SPE Application Note for TPH/Total Oil and Grease Determination

Application Note

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).

Structure:	Various (polar and non-polar compounds)
Structural considerations:	Both polar and non-polar analytes are extracted by this method. These compounds can be fractionated by using non-polar and mixed solvents consecutively. If fractionation is not required, only a single elution is performed.
Matrix considerations:	These compounds are being extracted from an aqueous matrix.
Analytical Method:	Gravimetric determination

Extraction Procedure

ISOLUTE® SPE Column:	ISOLUTE O & G (Part Number 753-0100-CD or 753-0300-FD).
Pre-treatment:	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 with 10 mL of methanol at 10 mL/minute.
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/minute. 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 box 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. Collection tubes may be placed on a heating block (35 °C) to expediate evaporation. When the solvent is almost gone, weigh the tubes at 1 minute intervals until weight loss is less than 1mg. Total Oil and Grease is the combined weight of residue from both elutions (or the THF/hexane elutions alone if step 1 is skipped).

Biotage

General comments

1. The Oil and Grease column is also available in a 70 mL configuration, with or without depth filter, for the rapid extraction of samples containing higher concentrations of oil and grease, or high levels of particulate material. Samples can be processed without chilling using this configuration. Contact Biotage for details.

2. Due to the heterogenous nature of oil and grease samples, the choice of column used should be determined by sample type. The 6 mL configuration should be used for samples containing SGTH EM< 50 ppm, and HEM < 50 ppm. For higher concentrations, consider the 70 mL configuration. Alternatively, the sample volume may be reduced.

3. 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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