SPE Application Note for
Total Petroleum Hydrocarbons using the AutoTrace

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 Part # 752-0100-C

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

Solvation: Program the AutoTrace to WASH SYRINGE with 5 mL of methanol, followed by a CONDITION COLUMN with 10 mL of methanol.

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

Sample application: Program AutoTrace 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 deionized water, pH~2. Return sample line to bottle, and press CONT on AutoTrace. Program 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 on AutoTrace. Dry column assembly for 30 min.

Analyte elution: Rinse 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 AutoTrace should be as follows:

Cond Flow:	40.0 mL / min	Push Delay	5 sec
Load Flow:	30.0 mL / min	Air Factor	1.0
Rinse Flow	40.0 mL / min	Autowash Vol	1.00 mL
Elute Flow	20.0 mL / min		
Cond Air Push	40.0 mL / min		

Rinse Air Push 40.0 mL / min
Elute Air Push 40.0 mL / min

The AutoTrace procedure should be written as follows:

Step 1: Process 6 samples using the following procedure:
Step 2: Wash syringe with 5 mL of methanol
Step 3: Condition column with 10 mL of methanol in SOLVENT WASTE
Step 4: Wash syringe with 10 mL of water pH=2
Step 5: Condition column with 10 mL of water to AQUEOUS WASTE
Step 6: Load 1100 mL of sample on column
Step 7: Pause and Alert operator, resume when CONTinue is pressed
Step 8: Load 60 mL of sample onto column
Step 9: Pause and Alert operator, resume when CONTinue is pressed
Step 10: Dry column with gas for 30 minutes
Step 11: Wash syringe with 5 mL of hexane
Step 12: Soak and Collect 4 mL fraction using hexane
Step 13: Pause for 2 minutes
Step 14: Collect 3 mL fraction into sample tube using hexane
Step 15: Clean each sample path with 10 mL into SOLVENT WASTE
Step 16: Clean each sample path with 50 mL into AQUEOUS WASTE
Step 17: Wash syringe with 10 mL of water
Step 18: END

NOTE: Place sample lines into methanol for step 15 and reagent water for step 16 to flush out lines.

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 retention mechanism.
Analytical method	Gravimetric analysis using an analytical balance having precision to 0.1 mg.
Reagents	

General comments

1. 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 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 AGNT1018 (TPH/Total Oil and Grease determination on the AutoTrace) which utilises a column specifically optimised for EPA 1664 extractions on the AutoTrace system.

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.

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 consult your local distributor.

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