

EPA 1664A; Oil and Grease by Solid Phase Extraction (SPE)



Introduction

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

The traditional alternative to SPE, LLE (Liquid-Liquid) Extraction, is both time consuming and labor-intensive plus it requires large volumes of solvent. In this automated SPE method, samples are rapidly pulled across a C₁₈ cartridge 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 system to extract aqueous samples for EPA 1664.

Instrumentation

- FMS, Inc. TurboTrace™ SPE system
- FMS, Inc. SuperVap™ 12 Concentrator
- FMS, Inc. 50 mL direct-to-vial concentrator tubes
- Mettler Toledo analytical balance

Consumables

- Fisher Pesticide Optima* n-Hexane
- Fisher Anhydrous Sodium Sulfate
- Fisher Optima* Methanol
- Fisher HPLC Grade Water
- FMS 2 gram C₁₈ Cartridges
- Restek Oil & Grease Mix (Cat#31954)
- Fisher Concentrated Sulfuric Acid

Procedure

Pre-weigh collection vials

1 liter aqueous samples acidified to PH <2 with H₂SO₄

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

SPE

1. Cartridges pre-wet with Hexane
2. Cartridges conditioned with MeOH
3. Cartridges conditioned with H₂O
4. Samples passed across C₁₈ cartridges at full vacuum
5. Cartridges dried with N₂ at 15 PSI
6. Sample bottles sprayed with n-Hexane
7. N-Hexane bottle spray loaded across cartridge and collected through in-line NaSO₄
8. Cartridges eluted with additional 10 mL n-Hexane
9. Cartridges purged with N₂ eluting n-Hexane directly to FMS SuperVap concentrator

SuperVap concentrator

1. Preheat temp: 20 minutes at 60 °C
2. Evap mode: 60 °C
3. Nitrogen Pressure: 10 PSI
4. Evaporate extracts to total dryness



Figure 1: TurboTrace SPE system with the SuperVap Concentrator.



Results

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

Sample Concentration	Amount Recovered	%	RPD
2 mg/L	2.1 mg/L	105%	4.9%
2 mg/L	2.0 mg/L	100%	
5 mg/L	5.0 mg/L	100%	0.0%
5 mg/L	5.0 mg/L	100%	
10 mg/L	9.99 mg/L	99.9%	1.0%
10 mg/L	9.89 mg/L	98.9%	

Conclusions

Analysis of oil and grease yielded excellent, consistent recoveries with minimal deviations between replicates. Extractions at 2 mg/L near the method MDL (1.4 mg/L), at the ML (5 mg/L) and at 10 mg/L all displayed excellent recoveries.

With a total of 40 mls n-Hexane required for the elution and bottle rinse combined with a total extraction time of <25 minutes from start to elution the Turbo Trace SPE proves to be a far more efficient and economical solution for EPA1664 extractions than LLE. Combined with SuperVap Concentrator, minimal sample manipulation is required eliminating possible recovery loss by human interaction.

For more information contact FMS:
 FMS Inc.
 580 Pleasant Street
 Watertown, MA 02472
 Phone: (617) 393-2396
 Fax: (617) 393-0194
 Email: onlineinfo@fms-inc.com
 Web site: www.fms-inc.com