



EPA Method 1664B: n-Hexane Extractable Material (HEM; Oil and Grease)

UCT Part Numbers:

ECUCTVAC6 - Enviro-Clean 6-Station Vacuum Manifold

ECROCKER400 - Rocker 400 Vacuum Pump 110 volt

ECUCTTRAP20 - 20L Manifold Trap

ECUCTADP - Enviro-Clean Glass Cartridge Adaptor

ECUNIBHD - Enviro-Clean Universal White Bottle Holder

ECUNIOGXF - Enviro-Clean Universal Oil & Grease XF 2000mg/83mL

ECSS25K - Enviro-Clean Bulk Anhydrous Sodium Sulfate 25kg

ECUNIMSS - Enviro Clean Universal Cartridge 20g Muffled Sodium Sulfate

Procedure

1. Assemble
 - a) Connect a 6-station vacuum manifold (ECUCTVAC6) to a 20-L manifold trap (ECUCTTRAP20), and attach the trap to a vacuum pump (ECROCKER400)
 - b) Attach the glass adapters (ECUCTADP) onto the manifold
 - c) Connect the bottle holders (ECUNIBHD) to the top of the SPE cartridges (ECUNIOGXF), and attach the cartridges to the glass adaptors on the manifold
2. Prepare Water Sample
 - a) Adjust the pH of the sample to < 2 by adding 6N HCl or H₂SO₄.
 - 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 n-hexane
 - b) Allow cartridge to soak for 1 min
 - c) Draw the hexane through the cartridge using vacuum, leave full vacuum on for 1 min, then release the vacuum
 - d) Add 10 mL of methanol to the cartridge, soak for 1 min
 - e) Slowly draw the methanol through leaving a thin layer on the cartridge frit
 - f) Add about 80 mL of DI water to the cartridge
 - g) Draw all of the water through the cartridge to waste, do not let sorbent go dry

4. Sample Addition

- a) Load sample bottle onto the bottle holder
- b) Draw the sample through the cartridge under low vacuum. This may take 20 or more minutes depending on the solids in the sample (Note 1). Increase vacuum pressure if necessary. Do not exceed 50 mL/min for optimum recoveries. This is a fast drip, but not a stream
- c) After all samples are passed through, turn full vacuum on. Remove the cartridges and tap/swing to remove any excess water from the bottom of the cartridges
- d) Replace the cartridges to the manifold, and dry under full vacuum for 10 min

5. Elution

- a) Insert extract collection vials with a thin layer (about 0.5-0.8 cm) of anhydrous sodium sulfate (ECSS25K) in the manifold
- b) Add a thin layer (about 0.5-0.8 cm) of anhydrous sodium sulfate into the SPE cartridge
- c) Rinse the sample bottle with 10 mL of n-hexane
- d) Add the hexane to the cartridge
- e) Soak cartridges for 2 min. A slow drip of hexane is permissible
- f) Turn on vacuum and draw the hexane through the cartridges and into the collection vials, leave vacuum on for 0.5 min
- g) Turn off vacuum then repeat steps 5 c) – f) 2 additional times with 10 mL of n-hexane
- h) Add 10 mL of hexane to the cartridges, rinsing the bottle holders
- i) Soak cartridges for 2 min
- j) Draw the hexane through the cartridges and collect, do not allow the solvent to splash into the collection vial

6. Dry the Extract

- a) Remove the collection vials from the manifold
- b) Dry the extracts by pouring them through the drying cartridges (ECUNIMSS) pre-rinsed with 10 mL n-hexane into the pre-weighed vials. Alternatively, use

glass wool stopped glass funnel containing approximately 10-20 g anhydrous sodium sulfate (pre-rinsed with n-hexane). Do not use filter paper

- c) Rinse the extract collection vials 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 samples using a nitrogen evaporator at 40° C until just reaches dryness

Note: Do not over dry otherwise low recoveries will result

- b) Allow the samples cool to room temperature in a desiccator before weighing
- c) Record the weight difference, and report as mg/L of HEM

Notes:

- 1) If 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 condition and elution steps.
- 2) Stearic acid must be in solution in the spiking solution otherwise 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.