



# Empore™ Extraction Disks

## EPA Method 1664

### Summary

This method summarizes only the N-Hexane Extractable Material (HEM) quantification portion of Method 1664. A one liter water sample is passed through the Empore disk and eluted with n-hexane. The extract is dried with sodium sulfate, hexane is evaporated and the residue is weighed and reported in mg/L as HEM.

### Description

EPA Method 1664 Revision A (February 1999) is a performance-based method for N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT – HEM; Non-polar Material) by Extraction and Gravimetry. The method permits a laboratory to use alternative methods to liquid-liquid hexane extraction and concentration “provided that all performance specifications are met.” (page iv of EPA Method 1664 Revision A). There is a further note on page 14 of Revision A that states, “Solid-phase extraction (SPE) may be used at the discretion of the discharger/generator and its laboratory.” This method summary is a step-by-step guide for the use of Empore™ Oil and Grease Solid Phase Extraction Disks to replace the liquid-liquid hexane extraction technique.

### Initial Precision and Recovery

The table at the right lists the results of 3M’s Initial Precision and Recovery (IPR) study for the solid phase extraction modification of Method 1664 as established in section 9.1.2.1.

Method 1664, Empore™ Oil & Grease Disk (47mm)

Recovery	Standard Deviation	MDL	ML
98%	0.216	0.679	2.159

10 mg/L spikes (n=7). Initial Precision and Recovery tests were conducted in 3M laboratory. Results may vary based on individual analyst technique.

### Method

#### STEP 1

**Assemble Glassware:** Assemble the filtration apparatus with the oil and grease disk. Disk must be inserted with dimpled surface down. For samples containing high concentrations of suspended solids, 90 mm disks and systems are recommended. The vacuum system should be capable of drawing a minimum of 25 inches Hg (0.85 bar) for 90 mm disk systems. *Note: Disk must be used with dimpled surface down.*

Place the waste-receiving vial in manifold; wash the extraction apparatus and the disk with n-hexane. Rinse down the sides of the glassware with hexane. Use enough solvent to completely cover the disk (20 ml for

47 mm disk and 30 ml for 90 mm disk). Apply vacuum to draw the solvent through the disk.

Repeat (for a total of two hexane washes).

Allow the disk to dry under vacuum for one minute after the second wash.

Remove the waste receiving vial and dispose of the solvent according to local, state and/or federal regulations.

#### STEP 2

**Condition Disk:** Condition the disk by adding 10 mL methanol (47 mm disk) to the reservoir. Use approximately 30 mL methanol for a 90 mm disk. Use enough

solvent to completely cover the disk. Draw a small amount of methanol through the disk with the vacuum; vent the system and allow the disk to soak for 60 seconds. Draw most of the remaining solvent through the disk, leaving enough methanol to cover the surface of the disk. **Do not allow the disk to become dry. If the disk becomes dry at any point before sample extraction, repeat the conditioning step.**

Rinse the disk with deionized water. Use 30 mL water for a 47 mm disk and 100 mL water for a 90 mm disk. Draw most of the water through, leaving enough to cover the disk surface. Do not allow the disk to become dry.

## Method – continued

### STEP 3

**Extract Sample:** Add the sample (pH adjusted to 2) to the reservoir and apply full vacuum. If the sample is high in suspended solids, allow the sediment to settle and decant as much liquid as possible into the reservoir before adding the sediment. Do not let the disk go dry before adding the sediment-containing portion. By extracting a majority of the liquid before adding the sediment, potential plugging problems will be minimized. Filter as quickly as the vacuum will allow. Drain as much water from the sample container as possible.

### STEP 4

**Dry Disk** under vacuum for no more than 5 minutes. Excessive drying (>5 min.) can lead to lower recoveries of more volatile fractions.

### STEP 5

**Elute Disk:** Lift filtration assembly and insert suitable collection vial for eluate collection.

Add 10 mL hexane (30 mL for 90 mm disk) to the original sample container making sure to rinse down the sides. Replace the cover on the container and invert 2-3 times.

Transfer the hexane from inside the sample container to the disk using a disposable glass pipette. As the hexane is transferred to the disk, allow it to wash down the sides of the reservoir and then pass through the disk to ensure complete rinsing of all glassware.

Carefully apply vacuum to draw a few drops of hexane through the disk, and then stop the vacuum.

Allow the remaining hexane to soak into the disk for no more than 2 minutes. Then slowly draw the remaining solvent through the disk under vacuum to remove residual hexane and dry the disk.

Repeat steps above using a second aliquot of hexane.

Wash the sides of the glass reservoir using another aliquot of 10 mL hexane. Apply vacuum and draw the entire volume of hexane through the disk.

Allow the disk to dry for approximately 5 minutes and then turn off the vacuum. The collection vial now contains three combined aliquots of hexane.

### STEP 6

**Dry the Eluate:** The eluate is dried using anhydrous sodium sulfate.

- Place glass wool into the bottom of a small funnel and add 5 gm sodium sulfate.
- Obtain a clean collection vial or weighing pan and record its weight. **Note:** *Wear gloves when handling preweighed collection vessels as oils from the skin may be transferred to the vial and affect results.*
- Pour or pipette the eluate onto the sodium sulfate and collect into the preweighed collection vessel.

- Rinse the sides of the collection vial with 5 mL hexane and add to the sodium sulfate.

- Rinse the sodium sulfate with another aliquot of 5 mL hexane, allowing all the solvent to run through the sodium sulfate and into the collection vial.

### STEP 7

**Analyze:** Evaporate 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.

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