Extraction of Oil & Grease from Water Samples Using ISOLUTE® O&G Solid Phase Extraction Columns

This method is applicable to the determination of Oil and Grease as defined by EPA Method 1664 as Hexane Extractable Material (HEM, also referred to as Total Oil and Grease) as well as Silica Gel Treated-Hexane Extractable Material (SGT-HEM, also referred to as Total Petroleum Hydrocarbons or TPH).

Oil and Grease samples contain a range of polar and non-polar compounds. Using ISOLUTE® O&G SPE columns, both polar and non-polar compounds are extracted from water samples.

The compounds can be fractionated into the non-polar total petroleum hydrocarbon (TPH or SGT-HEM) fraction and remaining polar fraction using a dual elution solvent strategy.

If fractionation is not required, a single elution, eluting total Oil & Grease (or HEM) is used.

O&G or TPH levels are determined gravimetrically after gentle evaporation of the elution solvent.

ISOLUTE® O&G solid phase extraction columns are available in two sizes, and contain an integral depth filter which prevents blocking by highly particulate laden or contaminated waste-water samples.

Extraction Procedure

Format

ISOLUTE® O & G ISOLUTE® O&G 1 g/6 mL Column (Depth Filter) (Part Number 753-0100-CD or

ISOLUTE® O & G ISOLUTE® O&G 3 g/70 mL Column (Depth Filter) Part Number 753-0300-FD

Acidify the sample to pH of 1.9 to 2.1 with 6 M HCl to neutralize fatty acids. Add 10 mL of methanol to 1 L of sample. Chill to 4 °C for samples with high concentrations of fatty acids.

Solvation

Rinse the extraction column with 10 mL of methanol at 10 mL/min.

Equilibration

Rinse the extraction column with 10 mL of reagent water, acidified to pH between 1.9 and 2.1 with 6 M HCl, at 10 mL/min.

Sample Application

The sample may be loaded at rates not exceeding 10 mL/min. After the method has been optimized, increased loading rates should be tested. Loading rates between 50 and 100 mL/min are realistic.

Interference Elution

Rinse the sample bottle with 20 mL of reagent water acidified to pH ~2 with 6 M HCl. If high concentrations of fatty acids are present, repeat this rinse step. Load onto the extraction column. Add 10 mL acetone to the sample bottle. Shake well, making sure that the sides come into good contact with the acetone. **Dilute the acetone with 40 mL of reagent water acidified to pH ~2 with 6 M HCl.** Swirl. Load onto the extraction column. Repeat this acetone rinse until the bottle washings appear clear. Dry the extraction column for approximately 30 minutes on a vacuum manifold or with gas (nitrogen or carbon dioxide at 4 L/min). When dry, the column will no longer feel cool to the touch.

Analyte Elution

1. For determination of the non-polar fraction (TPH, or SGT-HEM), elute with 2 volumes of 4 mL hexane into a tared collection tube. Include a 2 minute soak step with each elution volume. Follow the instructions for evaporation in step 3.
2. Elute the polar fraction into a separate tared collection tube using 2 volumes of 4 mL hexane/THF (1:1, v/v). Include a 2 minute soak step with each elution volume. If separate determination of polar and non-polar fractions is not required, skip step 1.
3. Concentrate both fractions to near dryness under a gentle stream of nitrogen at a maximum temperature of 35 °C. When the solvent is almost gone, weigh the tubes at 1 minute intervals until weight loss is less than 1 mg. Total Oil and Grease is the combined weight of residue from both elutions (or the THF/hexane elutions alone if step 1 is skipped).

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Ordering Information

Part Number	Description	Quantity
753-0100-CD	ISOLUTE® O&G 1 g/6 mL Column (Depth Filter)	30
753-0300-FD	ISOLUTE® O&G 3 g/70 mL Column (Depth Filter)	16

General Comments

1. Due to the heterogeneous nature of oil and grease samples, the choice of column size used should be determined by sample type. The 6 mL configuration (p/n 753-0100-CD) should be used for samples containing SGT-HEM concentration ‹ 50 ppm, and HEM concentration ‹ 50 ppm. For higher concentrations, the ISOLUTE® O&G column is available in a higher capacity, wider column format (p/n 753-0300-FD). This format facilitates the rapid extraction of samples containing higher concentrations of Oil & Grease, or high levels of particulate material. Samples can be processed without chilling using this format. Alternatively, the sample volume may be reduced.
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 Oil and Grease samples.

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