EPA Method 1664, Revision A
N-Hexane Extractable Material (HEM) (Oil & Grease) by
Solid-Phase Extraction & Gravimetry

ENVIRO-CLEAN® Universal Oil and Grease Cartridges
Part Number ECUNIOGXF
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Method Summary

A sample of water, pH adjusted to <2, is extracted using a UCT Universal Oil and Grease Cartridge containing the solid-phase sorbent C18.

1) **Sample Collection**
   a) All samples must be acidified prior to analysis
   b) Adjust the pH of a 1-liter sample to 2 or lower by adding 5 mL of 6N HCl or 2.5 mL of concentrated H_2SO_4. A smaller volume of sample may be used provided all quality control requirements are met
   c) If the acid was added to the sample in the field, it is not necessary to repeat this step unless the pH has increased during storage
   d) Refrigerate sample to 0-6ºC if analysis is delayed more than 4 hours from collection

2) **Assemble**
   a) Assemble the UCT cartridge adapter(s) on a vacuum manifold
   b) Add the **ENVIRO-CLEAN® Universal Oil and Grease Cartridge** (Note 1)
   c) Place a cartridge in an automated extraction system if used in instead of a vacuum manifold
   d) Connect the vacuum manifold to a suitable trap and attach to a vacuum system capable of attaining a minimum of 25” Hg (635 mm) of vacuum

3) **Sample Spikes**
   a) Prepare a matrix spike by adding 40 mg/L of a PAR (precision and recovery) standard (Note 2)
   b) A concentration of 20 mg/L may also be used as long as the spike concentration is higher than the background concentration

4) **Condition the Cartridge**
   a) Wash the cartridge, including the sides, with 10 mL of hexane
   b) Allow to soak for 1 minute
   c) Draw the hexane through the cartridge to waste using vacuum. Discard the hexane
   d) Draw full vacuum through the cartridge for 2 minutes to remove the hexane
   e) Add 10 mL of methanol to the cartridge and slowly draw the methanol through the cartridge leaving a layer on the surface of the cartridge. **Do not let the sorbent dry out.**
   f) Soak for one minute then add 30 mL of DI water to the cartridge
   g) Draw the water through the cartridge to waste
   h) Do not allow the sorbent to completely dry to a powder before adding the sample, otherwise repeat this step starting with the addition of methanol

5) **Sample Addition**
   a) Add the water sample directly to the cartridge and draw through under vacuum. This may take several minutes for complete flow through the cartridge depending upon the level of solids in the sample. Do not allow the flow rate to exceed 500 mL per minute. Note 3
   b) Remove the cartridge before drying and tap the excess water from the bottom of the cartridge
   c) Allow the cartridge to dry under full vacuum for 10 minutes to remove any residual water

6) Elution
   a) Remove any water remaining in the bottom support of the cartridge with a paper towel
   b) Place an extract collection vial in the manifold. Note 4
   c) Rinse the sample bottle with 10 mL of hexane. Add the hexane rinse to the cartridge, washing the sides of the cartridge and the bottle holder if used. Soak for 1 minute. **Do not use any other solvents except hexane.**
   d) Slowly draw the hexane through the cartridge and into the collection vial.
   e) Do not allow the vacuum to blow air over the extract
   f) Immediately, repeat this procedure 2 additional times using 10 mL of hexane for each rinse
   g) While collecting the hexane do not allow it to splash out of the collection vial
   h) Add another 10 mL of hexane to the cartridge, rinsing the bottle holder. Soak for 2 minutes. Draw the hexane through the cartridge and collect

7) **Dry the Extract**
   a) Remove the collection vial from the manifold and cover with a screw cap
   b) Shake the extract to form a water/hexane emulsion and immediately pour the extract through a sodium sulfate funnel or column containing approximately 40 g of anhydrous sodium sulfate. Do not use filter paper to hold the sodium sulfate. Glass wool is acceptable.
   c) Collect the extract in a clean, tared vessel
   d) Rinse the collection vial with 5 mL of hexane and add it to the sodium sulfate. Note 5

8) **Gravimetric Analysis**
   a) Carefully evaporate the hexane using an analytical evaporator or similar device at 40^{O}C until a constant weight is obtained
   b) Alternate concentration techniques and containers such as glass beakers or aluminum pans may be used
   c) Do not dry on a hot plate or in an oven
   d) Allow to cool in a desiccator before weighing
   e) Record this weight as the mass per unit volume of oil and grease and report HEM as mg/L. Note 6

Notes

1) This cartridge is designed to fit the Horizon SPE-DEX® 4790 automated extraction system. The cartridge will also fit a standard 3 (# ECUCTVAC3) or 6 station (#ECUCTVAC6) disk manifold with our optional adapter (#ECUCTADP). The cartridge also fits a standard vacuum manifold (#VMF016GL).

2) PAR standards may be prepared by dissolving 20 mg of stearic acid and 20 mg of hexadecane in 5 mL of acetone.

3) To achieve good flow if very high solids are present, add glass wool to the cartridge prior to extraction to prevent clogging. The glass wool must be thoroughly rinsed with hexane as part of the cartridge during the elution step.

4) Procedures for drying the hexane extract:
   - Place a plug of glass wool in the bottom of a small funnel, then add 3-5 grams of sodium sulfate to the top.
   - Record the weight of a clean vial or weigh pan and place under the funnel
   - Pour the hexane from the eluate onto the bed of sodium sulfate
   - Rinse the sides of the vial and the sodium sulfate bed with clean hexane and collect in the weighed vial
   - Evaporate the hexane and report the results as mg/L HEM

5) Gloves are recommended when handling the vial as skin oils may affect the actual sample weight.

6) It is important that the extract not be over dried or dried at high temperatures as low recoveries may result by evaporation of volatile oils.

For Method 1664 updates see: http://www.epa.gov/waterscience/methods/

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