

USEPA¹ Hexane Extractable Gravimetric Method

Method 10056

15 to 3000 mg/L HEM and SGT-HEM

Scope and application: For water, wastewater, brine solutions, produced waters and hydraulic fracturing waters.

¹ This procedure is equivalent to USEPA Method 1664. Adapted from *Standard Methods for the Examination of Water and Wastewater*, Section 5520B.



Test preparation

Before starting

Determine a blank value (350 mL of distilled or deionized water) with each new lot of reagents. If the blank result is greater than 5 mg, correct the source of error or remove interferences before analysis.

Use the equivalent quantity of acid to determine the blank and all samples from each sampling source.

Let the sample temperature increase to room temperature before analysis.

Do not use plastic tubing to pour the solvent between containers.

Do not rinse the collecting vessel with sample before analysis.

Anhydrous sodium sulfate is used to remove traces of water from the hexane extraction layer. Dry the sodium sulfate at 200–250 °C for 24 hours for best performance.

Spilled reagent can have a negative effect on the test accuracy. Spilled reagent is hazardous to skin and other materials.

To determine both, the HEM and the SGT-HEM, clean and dry two distillation flasks (one for each procedure) before analysis.

- HEM = n-hexane extractable materials
- SGT-HEM = Silica Gel Treated n-hexane extractable materials

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Description	Quantity
Hydrochloric Acid Solution, 6.0 N (1:1)	4 mL
Hexane, ACS grade	100–200 mL
pH paper	varies
Silica gel with indicator (for desiccator)	varies
Silica gel, 100–200 mesh	1–30 g
Sodium sulfate, anhydrous	10 g
Adapter, vacuum connector/gas inlet, 28/15	1
Aspirator, vacuum pump	1
Balance, analytical, 115 VAC 60 Hz	1
Boiling chips, silicon carbide	3–10
Clamp, 3-prong	2
Clamp holder	2

Items to collect (continued)

Description	Quantity
Clamp, pinch type, No. 28, F/Glass Joints	2
Condenser, reflux, with ground glass joints, 28/15	1
Cylinder, graduated, 500-mL	1
Cylinder, graduated, 50-mL	1
Desiccator	1
Desiccator plate	1
Filter funnel, 65-mm, short stem	1
Filter paper, 12.5-cm, folded, pore size 8 to 12 µm	1
Flask, Erlenmeyer, 125-mL	1
Flask, Erlenmeyer, 125-mL, with ground glass joint 28/15	2
Funnel, separatory, 500-mL	1
Marker, laboratory	1
Oven, drying, 120 VAC, 50 Hz	1
Pipet filler, safety bulb	1
Pipet, serological, 5-mL	1
Ring support, 4-inch	1
Rod, glass	1
Steam bath, 8-inch, 5-ring	1
Hot plate, 7 inch x 7 inch, digital, 120 VAC	1
Stir bar, 22.2 x 7.9 mm	1
Support, ring stand, 5-inch x 8-inch base	2
Tongs, crucible, 9-inch	1
Tube, connecting, J-shaped, with ground glass joint, 28/15	1
Tubing, rubber, 7.9 mm x 2.4 mm	1

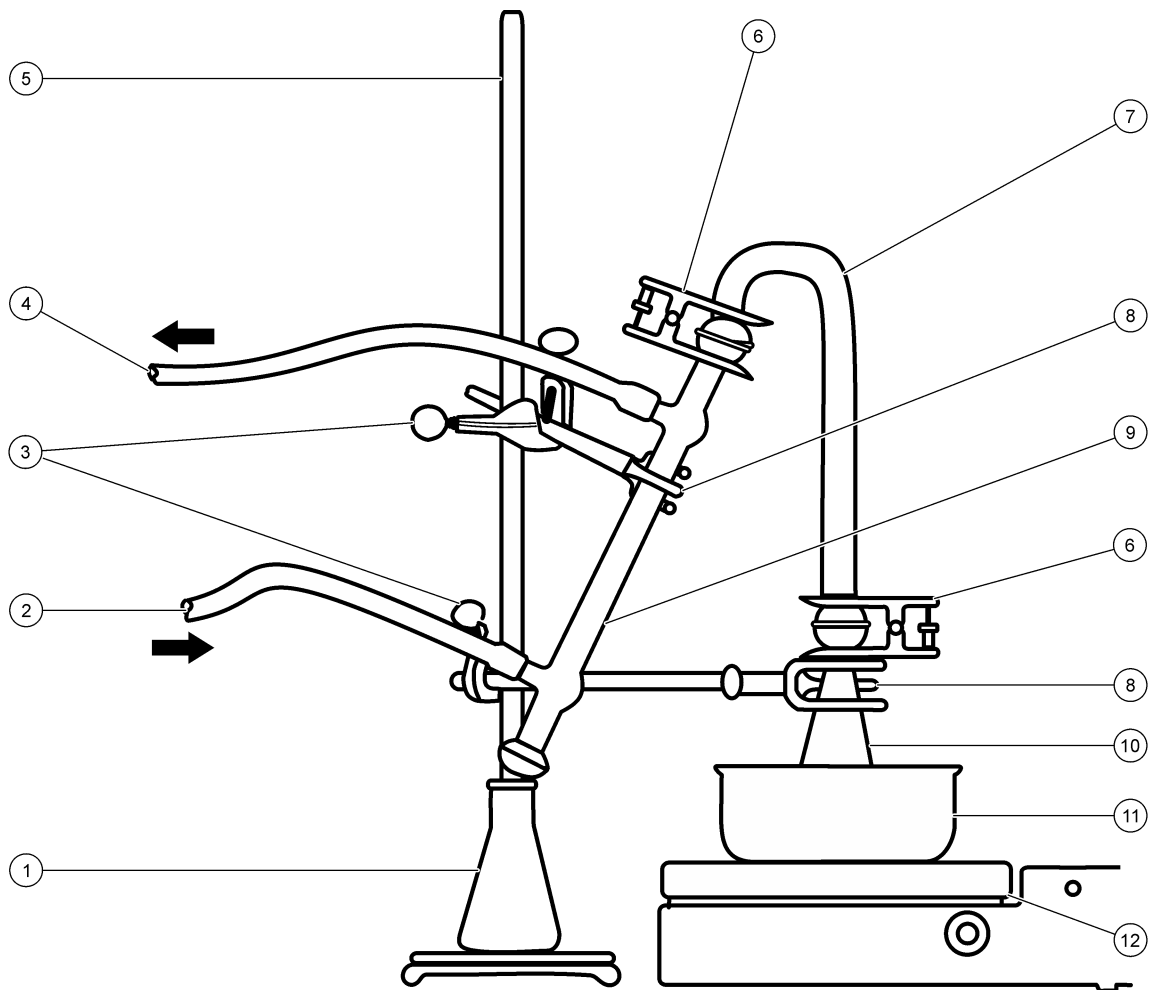
Refer to [Consumables and replacement items](#) on page 13 for order information.

Assemble the distillation apparatus

The distillation apparatus is necessary to determine HEM. Refer to [Figure 1](#).

Note: *If only SGT-HEM is determined and the HEM is known, distillation is not necessary.*

Figure 1 Distillation apparatus assembly



1 Receiving flask	5 Support stand and rod assembly	9 Condenser
2 Water in	6 Pinch clamp	10 Distillation flask
3 Clamp holders	7 J-Shaped connector	11 Water bath
4 Water out	8 Clamp, 3-Prong	12 Hot plate

Sample collection and storage

- Do not rinse the bottle or the separatory funnel with sample.
- Collect samples in wide-mouth glass bottles or directly in the separatory funnel for immediate analysis.
- If samples are collected directly in the separatory funnel determine the sample volume as follows.
 1. Measure 350 mL of water with a graduated cylinder.
 2. Pour the water into the separatory funnel.
 3. Use a laboratory pen to put a mark on the 350-mL level.
 4. Discard the water. Fill with sample to this mark.
- To preserve samples for later analysis, add 6 mL of 1:1 Hydrochloric Acid Solution for each liter or quart of sample. Use a glass rod and a pH paper to measure the sample pH after the acid addition.
 1. Put the glass rod into the sample momentarily.
 2. Let some drops of sample touch the pH paper. Do not put the pH paper into the sample.
 3. Rinse the glass rod with a small quantity of n-hexane back into the sample container to remove all grease or oil bonded to the rod.
- If necessary add more acid to adjust pH below 2.
- Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 28 days.
- If prompt analysis is not possible, keep the sample at or below 6 °C (43 °F) for a maximum of 28 days.
- Let the sample temperature increase to room temperature before analysis.

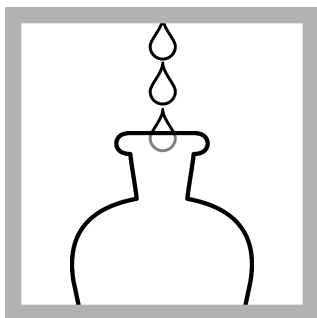
Prepare analysis glassware

Before analysis, it is necessary to carefully clean and dry all of the glassware and boiling chips. Always use tongs or an anti-lint wipe to prevent contamination.

1. Clean the chips and distillation flask with hot water and detergent.
2. Rinse with distilled water. Then, rinse with acetone or n-hexane.
3. Put the cleaned flask and boiling chips in a drying oven at 105–115 °C (220–240 °F) for 2 hours.
4. Let the temperature of the flask and boiling chips decrease to room temperature in a desiccator for at least 30 minutes.
5. Keep the flask and boiling chips in the desiccator for the next use.

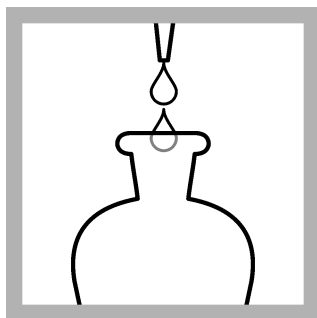
If the same flasks are used again and again, record the weight of the flasks (after each is dried in the oven with no boiling chips). Do not do the drying step, if the weight of a dried flask is the same as the weight of the flask after the acetone or n-hexane rinse. The weight of each boiling chip is not the same. Make sure to add the weight of the boiling chips to the flask weight.

Test procedure



1. Collect 350 mL of sample in a clean 500-mL separatory funnel.

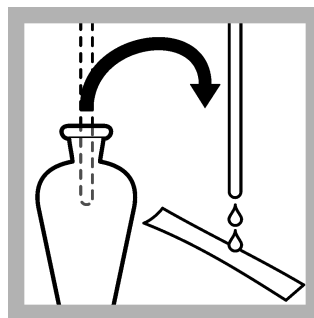
If the sample is not collected in the separatory funnel, set the empty container and lid aside for use in step 5.



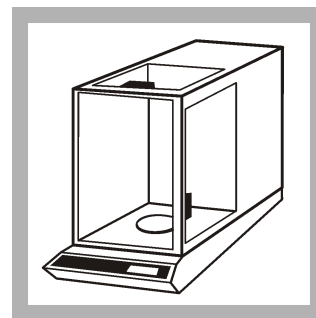
2. Use a pipet and a pipet filler to add 4 mL of 1:1 Hydrochloric Acid solution to the separatory funnel. Mix well.

The pH must be 2 or less to hydrolyze oils and grease and prevent a sodium sulfate interference.

Note: Do not do the acid addition if the sample was preserved with 1:1 Hydrochloric Acid solution.



3. Use a glass rod and a pH paper to measure the sample pH after the acid addition. Refer to [Sample collection and storage](#) on page 4.



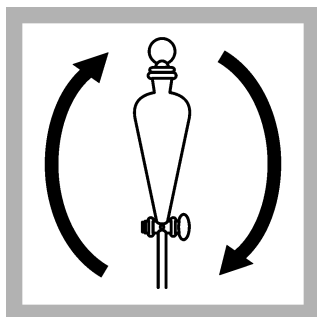
4. Clean and dry a 125-mL distillation flask that contains 3–5 boiling chips. Use an analytical balance to weigh the flask to the nearest 0.1 mg. Record the weight of the flask.



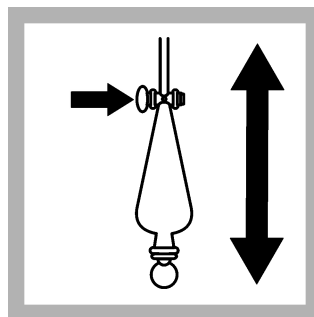
5. Add 20 mL of n-hexane to the separatory funnel.

If the sample was collected in a separate container, rinse the collecting vessel which contained the sample with 20 mL of n-hexane. If this step is for water layer extraction, rinse the volumetric flask that contained the water layer with 20 mL of n-hexane (refer to step 14).

Add the 20-mL n-hexane rinse to the separatory funnel.

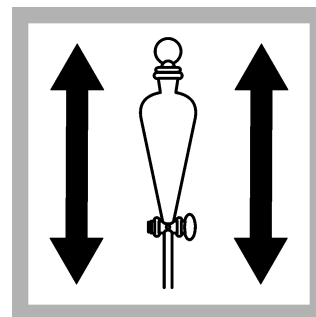


6. Put the stopper and invert the separatory funnel.

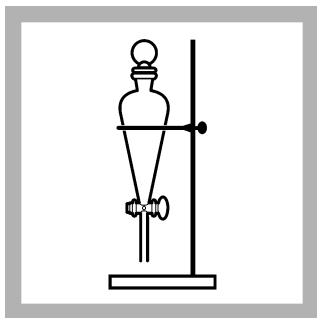


7. Release the gases through the stopcock.

To release gases from the separatory funnel, invert it and shake it once very hard. Make sure to hold the stopper. Under a hood, point the delivery tube in a safe direction. Slowly open the stopcock to release all of the gas. Close the stopcock. Do this procedure until the release of gas is not heard.



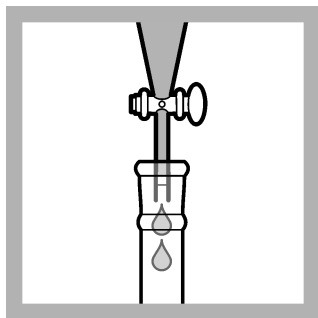
8. Vigorously shake the separatory funnel for 2 minutes.



9. Put the separatory funnel in the stand. Do not move the separatory funnel or the stand for a minimum of 10 minutes to let the separation of the lower water layer and the upper solvent layer.

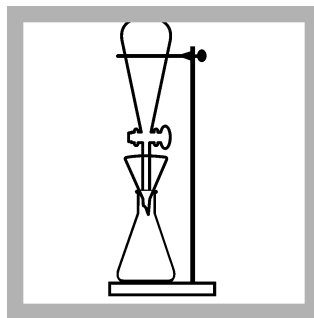
If the solvent layer is brown, the sample can have oil with color on it.

If this step is done again (for a third time), and the water layer is cloudy, do not move the separatory funnel for 20 minutes to make sure of the separation of the water and solvent layers.



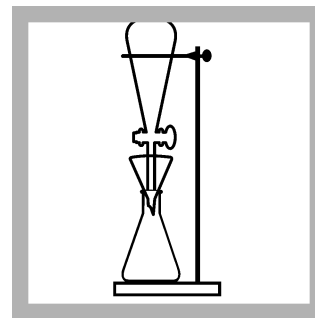
10. Slowly drain the lower water layer from the separatory funnel into the initial sample container or a 500-mL volumetric flask. The drain must take approximately 3–4 minutes. Keep the water layer for use in step 13.

To make sure that water is not used in step 12, let some drops of solvent layer drain into the water layer until the solvent layer is visible on top of the water. If the water layer drains too quickly, there will be too much water in the solvent layer. This causes sodium sulfate and water interference.



11. Set up the filtering funnel. Put the glass funnel in the neck of the distillation flask. Put a folded 12.5 cm filter paper in the funnel. Add 10 g of anhydrous sodium sulfate to the filter paper. Rinse the sodium sulfate with a small amount of n-hexane. Discard the n-hexane correctly.

For the second and third extractions, use the same filter, funnel and sodium sulfate. Between extractions, remove the large, hard sodium sulfate chunks to decrease sodium sulfate contamination.



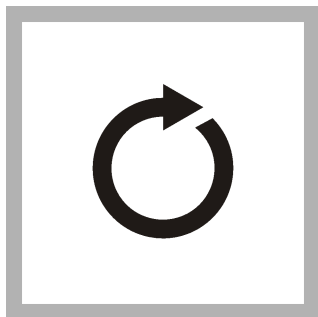
12. Drip-drain the solvent layer into the pre-weighed boiling flask through a funnel that contains filter paper and 10-g anhydrous sodium sulfate. Carefully stir the sodium sulfate with a glass rod while the solvent layer drains. Be careful and do not damage the filter paper.

Any spillage will cause inaccurate results. To reduce spillage, use the glass rod to route the sample solution into the filter.



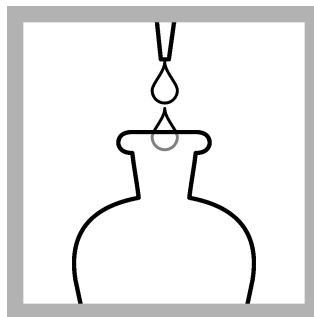
13. Return the water layer to the separatory funnel. Use the same glass funnel for the second and third extraction.

To reduce spillage, use a second funnel to pour the water layer into the separatory funnel.

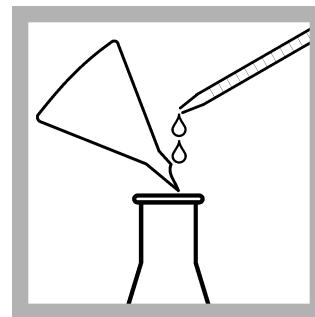


14. Do steps 5 through 13 again two more times. After the third extraction, discard the water layer.

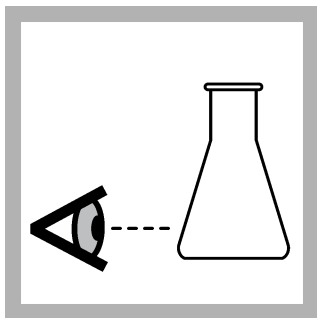
Make sure to correctly discard the water layer. The water layer can have small amounts of acetone and/or n-hexane on it.



15. Rinse the separatory funnel with three different 5-mL aliquots of fresh n-hexane to remove oil film that stayed on the funnel walls. Drain each aliquot through the funnel that contains the sodium sulfate into the distillation flask.

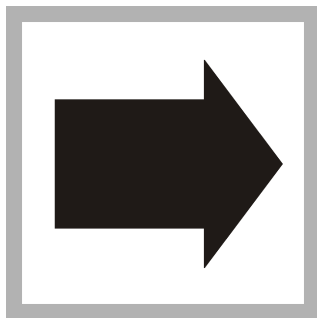


16. Rinse the tip of the glass funnel with 5 mL of n-hexane while removing it from the distillation flask.



17. Examine the distillation flask for sodium sulfate contamination.

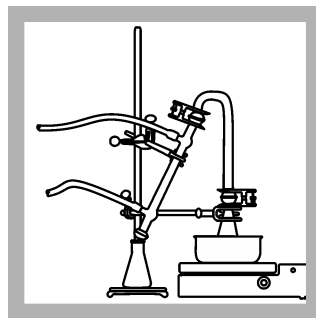
Sodium sulfate contamination will show as cubic crystals at the bottom of the distillation flask. If there is sodium sulfate contamination, filter the solvent layer again through filter paper without sodium sulfate. It is necessary to clean, dry and weigh the boiling flask and boiling chips again. Or make sure to have an additional boiling flask.



18. Continue with step 19 to determine HEM.

If the HEM is known and only the SGT-HEM is analyzed, go to step 26.

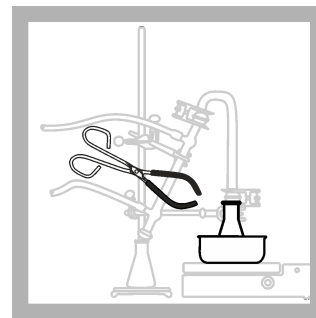
Note: *The HEM value is necessary to determine the quantity of silica gel necessary for the SGT-HEM. For each group of samples from a discharge, determine the HEM before the SGT-HEM.*



19. Use the distillation assembly shown in Figure 1 on page 3 to distill off the n-hexane. Distillation is complete when there are no boiling bubbles or the distillation flask is dry.

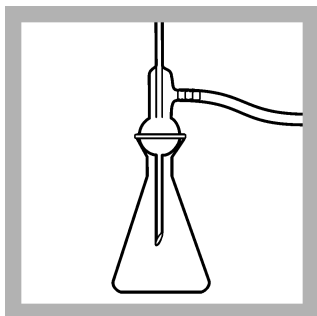
Use a steam bath or a hot plate to keep a water bath at the correct temperature for the distillation. Do not put the flask directly on a hot plate. This will cause low results and is dangerous because n-hexane is volatile.

Evaporation will be faster if the long vertical arm of the condenser is wound with insulation (e.g., paper towel, cloth or asbestos insulating tape). The distillation is complete in 30 minutes or less.

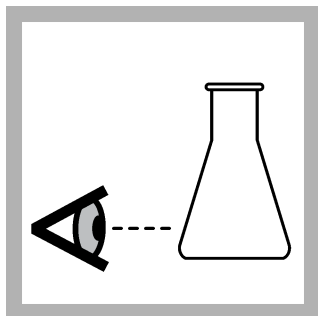


20. Disconnect the condenser/connector portion of the distillation assembly at the pinch clamp. Remove the distillation flask from the heat source with tongs or a lint-free cloth.

The distilled n-hexane is applicable for future HEM extractions, but is not recommended for SGT-HEM because of the possible increase of water content of the solvent.



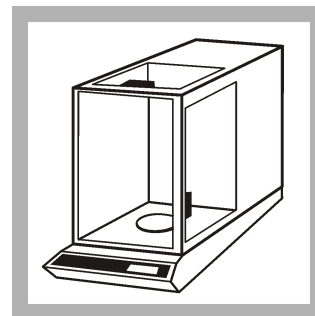
21. Attach the vacuum connector/gas inlet adapter to remove the remaining solvent vapors from the distillation flask. Apply a vacuum for 1–2 minutes or until all n-hexane solvent vapors are removed.



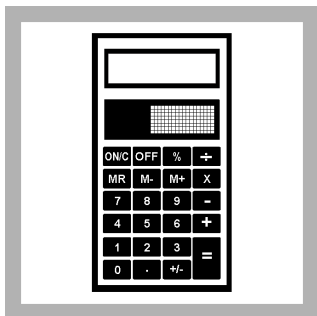
22. Examine the distillation flask for sodium sulfate contamination. Sodium sulfate contamination will show as cubic crystals at the bottom of the distillation flask. If there is sodium sulfate contamination, dissolve the extract again in n-hexane, filter into another pre-weighed flask and do steps 18–20 again. This is not necessarily true for the “standard” extraction since stearic acid is crystalline below 69 °C (156 °F). If there is sodium sulfate in the standard, big cubical crystals (not the flattened stearic acid crystals) will show. Also, an unusually high yield compared to the expected value will result.



23. Put the flask in a desiccator for 30 minutes (or longer if necessary) until the flask temperature decreases to room temperature. If the silica gel indicator changes to red, replace the silica gel.



24. Use an analytical balance to weigh the flask to the nearest 0.1 mg. Record this weight. Do not touch the flask it is weighted because fingerprints add weight. Always use a tong or a lint-free wipe to touch the flask. Precise weigh measurement is necessary for accurate results. Multiple weight measurements are recommended. Clean the flask again before each measurement to make sure that all contaminants are removed. Record each weight and use the lowest repeatable value for calculations.



25. Calculate the test results:

$$[(A - B) \div \text{Sample volume}] \times 1000 = \text{mg/L HEM}$$

Where:

A = Weight (mg) of residue

B = Weight (mg) of flask with boiling chips (step 4)

Example:

A = 92.4659 g

B = 92.4206 g

Sample volume = 0.350 L (350 mL)

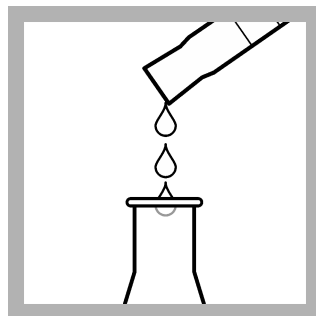
$$[(92.4659 - 92.4206) \div 0.350] \times 1000 = 129.4 \text{ mg/L HEM}$$

If yield is less than 15 mg/L and additional precision is needed, use a 1-liter sample.

For HEM determination stop here and do not continue the procedure. Continue with next step to determine SGT-HEM.



26. Dissolve the residue again with approximately 85 mL of fresh n-hexane. Heat slightly to make sure that all HEM materials are dissolved.



27. If necessary, dilute the dissolved HEM. For a 350-mL water sample, dilution is necessary if the HEM is more than 2850 mg/L (more than 1000 mg/L for a 1-L water sample).

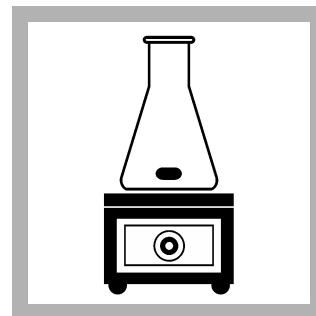
To dilute to a 1000 mg/L sample, pour the dissolved HEM into a 100-mL volumetric flask. Rinse the distillation flask 3–4 times with 2–3 mL of n-hexane. Fill the volumetric flask to volume with n-hexane. Mix well. Use a volumetric pipet to add into a 100-mL beaker the quantity (V_a) determined by this equation:

$$V_a = 10,000 \div W_h$$

Where:

V_a = Volume of aliquot to be withdrawn (mL) to get 1000 mg of HEM

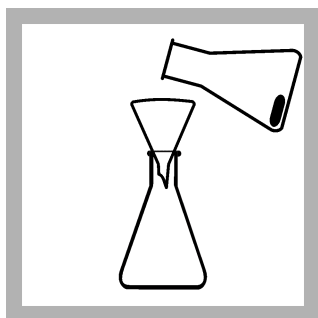
W_h = Weight of HEM (mg) $(A - B) \times 1000$ in step 24 (mg). Dilute to about 100 mL with n-hexane.



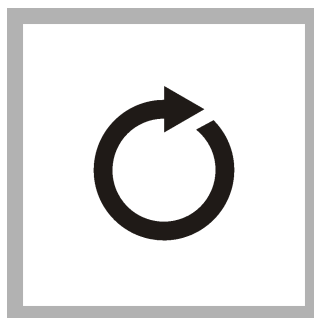
28. Put a magnetic stir bar and the correct amount of silica gel (based on the equation below) into the flask with the solvent/product from step 27. $(3 \times \text{mg/L HEM}) \div 100 = \text{silica gel (g} \pm 0.3)$



29. Stir the solution on a magnetic stirrer for a minimum of 5 minutes.



30. Put a funnel on a clean, dry distillation flask with 3–5 boiling chips in it. Put a 12.5-cm filter paper in the funnel. Pre-moisten the filter paper with fresh n-hexane. Filter the solution through filter paper. Rinse the beaker that contains the remaining silica gel three times with 5-mL aliquots of fresh n-hexane. Pour the aliquots into the distillation flask.



31. Do steps 19 through 25. Weigh the product that remains in the bottom of the flask. Calculate the results with this equation:

$$(A - B) \div \text{Sample Volume} = \text{mg/L SGT-HEM}$$

Where:

A = Weight (mg) of residue

B = Weight (mg) of flask with boiling chips.

Interferences

Substances extracted from samples will change from source to source. This change is dependent on the diversity of the site where the samples are collected. Some samples can contain high quantities of detergents or particulates that can interfere with the extraction procedure. For these samples, use a 350-mL sample as an alternative to the 1-liter sample (which is an option). In this condition, the 350-mL sample is EPA accepted for reporting. Wash all glassware in hot water with detergent, rinse with tap and distilled water and rinse with n-hexane or acetone.

If an emulsion forms between the two phases (at step 9) and is more than one-third of the volume of the solvent layer, filter the emulsion and solvent layer through a funnel with glass wool in it. Other possible solutions to remove the emulsion are: stir the solvent and emulsion layer with a stir bar, use the solvent phase separation paper, use centrifugation, use an ultrasonic bath with ice, add NaCl or apply other physical methods. Solid phase or other extraction techniques are performance-based modifications.

If there is water in the solvent layers, a milky solvent/product layer shows in the distillation flask. Do not move the flask for 1 hour to let the water collect at the bottom. Filter the solvent layer again through sodium sulfate to remove the remaining water.

Extremely low yields could indicate an unsatisfactory extraction (step 6 through step 11) and a high yield could indicate a problem in the drying process of the solvent (step 11). Follow these steps very carefully. Do a test in the blank before the test in the samples to identify possible interferences related to these steps. If the blank shows a yield above 1 mg for each test, do not continue until the source of contamination is identified. Usual contamination sources are sodium sulfate contamination and improperly rinsed glassware.

The method can measure HEM and SGT-HEM in the range of 15–3000 mg/L when a 350-mL sample is used. The lower limit can decrease to 5 mg/L if a 1-liter sample is used. If the 1-liter sample volume is used, refer to [Accuracy check](#) on page 11 for the amount of reagents to use.

Detection limit

This method is not applicable to measurements of materials that volatilize at temperatures below approximately 85 °C (185 °F). Petroleum fuels from gasoline through #2 fuel oil can be partially lost in the solvent removal operation. Some crude oils and

heavy fuel oils contain a important percentage of materials that are not soluble in n-hexane. Recoveries of these materials can be low.

Accuracy check

Standard preparation

Items to collect:

- Stearic acid, 98% minimum
 - Hexadecane, 98% minimum
 - Acetone for Organic Residue Analysis, residue less than 1 mg/L
 - 100-mL Class A volumetric flask
 - 10.0-mL Class A volumetric pipet
1. Put 400 (\pm 4) mg stearic acid and 400 (\pm 4) mg hexadecane into a 100-mL volumetric flask.
 2. Add 75 mL of acetone to the flask. Cover with a small beaker. Stir the solution gently. Heat slightly until all material is dissolved. Over-heating with the lid on causes pressure build up.
 3. Fill to volume with acetone. Cover with a small beaker. Let the solution temperature decrease to room temperature. Continue to fill to volume until solution is at stable volume.
 4. Use a pipet to add 5 mL of the solution from step 3 into 350 mL of deionized reagent water. This standard solution must be 114.3-mg/L HEM or 57.1-mg/L SGT-HEM. If 1-liter reagent water is used, 5 mL gives concentrations of 40 mg/L HEM and 20 mg/L SGT-HEM.

Note: To verify the concentration, use a pipet to add 5 mL of the solution from step 3 in a pre-weighed flask. Put the flask in a hood to let the acetone dry. Weigh the flask. Verify that the weight difference before and after solution addition is 40 (\pm 1) mg.

EPA monitoring

If the Oil and Grease tests are used for compliance reporting to the USEPA, make the changes that follow to the procedure:

1. Use a 1-liter sample in a 2000-mL separatory funnel rather than a 350-mL sample in a 500-mL separatory funnel (step 1).
2. Use 6 mL (instead of 4 mL) of 1:1 hydrochloric acid to adjust the pH below 2 (step 2) and 30 mL of n-hexane instead of 20 mL of n-hexane for the extraction (step 5).

MDL standard solution

The recommended standard concentration to determine the minimum detection limit (MDL) is approximately 5 mg/L.

1. To prepare the standard for HEM complete steps 1–3 in [Standard preparation](#) on page 11, but change step 1 to transfer 100 (\pm 4) mg stearic acid and 100 (\pm 4) mg hexadecane to a 250-mL volumetric flask.
2. To prepare the standard for SGT-HEM standard, put 200 (\pm 4) mg of decahexane only into a 250-mL volumetric flask. Transfer 5 mL of one of the two standards into 1-liter of reagent water.

Analysis of the standard must give 5 mg/L for HEM or SGT-HEM.

IPR standard solution

Use deionized water—void of any oil and grease—as the blank (standard solution) for the initial precision and recovery (IPR).

Complete the procedure four separate times with 5 mL of the standard (40 mg/L 1:1 stearic acid/hexadecane) diluted into 1 liter of demineralized water. Refer to [EPA requirements for MDL and IPR](#) on page 12.

EPA requirements for MDL and IPR

Before analysis on real samples for oil and grease, the user must get a MDL less than or equal to the EPA reported MDL and to report an IPR. It is highly recommended that the laboratory reagent water blanks are measured to remove all interferences before the MDL and IPR are measured.

MDL: Complete the procedure seven separate times with the standard solution. Find the standard deviation and multiply the standard deviation by 3.143 (Student's t test). The permitted limits are:

- HEM: ≤ 1.4 mg/L
- SGT-HEM: ≤ 1.6 mg/L

IPR: Complete the procedure for HEM and SGT-HEM (if necessary) four separate times with the standard solution. Report the average percent recovery (x) and the standard deviation for both HEM and SGT-HEM. The permitted limits are:

- HEM: Precision(s) ≤ 10 %; Recovery (x) 83–101 %
- SGT-HEM: Precision(s) ≤ 13 %; Recovery (x) 83–116 %

If not within these ranges, correct the problem and do IPR again.

After get satisfactory values for the MDL and IPR, keep records for USEPA verification.

Report the test results to the EPA

Include the data that follows with the HEM and/or SGT-HEM results for each set of 10 (maximum) samples for each discharge source.

1. **Blank value:** The value must be less than 5.0 mg/L for HEM and SGT-HEM.

Note: Use a standard that agrees with the regulatory concentration limit. This concentration is 1–5 times higher than the concentration of the sample (B) or is the same concentration as the OPR, the one that is highest. Divide the concentration of the spike (T) by 2 for SGT-HEM if the standard is used (40 mg/L 1:1 stearic acid/hexadecane).

2. **OPR (Ongoing Precision and Recovery):** Add 5 mL of the standard (40 mg/L 1:1 stearic acid/hexadecane) to a 1-liter sample and complete the test. The permitted limits for recovery are:

- HEM: 70–114%
- SGT-HEM: 66–114%

If recovery is lower, there is a possible interference or the technique is not correct. Identify the cause and do OPR again until within the range.

3. **MS and MSD (matrix spike and matrix spike duplicate):** Measure the HEM and SGT-HEM concentration of the sample (B). Spike two 1-L samples with 5 mL of the standard and measure the concentration after spiking (A).

Calculate the Percent Recovery (P) as follows:

$$P_{\text{HEM}(40 \text{ mg/L})} = [100 \times (A - B)] \div T$$

$$P_{\text{SGT-HEM}} = [100 \times (A - B)] \div (T \div 2)$$

Where:

A = concentration of the unspiked sample

B = concentration of the spiked sample

T = concentration of the spike solution

If the recovery for HEM and SGT-HEM is within the permitted limits for OPR, then calculate the Relative Percent Difference (RPD).

$$\text{RPD} = [(Conc_{\text{MS}} - Conc_{\text{MSD}}) \div (Conc_{\text{MS}} + Conc_{\text{MSD}})] \times 200$$

If the RPD for HEM is ≤ 18 and for SGT-HEM ≤ 24 , then continue to the next step. If the recovery is lower than the RPD, there is a possible interference. Identify and correct the interference, then do the MS and MSD measurement again.

After every five MS/MSD tests, calculate the average percent recovery (Pa) and standard deviation of the percent recovery (sp). Record these numbers as $Pa \pm 2sp$.

Update the accuracy assessment on a regular basis (e.g., after 5–10 new accuracy measurements).

- 4. Balance calibration:** Measure a 2 mg and a 1000 mg class “S” weight on the analytical balance before and after each analytical batch. If the values are not within 10% of the actual weight, calibrate the balance.

Each laboratory must first verify the MDL and IPR and make sure that they are within correct parameters before oil and grease test results are reported to the EPA. Once this is established for a laboratory, it does not need to be done again.

For each 10 samples of each discharge source, calibrate the balance, report one blank, one OPR, one MS and one MSD. The user must keep logs on percent recovery and relative percent differences for MS/MSD tests. For each five MS/MSD test, calculate and record the average percent recovery and standard deviation.

Summary of method

Oil and Grease and Total Petroleum Hydrocarbons (TPH) include any material collected as a substance that is soluble in the n-hexane extractant. These include substances such as relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related materials. When measuring oil and grease (HEM) gravimetrically, the substances are removed from the sample with n-hexane, then the n-hexane is dried. The residue left is weighed to determine the concentration of oil and grease materials in mg/L.

When Total Petroleum Hydrocarbons (SGT-HEM) is gravimetrically measured, the substances are removed from the sample with n-hexane, then mixed with silica gel to absorb non-TPH components. Then, the n-hexane is dried. Like the HEM, the residue left is weighed to determine the concentration of total petroleum hydrocarbons.

Definition of HEM and SGT-HEM

The term oil and grease was used to define pollutants of this nature. The newer term n-Hexane Extractable Materials (HEM) shows that can apply this method to materials other than oils and greases.

Likewise, the term Total Petroleum Hydrocarbons (TPH) was used to classify aliphatic hydrocarbon materials. The newer term Silica Gel Treated n-Hexane Extractable Material (SGT-HEM) shows that can apply this method to materials other than aliphatic petroleum hydrocarbons that are not adsorbed by silica gel.

Note: Careful technique is necessary for accurate results.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Hydrochloric Acid Solution, 6.0 N (1:1)	varies	500 mL	88449
Hexane, ACS grade	100–200 mL	500 mL	1447849
pH paper	varies	100/pkg	2601300
Silica gel with indicator (for desiccator)	varies	454 g	1426901
Silica gel, 100–200 mesh (for SGT-HEM)	1–30 g	500 g	2665034
Sodium sulfate, anhydrous	10 g	113 g	709914

Required apparatus

Description	Quantity/test	Unit	Item no.
Adapter, vacuum connector/gas inlet, 28/15	1	each	1433900
Aspirator, vacuum pump	1	each	213100
Balance, analytical, 115 VAC	1	each	2936801

Required apparatus (continued)

Description	Quantity/test	Unit	Item no.
Boiling chips, silicon carbide	2–3	500 g	2055734
Clamp, 3-prong	1	each	42200
Clamp holder	1	each	32600
Clamp, pinch type, No. 28, F/Glass Joints	2	each	1433800
Condenser, reflux, with ground glass joints, 28/15	1	each	1433700
Cylinder, graduated, 500-mL	1	each	50849
Cylinder, graduated, 50-mL	1	each	50841
Desiccator	1	each	2088800
Desiccator plate, ceramic	1	each	1428400
Filter funnel, 65-mm, short stem	1	each	2664700
Filter paper, 12.5-cm, folded, pore size 8 to 12 µm	1	100/pkg	69257
Flask, Erlenmeyer, 125-mL	2	each	50543
Flask, Erlenmeyer, 125-mL, with ground glass joint 28/15	2	each	1434000
Funnel, separatory, 500-mL	1	each	52049
Marker, laboratory	1	each	2092000
Oven, drying, 120 VAC	1	each	1428900
Pipet filler, safety bulb	1	each	1465100
Pipet, serological, 5-mL	1	each	53237
Ring support, 4-inch	1	each	580-01
Rod, glass	1	3/pkg	177001
Steam bath, 8-inch, 5-ring	1	each	2347900
Hot plate, 7 inch x 7 inch, digital, 120 VAC	1	each	2881500
Stir bar, 22.2 x 7.9 mm	1	each	2095350
Support, Ring Stand, 5-inch x 8-inch base	1	each	56300
Tongs, crucible, 9-inch	1	each	56900
Tube, connecting, J-shaped, with ground glass joint, 28/15	1	each	1814300
Tubing, rubber, 7.9 mm x 2.4 mm	varies	12 ft	56019

Recommended standards

Description	Unit	Item no.
Hexadecane, 99%, 400 mg	100 mL	2664842
Stearic Acid, 400 mg	500 g	2664934

Optional reagents and apparatus

Description	Unit	Item no.
Acetone, ACS	500 mL	1442949
Separatory funnel, 2-liters	each	52054
Beaker, 50-mL	each	50041H
Weight set	each	2617601

Optional reagents and apparatus (continued)

Description	Unit	Item no.
Hotplate/Stirrer 7.25 x 7.25 in., 220–240 VAC	each	2881602
Ring support, 4.5 in.	each	2656300
Furnace, Muffle, 120 VAC	each	1429600
Pipet, volumetric 5.0 mL	each	14515-37
Flask, volumetric	100 mL	14574-452
Cylinder, graduated, 100 mL	each	508-42



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