



## Empore™ EZ-Trace SPE Workstation Produces a Clean and Efficient Extraction Under EPA Method 1664

### Application Note

Environmental

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### Abstract

The CDS Empore™ (formerly 3M™ Empore™) EZ-Trace SPE workstation is combined with Empore™ Oil and Grease (O&G) SPE disks to perform 4 extractions simultaneously under EPA Method 1664. O&G disks facilitate reliable sample preparation and provide excellent analyte recovery. This application uses a modified EPA 1664 method that reduces 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™.<sup>1</sup> In this way, performance is measured through the combined recovery of both hexadecane and stearic acid.<sup>2</sup> 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.<sup>3,4</sup> 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.

Performing a single extraction under EPA Method 1664 can often be time consuming, however. The Empore™ EZ-Trace addresses this challenge by allowing users to perform up to 4 extractions simultaneously. In combination with high throughput and precision control valves, the EZ-Trace is designed to increase sample processing efficiency. Other features such as the simple flow design and separation of organic and aqueous waste streams help to create a user friendly extraction experience. The EZ-Trace is convenient for this method and many other EPA methods as well.

In this application note, four, one-liter water samples containing hexadecane and stearic acid are passed through a 47mm Empore™ O&G disks on the Empore™ EZ-Trace and eluted with n-hexane under negative pressure. Then the extract was dried to determine the extracted mass of hexadecane and stearic acid. The validation data presented herein was determined on four replicate measurements of the same lot of O&G disks. MDLs were not determined as part of this validation.

### Experiment Setup

SPE Disk:

Empore™ Oil & Grease 47mm disks (catalog # 2270; Fisher Scientific PN 13110020; VWR PN 76333-102; MilliporeSigma PN 66887-U).

Extraction System:

Four extractions were performed simultaneously with the Empore™ EZ-Trace SPE vacuum system (catalog No. 8000; Fisher Scientific PN 01-189-101; VWR PN 76449-580).



#### 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. The Empore™ EZ-Trace was set up for extraction with four, 47 mm Empore™ O&G SPE disk.

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

3. Condition the disks by adding approximately 30 mL of methanol to each reservoir, pulling a small amount through the disk then soaking 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 each disk pulling most of the water through, again leaving 3 to 5 mm of water on the surface of the disk.

5. Add the water sample to the reservoir and filter as quickly as the vacuum will allow. Dry for 5 minutes.

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

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

8. Using a disposable pipette, rinse down the sides of the disk holders with 10 mL of n-hexane.

9. 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.

10. Dry extract under a gentle stream of nitrogen (may be warmed gently). Weigh the collection vial, compare weight to the initial empty vial weight to determine the quantity of HEM 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 for 4 water samples and is compared to the method blank. The average recovery was 99.6% with a 3% RSD. The average recovered background mass from the method blank was 0.15mg with an 86% RSD. This background mass is consistently <1% of the expected sample mass. The lower limit for acceptable recoveries under this method is 80%.

Table 1: Combined recovered mass (mg) of hexadecane and stearic acid in EPA Method 1664 compared to the method blank (n=4).

	Average (mg)	%RSD	Recovery Rate (%)
Sample	39.8	3.0	99.6
Blank	0.15	86.0	--

#### Conclusions:

The Empore™ EZ-Trace was used to increase the efficiency of extracting HEM from multiple water samples under the guidance of EPA Method 1664. Empore™ O&G disks were used for the extraction of HEM. The percent recovery obtained by using the EZ-Trace to assist with extraction was 99.6% with a 3% RSD (n=4). Additionally, the method blank is consistently <1% of the extracted sample mass. These results demonstrate that the EZ-Trace produces clean extracts and is suitable for high recoveries of HEM under EPA Method 1664. High recoveries and clean extracts are all achieved using a modified method using 40% less n-hexane for wash and elution steps than the original EPA method..

#### 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.