# **EPA Method 1664 Hexane Extractable Material (HEM)**



## **UCT Part Numbers**

#### **ECUNIOGXF**

ENVIRO-CLEAN® - 2000 mg endcapped C18, 83 mL cartridge

# **Procedure:**

#### 1. Assemble

- a) Assemble cartridge adapters on the vacuum manifold.
- b) Place **ECUNIOGXF** cartridge(s) on each vacuum station needed.
- c) Connect the manifold to a suitable trap and attach the trap to a vacuum system capable of attaining a minimum of 25" Hg (635 mm) of vacuum.
- d) Insert waste collection vial in manifold (optional).

## 2. Prepare Water Sample

- a) Adjust the pH of the sample to < 2 by adding 5 mL of 6N HCl or 2.5 mL of concentrated H₂SO₄. The pH of deionized water cannot be accurately determined using pH paper.
- b) If acid was added to the sample in the field, do not add more unless the pH > than 2.

**Note:** Gloves are recommended as skin oils may affect final sample weight.

# 3. Condition the Cartridge

- a) Rinse the sides of the cartridge and bottle holder with 10 mL of hexane.
- b) Allow cartridge to soak for 1 minute.
- c) Draw the hexane through the cartridge using vacuum.
- d) Discard the hexane from the waste vial.
- e) Draw full vacuum through the cartridge for 2 minutes to dry.
- f) Add 10 mL of methanol to the cartridge.
- g) Slowly draw the methanol through leaving a layer on the cartridge frit.
- h) Soak for one minute.
- i) Draw methanol through the cartridge.
- j) Remove waste vial and discard methanol.
- k) Immediately, add 80 mL (fill cartridge) of DI water to the cartridge.
- I) Soak for 1 minute.
- m) Draw all of the water through the cartridge to waste.

#### 4. Sample Addition

- a) Add the 1 liter water sample directly to the cartridge.
  b) Draw the sample through the cartridge under low
- b) Draw the sample through the cartridge under low vacuum. This may take several minutes depending on the solids in the sample. (Note 1) Increase vacuum pressure if necessary. Do not exceed 50 mL/minute for optimum recoveries. This is a fast drip, but not a stream.
- c) Remove the cartridge and tap any excess water from the bottom of the cartridge.
- Replace cartridge and allow to dry under full vacuum for 10 minutes.
- e) Remove any water remaining in the bottom support of the cartridge with a paper towel if necessary.

#### 5. Elution

- a) Prepare an extract collection vial containing about 8 mm (0.3 inch) of anhydrous sodium sulfate.
- b) Place the vial in the manifold station under the cartridge.
- Add a thin layer of anhydrous sodium sulfate to the top of the cartridge.
- d) Rinse the water sample bottle with 10 mL of hexane.
- e) Add the hexane to the cartridge.
- f) Soak cartridge for 3 minutes. A slow drip of hexane is permissible.
- g) Turn on vacuum and slowly draw the hexane through the cartridge and into the collection vial.
- h) Turn off vacuum then repeat steps 5 d) g) 2 additional times with 10 mL of hexane.
- i) Do not allow the solvent to splash into the collection vial.
- j) Add another 10 mL of hexane to the cartridge, rinsing the bottle holder.
- k) Soak cartridge for 3 minutes.
- l) Draw the hexane through the cartridge and collect.

### 6. Dry the Extract

- a) Remove the collection vial from the manifold and cover with a screw cap.
- b) If water is still present in the extract (no free moving sodium sulfate is visible) shake the extract to form a water/hexane emulsion and immediately pour the extract through a sodium sulfate funnel or column containing approximately 30 g of anhydrous sodium sulfate held in place with a glass wool plug or frit. Do not use filter paper.
- c) Collect the extract in a clean, pre-weighed vessel.
- d) Rinse the collection vial with hexane and add it to the sodium sulfate and collect. This will rinse the vial and the sodium sulfate. Poor rinsing of the sodium sulfate will result in low recoveries.

# 7. Gravimetric Analysis

a) Carefully evaporate the hexane using a nitrogen evaporator at 40° C until the extract just reaches dryness.

Note: Do not over dry or low recoveries will result

- b) Allow to cool to room temperature in a desiccator before weighing.
- Record this weight as the mass per unit volume of HEM and report as mg/L.





## **Notes:**

- 1) If very high solids are present, add a small plug of glass wool to the cartridge prior to extraction to prevent clogging and improve flow. The glass wool must be thoroughly rinsed with hexane as part of the cartridge during the elution step.
- 2) Stearic acid must be in solution in the spiking solution or low recoveries will result. If small crystals are present in the spiking solution, sonicate or shake until dissolved.
- 3) If white crystals are present in the sample bottle after elution, the sample pH was not low enough prior to extraction. Repeat with lower pH.
- 4) HCl will lose strength over time. Sulfuric acid is a good substitute.
- 5) Any residue that does not rinse from the bottle or elute from the cartridge is not HEM.

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