

# Empore™

## Solid Phase Extraction Disks

### EPA Method 1664 n-Hexane Extractable Material Quantification

#### 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™ Oil and Grease Solid Phase Extraction Disk and eluted with n-hexane. The extract is dried with sodium sulfate, the n-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 n-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 n-hexane extraction technique.

#### Initial Precision and Recovery

The table below lists the results of an internal 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	98%
Standard Deviation	0.216
MDL	0.679
ML	2.159

10 mg/L spikes (n=7). Initial Precision and Recovery tests were conducted in an internal 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.

When using solvents and other chemicals, be sure to read and follow the supplier’s precautions and directions for use before using. Place the waste-receiving vial in the manifold; wash the

extraction apparatus and the disk with n-hexane. Rinse down the sides of the glassware with n-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 n-hexane washes).

Allow the disk to dry under vacuum for five minutes 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:** Replace the waste receiving vial and condition the disk by adding 20 mL methanol (47 mm disk) to the reservoir. Use approximately 40 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 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.**

Remove the waste receiving vial and dispose of the solvent according to local, state and/or federal regulations. Rinse the disk with deionized water. Use 50 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.

**STEP 3 Extract Sample:** Add the sample (pH adjusted to less than 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. Rinse the sample container with 10 mL of reagent water and add to the disk. Do not let the disk dry before adding the rinse solution.

## STEP 4 Dry Disk

under vacuum for at least 5 minutes. Best performance will be achieved when the disk is completely dried.

## STEP 5 Elute Disk:

Lift filtration assembly and insert suitable collection vial for eluate collection. Add 10 mL n-hexane (30 mL for 90 mm disk) to the original sample container, replace the cover on the container and invert 2-3 times to rinse the sides of the container.

Transfer the n-hexane from inside the sample container to the disk using a disposable glass pipette. As the n-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 n-hexane through the disk, and then stop the vacuum.

Allow the remaining n-hexane to soak into the disk for at least 2 minutes. Then slowly draw the remaining solvent through the disk under vacuum to remove residual n-hexane and dry the disk.

Repeat steps above using a second aliquot of n-hexane.

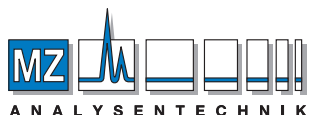
Wash the sides of the glass reservoir using another aliquot of 10 mL n-hexane. Apply vacuum and draw the entire volume of hexane through the disk.

Allow the disk to dry for at least 5 minutes and then turn off the vacuum. The collection vial now contains three combined aliquots of n-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 n-hexane and add to the sodium sulfate.
- Rinse the sodium sulfate with another aliquot of 5 mL n-hexane, allowing all the solvent to run through the sodium sulfate and into the collection vial.



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**Note:** Empore Solid Phase Extraction Products are intended for solid phase extraction during scientific research only. These products are not intended for use in medical devices or in assessment and treatment of clinical patients.

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