Determination of Mineral Oil in Water According to ISO 9733-2:2000 Using ISOLUTE® Na_2SO_4/FL Solid Phase Extraction Columns

Introduction

This application note describes the extraction and clean-up of mineral oil (petroleum oil) from water samples according to ISO 9377-2:2000. This method involves a liquid-liquid extraction step followed by clean up using an SPE column containing layers of sodium sulfate and florisil, to remove halogenated and other polar interferences from the extract.

Sample Preparation Procedure

Format

ISOLUTE® Na_2SO_4/FL 4 g/6 mL, Part Number 976-0400-C

Pre-Treatment

1. Cool sample (250–1000 mL) to ~ 10 °C (if required, to prevent volatilization).
2. Acidify to pH 2 with HCl. This step may be performed in the field at collection.
3. Add magnesium sulfate to the sample (80 g/900 mL, as required (see Additional Information section).
4. Add hexane (50 mL), close bottle, and shake or stir vigorously for 30 mins. Further treatment such as centrifugation may be required if an emulsion is formed.
5. Remove hexane layer and transfer to a pre-solvated SPE column for clean-up.

Column Solvation

Rinse column with hexane (5 mL)

Sample Application

Apply the hexane extract to the column, and allow to flow under gravity. Low vacuum or pressure may be used (optimize flow rate as required).

Analyte Elution

Elute hydrocarbons with hexane (2 x 10 mL), under gravity or low vacuum/pressure (optimise flow rate as required).

Post Extraction

1. Concentrate the extract using a rotary evaporator with controlled vacuum to a volume of approximately 6 mL.
2. Concentrate further to just below 1 mL using a gentle flow of nitrogen.
3. Make up to exactly 1 mL with hexane, or calculate the exact volume of the concentrated extract by weighing.
4. Transfer an aliquot of the final extract to a septum vial for GC analysis. If a high HC oil index is expected or if a large volume injection system is used, concentration of the extract to 1 mL is not required, but a known volume should be used.

Chemicals and Reagents

- Hexane
- Magnesium Sulfate

Additional Information

1. Hexane is suggested as extraction solvent, however other non-branched alkanes may be suitable.
2. Samples may need to be preserved in the field by adjustment with mineral acid (e.g. HCl) to pH 2. If high levels of humic acids are present however, acidification should be avoided to prevent precipitation of the humic substances.
3. Addition of magnesium sulfate is only required if the samples are known to emulsify.
4. For full details of the method, see ISO 9377-2:2000 Determination of hydrocarbon oil index. Part 2: Method using solvent extraction and gas chromatography.

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Ordering Information

Part Number	Description	Quantity
976-0400-C	ISOLUTE® Na_2SO_4/FL 4 g/6 mL	30

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