

EPA 1664B Oil and Grease by Automated Solid Phase Extraction (SPE)



Introduction

EPA 1664B refers to the US EPA Solid Phase Extraction (SPE) protocol for n-hexane extractable materials (HEM) - usually oil and grease - from water samples. The method calls for the passage of a 1 Liter aqueous sample across an SPE cartridge, then eluting the cartridge with n-hexane.

The traditional alternative to SPE, LLE (Liquid-Liquid) Extraction, is both time consuming and labor-intensive, and large volumes of solvent. In this automated SPE method, samples are loaded across C₁₈ cartridges and then eluted with less than one quarter of the solvent required for eluting the same sample by LLE.

The following application covers the extraction of aqueous samples using the FMS, Inc Turbo Trace ABN SPE system to extract aqueous samples for EPA 1664B.

Instrumentation

- FMS, Inc. TurboTrace ABN® SPE system
- FMS, Inc. 250 mL concentrator tubes
- Mettler Toledo analytical balance

Consumables

- FMS Inc 2-gram C₁₈ Cartridges
- Fisher hexadecane and stearic acid
- Fisher 6 N Hydrochloric Acid
- Fisher Optima* n-Hexane
- Fisher Optima* Methanol
- Ultrapure DI water
- Fisher Oil & Grease aluminum weighing pans

Procedure

1 Liter aqueous samples acidified to pH <2 with HCl

Samples spiked with varying concentrations of oil and grease spiking solution.

SPE

1. Cartridges conditioned with 15 mL hexane
2. Cartridges conditioned with 15 mL MeOH
3. Cartridges conditioned with 15 mL H₂O
4. Samples passed across C₁₈ cartridges at 10-12 inch Hg vacuum
5. Cartridges dried with N₂ at 10 PSI
6. Sample bottles sprayed with 80 mL n-hexane
7. N-Hexane bottle spray loaded across cartridge and collected
8. Cartridges eluted with additional 10 mL n-hexane
9. Cartridges purged with N₂ eluting n-hexane directly to FMS collection tubes

Gravimetric analysis

1. Pre-weigh aluminum pan
2. Pour content of collection tube with hexane into weighing pan
3. Evaporate content (hexane) on heating-plate
4. Weigh aluminum pan again and subtract the pre-weight values.



Results

Table 1; Results of oil and grease LCS samples at five concentrations.

Sample Concentration	Amount Recovered (%)
2.5 mg/L	95%
2.5 mg/L	95%
5 mg/L	85%
5 mg/L	95%
10 mg/L	85%
10 mg/L	90%
20 mg/L	85%
20 mg/L	85%
40 mg/L	90%
40 mg/L	95%

Conclusions

Analysis of oil and grease yielded excellent, consistent recoveries with minimal deviations between replicates.

Extractions at 2.5, 5, 10, 20 and 40 mg/L all displayed excellent recoveries within 78-114% windows.

With a total of 90 mL n-hexane required for the elution and bottle rinse combined with a total extraction time of < 60 min from start to elution, the Turbo Trace ABN SPE proves to be a more efficient and economical solution for EPA1664B extractions than LLE.

Combined with gravimetric analysis, minimal sample manipulation is needed, eliminating recovery loss by human interaction.



FMS TurboTrace SPE system

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