Empore™ Oil and Grease SPE Disk for Water Samples by Using a Cleaner EPA Method 1664 with Less n-Hexane Usage

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Application Note
Environmental

Abstract
CDS Empore™ (formerly 3M™ Empore™) Oil and Grease (O&G) Solid Phase Extraction (SPE) disks facilitate reliable sample preparation and provide excellent analyte recovery. This application note demonstrates the performance of such disk under EPA Method 1664 while reducing the total volume of n-hexane by 40%.

Introduction
EPA Method 1664 was originally designed as a performance-based method for the recovery of hexadecane from water samples by liquid-liquid extraction. However, the original method permits the use of alternative methods, such as solid phase extraction (SPE), as long as all performance specifications are met. EPA Method 1664 was originally adapted for SPE by 3M™.[1] In this way, performance is measured through the combined recovery of both hexadecane and stearic acid.[2] Elution of analytes from SPE sorbents is effectively accomplished using n-hexane. n-Hexane, however, is difficult to dispose of through waste streams and are frequently found in high concentrations in landfills.[3,4] Therefore, it is desirable to minimize the volume needed for elution to reduce not only environmental impact but also experimental costs, provided that analyte recovery is not compromised.

In this application note, a one-liter water sample was passed through a 47mm Empore™ O&G disk and eluted with hexane under negative pressure. Then the extract was dried to determine the extracted mass of hexadecane and stearic acid. The effect of reduced eluant volumes was assessed. The validation data presented herein was determined on three replicate measurements of the same lot of Oil and Grease disks. MDLs were not determined as part of this validation.

Experiment Setup
Chemicals:
EPA Method 1664 analytes, stearic acid and hexadecane, were purchased from Chem Service (West Chester, PA) and Sigma Aldrich (St. Louis, MO) respectively. HPLC grade n-Hexane, Methanol and Acetone were all purchased from VWR International (Radnor, PA). Sulfuric acid was purchased from VWR International. Water was treated in house using a Milli-Q Water Treatment System.

Preparation of Standard:
To prepare the stock solution, 200 mg of both stearic acid and hexadecane were dissolved in 100 mL of acetone. 1 L of water sample was made by first adjusting the pH to at least pH 2 with concentrated sulfuric acid and followed by the addition of 10 mL of stock solution.

Methods:
1.Assemble an all glass filtration assembly using a 47 mm Empore™ O&G SPE disk. Use of a manifold for multiple extractions is acceptable.

2. Wash the extraction apparatus and disk by adding 20 mL of n-hexane to the reservoir. Pull a small amount through the disk with a vacuum; turn off the vacuum and allow the disk to soak for about three minutes. Pull the remaining solvent through the disk and allow the disk to dry for five minutes.

3. Condition the disk by adding approximately 30 mL of methanol to the reservoir, pulling a small amount through the disk then letting it soak for about one minute. Pull most of the remaining methanol through the disk, leaving 3 to 5mm of methanol on the surface of the disk.

4. Add 50 mL of reagent water to the disk and using the vacuum pull most through, again leaving 3 to 5 mm of water on the surface of the disk.

5. Add the water sample to the reservoir and, under vacuum, filter as quickly as the vacuum will allow. Drain as much water from sample bottle as possible. Dry for 5 minutes.

6. Remove filter assembly and insert suitable sample tube for eluate collection.

7. Add 10 mL of n-hexane to the sample bottle. Rinse bottle thoroughly and transfer solvent to the disk and filtration reservoir, rinsing all sides in the process.

8. Pull half of solvent through disk then release the vacuum. Allow the remaining solvent to soak the disk for three minutes, then draw remainder through under vacuum.

9. Using a disposable pipette, rinse down the sides of the filtration glassware with 10 mL of n-hexane.

10. Dry the combined eluant with 5-10 grams granular anhydrous sodium sulfate. Rinse the collection tube and sodium sulfate each with a 5 mL portion of n-hexane and place combined solvent into a concentrator tube.

11. Dry extract to 1 mL under gentle stream of nitrogen (may be warmed gently).

Analysis:
Evaporate the n-hexane from the collection vessel until a constant weight is reached. Weigh the collection vial, compare weight to the tared weight, and calculate the quantity of HEM (oil and grease residue) present in units of mg/L.

Results and Discussions

Table 1 shows the combined recovery data of stearic acid and hexadecane from EPA Method 1664 using both 80 and 50 mL of n-hexane during the extraction process. 80 mL of n-hexane is the volume used by 3M™ from the previous application report on Empore™ O&G disks.[1] The average recovery was 89% and 85% for 80mL and 50mL of n-hexane with relative standard deviations (RSD) of 0.4% and 8.0%, respectively. These results indicate that reducing the total volume of n-hexane during the extraction process produces similar recoveries. Recoveries greater than 80% are acceptable.

Table 1: Combined recovered mass (mg) of hexadecane and stearic acid in EPA 1664.

mL of n-Hexane	1	2	3	**Average (mg)**	**RSD**	**Recovery Rate**
80 mL	35.3	35.5	35.5	**35.4**	**0.4%**	**89%**
50 mL	36.2	34.9	31.0	**34.0**	**8.0%**	**85%**
50 mL Blank	0.6	0.6	0.4	**0.53**	**21.5%**	

Conclusions:

A simple modification made to EPA Method 1664 to reduce the total volume of n-hexane used during the wash and elution steps of extraction of hexadecane and stearic acid from Empore™ Oil and Grease disks. The total volume of n-hexane was reduced by 40% without having an appreciable effect on the recovery of stearic acid and hexadecane. The percent recovery with the reduced volume of n-hexane was 85% with 8.0% RSD. Meanwhile, the recovered background mass was consistently less than 2% of the extracted stearic acid and hexadecane mass. The results indicate that the Empore™ Oil and Grease Solid Phase Extraction disks are suitable for high recoveries while also reducing the total volume of n-hexane used during the extraction process.

References

(1) 3M. EPA Method 1664: N-Hexane Extractable Material Quantification; 2009.
(2) Environmental Protection Agency. Method 1664B: N-Hexane Extractable Material and Silica Gel Treated n-Hexane Extractable Material by Extraction and Gravimetry; 2010.
(3) Environmental Protection Agency. Air Emissions from Municipal Solid Waste Landfills - Background Information for Final Standards and Guidelines; 1991.
(4) Environmental Protection Agency. Characterization of Municipal Solid Waste by Weight; 1992.