutoTrace syringe with 5 mL of hexane. ELUTE TO SOAK with 4 mL of hexane. Follow this step with a TIMED PAUSE for 2 min. ELUTE TO COLLECT with 4 mL of hexane into tared collection tubes. Concentrate solvent to near dryness in a gentle stream of nitrogen. Collection tubes may be placed in a heating block held at 35 °C to expediate evaporation. Once solvent is almost gone, weigh tubes in one minute intervals until weight loss is less than 1 mg. Total petroleum hydrocarbons is the weight of residue.

## Setup parameters on the Dionex AutoTrace should be as follows:

### Flow Rates

Cond Flow: 40.0 mL/min  
Cond Air Push: 40.0 mL/min  
Load Flow: 30.0 mL/min  
Rinse Air Push: 40.0 mL/min  
Rinse Flow: 40.0 mL/min  
Elute Air Push: 40.0 mL/min  
Elute Flow: 20.0 mL/min

### SPE Parameters

Push Delay: 5 sec  
Air Factor: 1.0  
Autowash Vol.: 1.00 mL

\* ISOLUTE column part numbers represent the product configuration of choice for use with the Dionex AutoTrace instrument.

## The AutoTrace Instrument Procedure Should Be Written as Follows

Step	Method
1	Process 6 Samples using the following method steps:
2	Wash syringe with 5 mL of CH <sub>3</sub> OH.
3	Condition column with 10 mL of CH <sub>3</sub> OH.
4	Wash syringe with 10 mL of water pH=2.
5	Condition column with 10 mL of water to aqueous waste.
6	Load 1100.0 mL of sample on column.
7	Pause and Alert operator, resume when CONTINUE is pressed.
8	Load 60 mL of sample onto column.
9	Pause and Alert operator, resume when CONTINUE is pressed.
10	Dry column with gas for 30 min.
11	Wash syringe with 5 mL of hexane.
12	Soak and collect 4 mL fraction using hexane.
13	Pause for 2 min.
14	Collect 3 mL fraction into sample tube using hexane.
15	Clean each sample path with 10 mL into solvent waste.
16	Clean each sample path with 50 mL into aqueous waste.
17	Wash syringe with 10 mL of water.
18	End

### Structural 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 retention mechanism.

### Analytical Method

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

### General Comments:

- This method describes an automated procedure for the determination of silica gel treated-hexane extractable material (SGT-HEM) from an aqueous sample. This column can also be used for total oil and grease type extractions (EPA 1664 HEM) if the concentration of polar components is low. In this situation, the hexane elution step should be followed by an elution (into a second tared vial) using 2 × 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, refer to application note IST1018 (TPH/Total Oil and Grease determination on the AutoTrace instrument) which uses a column specifically optimized for EPA 1664 extractions on the Dionex AutoTrace instrument.
- 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.

### Conclusion

The Dionex AutoTrace instrument is specifically designed for extraction of large volume water samples. The extraction of silica gel treated hexane extractable material (SGT-HEM) is efficient using ISOLUTE SPE column TPH. The positive flow feature of the Dionex AutoTrace instrument ensures that each extraction is uniform and optimizes EPA Method 1664.

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