Oil and Grease

DOC316.53.01186

USEPA[1] Solid Phase Extraction Method — Method 10300

5 to 1000 mg/L HEM and SGT-HEM — Hexane Extractable Gravimetry

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

[1] This procedure is equivalent to USEPA Method 1664A, Solid Phase Extraction (SPE)

Test preparation

Before starting

Make sure to obey the instructions in Sample collection and storage on page 3 for sample collection and acidification.
Refer to Figure 1 on page 2 to assemble the Xenosep SPE apparatus. Make sure to put the waste collection tube in the large flask before the funnel assembly is installed. Refer to Figure 2 on page 3 to assemble the funnel. Make sure to put the pattern side of the SPE filter down in the SPE filter support.
Wash all glassware in hot water with detergent, rinse with tap and distilled water. Then, rinse with acetone or n-hexane.
Fully rinse all glassware with n-hexane to make sure that the analyte is removed from the apparatus. One incorrect rinse will cause low recovery.
Determine a blank value (1 liter 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.
To analyze SGT-HEM rinse the inner cavity of the aluminum dish with a few milliliters of acetone. Then, use a few milliliters of n-hexane to remove all possible artifacts.
Put the dishes in a drying oven at 103–105 °C (217–221 °F) for 1 hour. Keep the dishes in the desiccator for the next use .
Make sure to remove the aluminum dish from the hot plate before the solution has fully evaporated. Do not apply too much heat to the solution because it will cause low recovery.
Use a vacuum pump that can generate a minimum of 1 CFM free air flow (3 CFM recommended). If the SPE dish is not sufficient dried, it will cause low recovery.
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 N (1:1)	6 mL
Hexane (n-hexane) in Teflon-FEP wash bottle	1 wash bottle
Methanol in PE wash bottle	1 wash bottle
Deionized water in PE wash bottle	1 wash bottle
SPE Starter Kit, EPA Method 1664A	1 kit
SPE Consumables Kit	1 kit
SPE Solvent Recovery Kit	1 kit
Aluminum weighing dish	1
Hot plate (Thermolyne)	1
Pump, vacuum, 27 in. Hg, 1.3 CFM	1
pH paper, 0–14 pH units	1

Items to collect (continued)

Description	Quantity
Lab stand	1
Clamp, swivel	2
Desiccator	1
For SGT-HEM	
Silica gel	1 bottle
125-mL Erlenmeyer flask	1
100-mL volumetric flask (for HEM results over 1000 mg/L)	1
Magnetic stir plate	1
Aluminum weighing dish	1
Additional sodium sulfate column	1

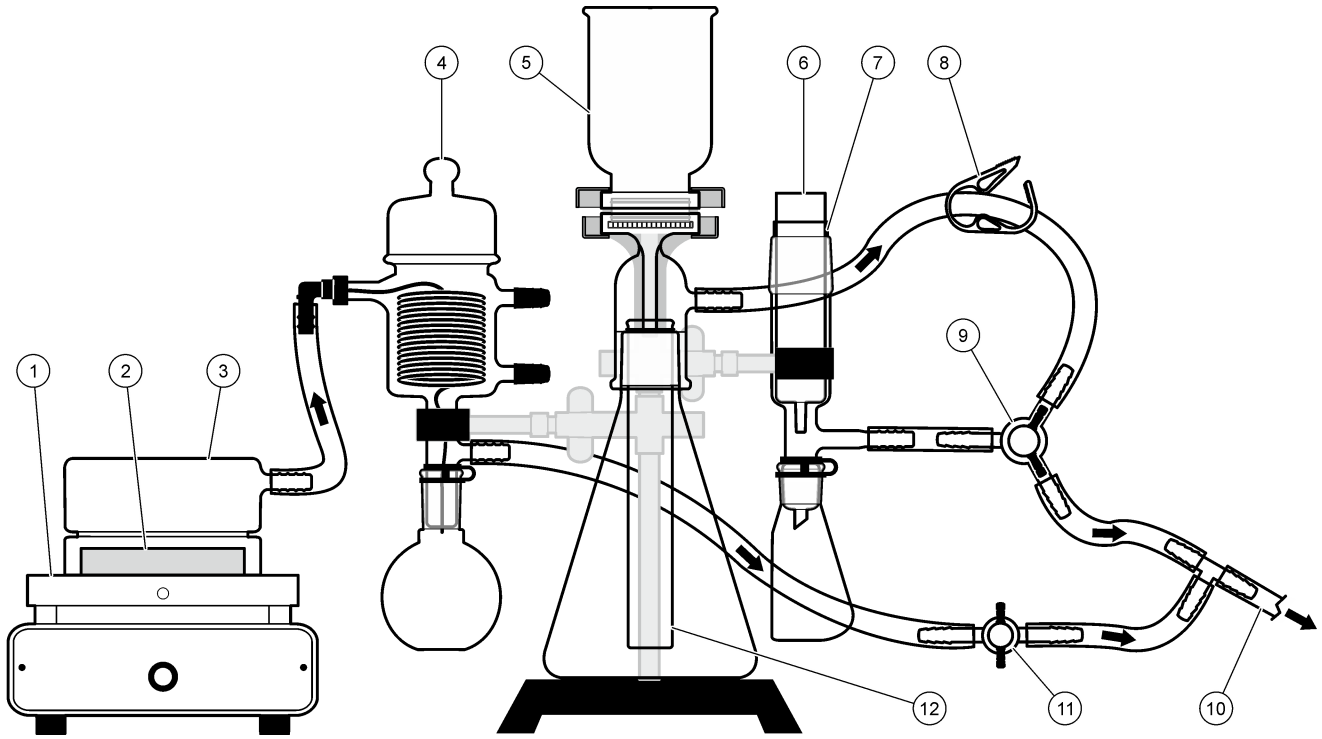
Refer to Consumables and replacement items on page 12 for order information.

Assemble the SPE apparatus

Refer to Figure 1 to assemble the Xenosep SPE apparatus. Make sure to put the waste collection tube in the large flask before the funnel assembly is installed.

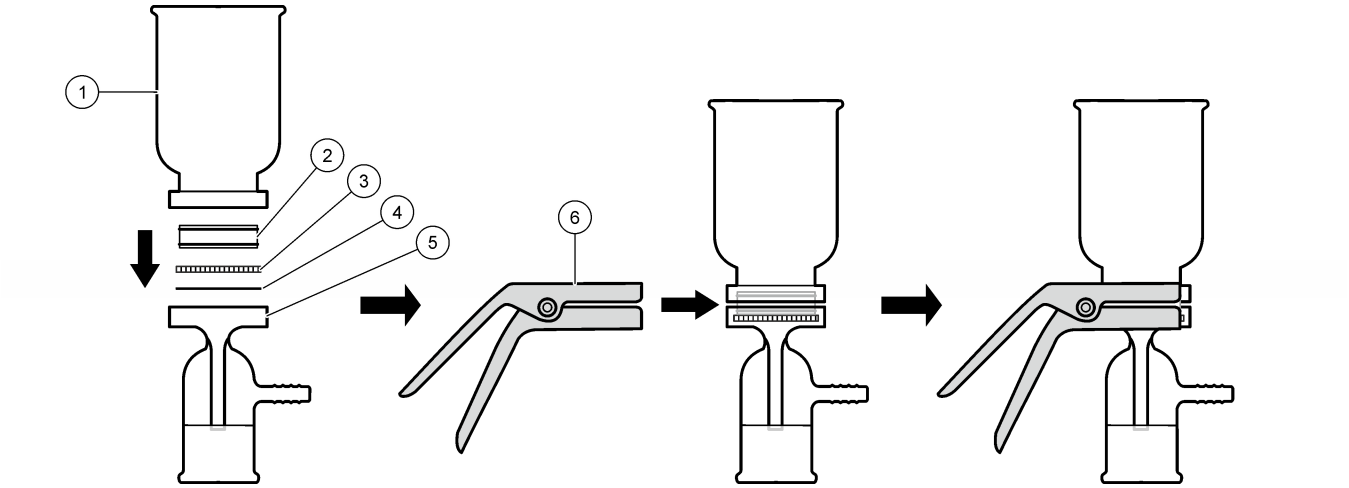
Refer to Figure 2 to assemble the funnel. Make sure to put the pattern side of the SPE filter down in the SPE filter support.

Figure 1 Apparatus assembly for solid phase extraction



1 Hot plate	**5** Funnel assembly (refer to Figure 2)	**9** 3-way valve
2 Aluminum dish	**6** Sodium sulfate column	**10** 2-way valve
3 Glass dome	**7** Eluter tube	**11** To vacuum
4 Solvent recovery assembly	**8** Tube clamp	**12** Waste collection tube with O-ring

Figure 2 Funnel assembly



1 Funnel	**4** Stainless steel support
2 Coupler with O-rings	**5** SPE starter holder
3 SPE filter with pattern side down	**6** Aluminum clamp

Sample collection and storage

- Do not rinse the bottle with sample before collection.
- Collect 1 L (950–1050 mL) of sample in a wide-mouth glass bottle.
- Let the sample temperature increase to room temperature before analysis.
- Adust the sample pH to less than 2 with 1:1 hydrochloric acid (HCl) solution before analysis.

Sample volume

Complete the steps that follow to measure the sample volume:

1. Use a laboratory pen to put a mark on the sample bottle at the liquid level of the sample.
2. When the test is complete, fill the bottle to this mark with tap water.
3. Pour the tap water into a 1-L graduated cylinder and record the volume.
4. Use this volume for the sample volume in the final step of the test procedure for HEM or SGT-HEM.

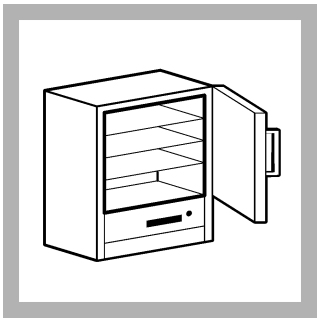
Sample acidification

Complete the steps that follow to find the volume of HCl to use:

1. Collect a separate aliquot of sample.
2. Add HCl until the pH is less than 2.
3. Add this volume of acid to each sample bottle before collection.

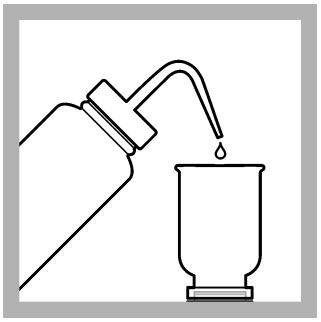
   Note: *Do not put pH paper, a pH electrode, a glass rod or other materials into the sample because oil and grease in the sample can bond to these items.*

Test procedure—HEM

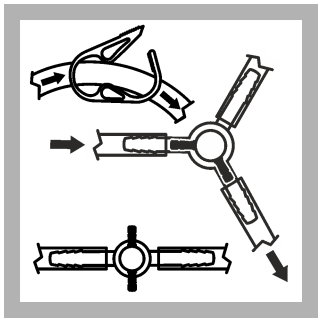


1. Put an aluminum dish in a drying oven at 103–105 °C for 1 hour.

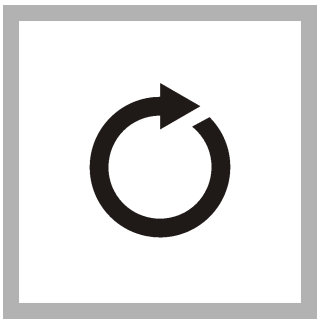
2. Remove the dish from the oven. Let the dish temperature decrease to room temperature in a desiccator.



3. Add approximately 10 mL of n-hexane to the funnel. Wait 5 seconds.

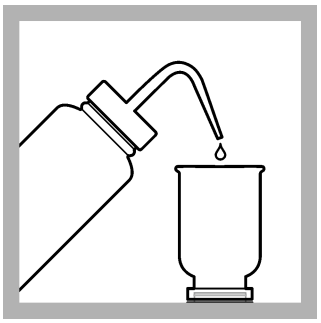


4. Set the vacuum to on and then to off to pull the solvent into the waste collection tube. Make sure that the valves are set to apply vacuum from the funnel holder and the tubing clamp is open.

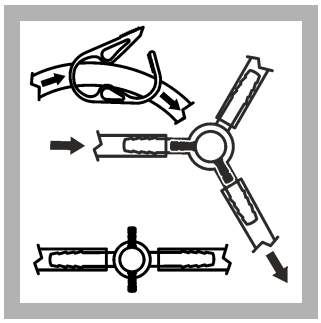


5. Do steps 3–4 again.

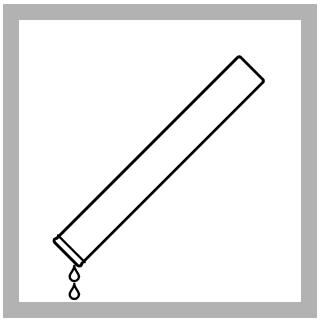
6. Set the vacuum to on for 1 minute to dry the filter. When the timer expires, set the vacuum to off.



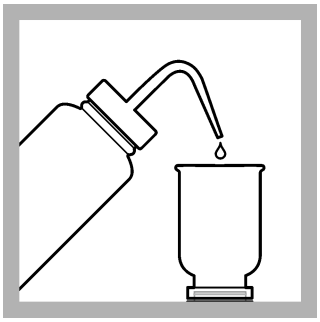
7. Add approximately 10 mL of methanol to the funnel. Wait 5 seconds.



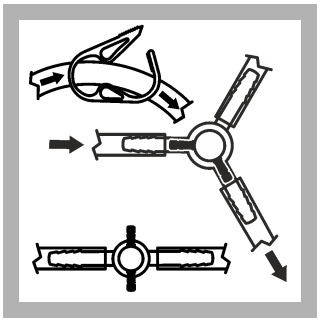
8. Set the vacuum to on and off to pull the solvent into the waste collection tube. Do not let the filter become dry.



9. Remove the waste collection tube. Discard the solvent waste.

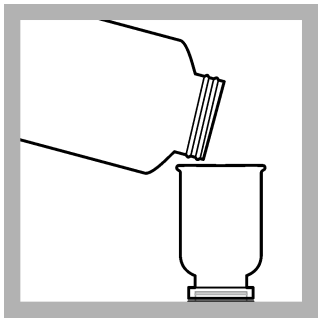


10. Add approximately 20 mL of deionized water to the funnel. Wait 5 seconds.



11. Set the vacuum to on and then to off to pull water into the flask. Do not let the filter become dry.

If the filter becomes dry, do steps 7–11 again.

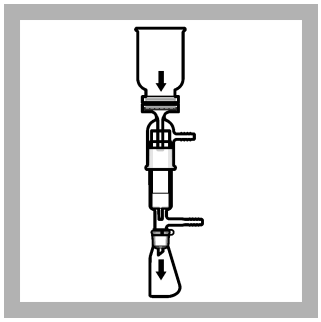


12. Slowly pour the acidified sample into the funnel and set the vacuum to on. Use deionized water to rinse all contamination from the walls of the funnel.

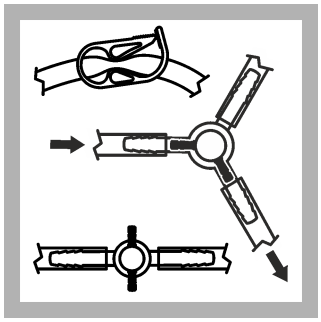
Refer to Sample acidification on page 3.

13. Keep the vacuum on for 4–8 minutes to air dry the filter. When the timer expires, set the vacuum to off.

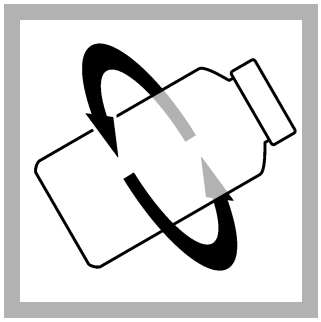
Do not leave the vacuum on for more than 8 minutes.



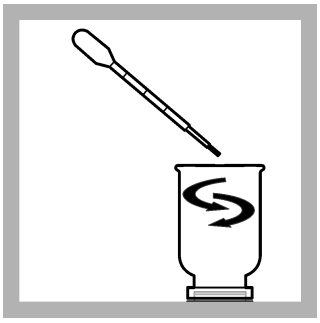
14. Put the funnel assembly on the eluter tube.



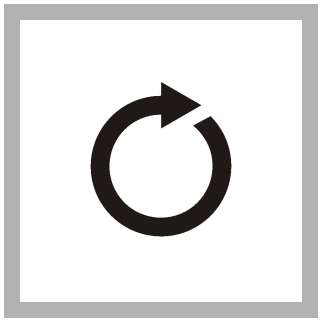
15. Turn the valve to apply vacuum to the eluter tube. Close the tubing clamp on the funnel holder tube.



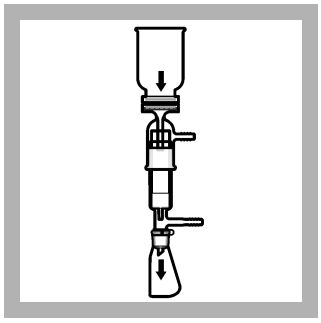
16. Add 10 mL of n-hexane to the empty sample bottle. Swirl the bottle in a horizontal, circular movement for 10 seconds to rinse the bottle.



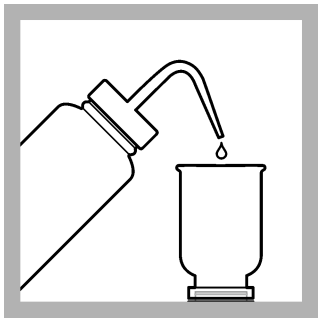
17. Use a transfer pipet to collect the n-hexane from the top of the sample bottle. Slowly rinse the walls of the funnel with the n-hexane. Go around the funnel at least three times.



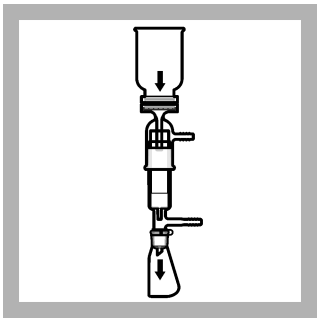
18. Do steps 16–17 again two more times.



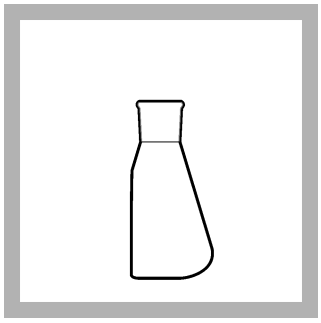
19. Set the vacuum to on and then to off to pull the solvent into the flat-sided flask.



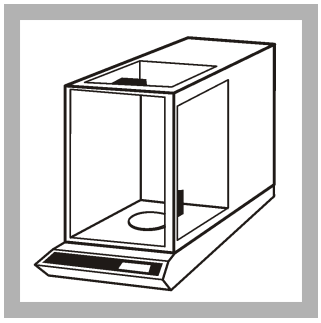
20. Rinse the walls of the funnel with approximately 10 mL of n-hexane. Wait 5 seconds.



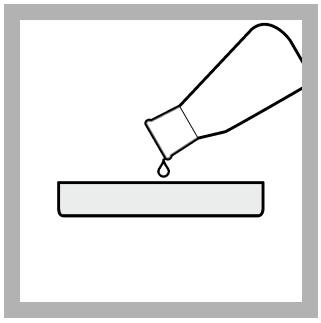
21. Set the vacuum power to on, then to off.



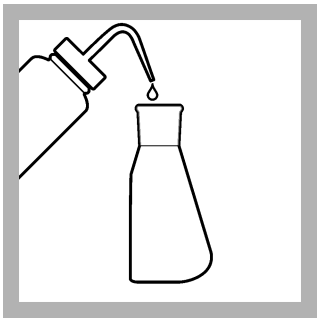
22. Remove the flat-sided flask from the eluter tube.



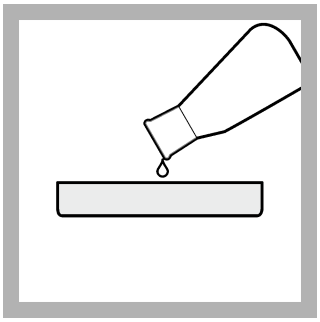
23. Use an analytical balance to weigh the dish to the nearest 0.1 mg (0.0001 g). Record this mg value as B.



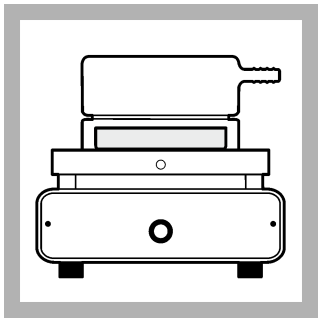
24. Add the n-hexane to the dish.



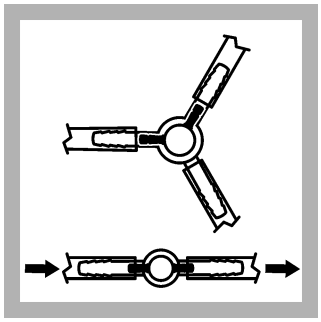
25. Rinse the flat-sided flask with approximately 5 mL of n-hexane.



26. Add the n-hexane to the dish.

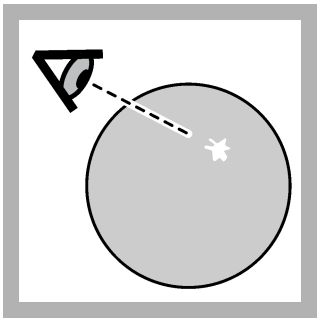


27. Put the dish on the hot plate. Put on the glass cover.



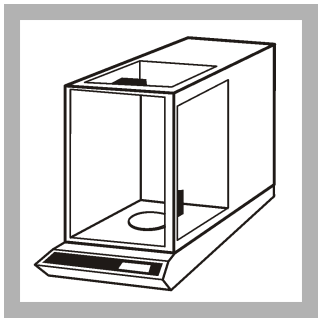
28. Close the 3-way valve and open the 2-way valve to apply vacuum from the solvent recovery apparatus.

29. Set the vacuum to on. Set the hot plate to on at low heat (35–85 ºC). Keep the vacuum and hot plate on for approximately 2 minutes.

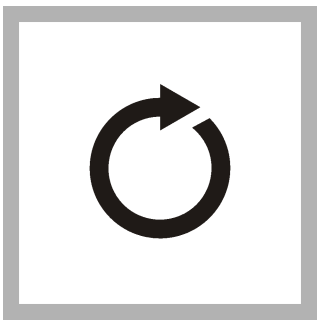


30. Examine the dish for dry spots. As soon as there is a dry spot remove the dish from the hot plate. Put the dish in a fume hood until the rest of the n-hexane has dried.

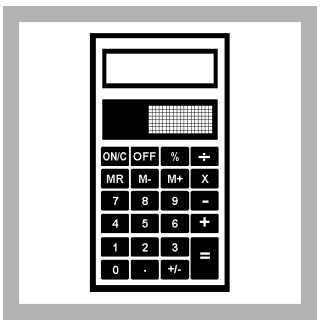
31. When the dish is dry, put the dish in a desiccator for 30 minutes.



32. Weigh the dish to the nearest 0.1 mg.



33. Do steps 31–33 again until the weight loss is less than 0.5 mg from the previous weight. Record this mg value as A.



34. Calculate the test results:

[(A – B) ÷ Sample volume] × 1000 = mg/L HEM

Where:
A = weight of dish with residue (g)

B = weight of dish (g)

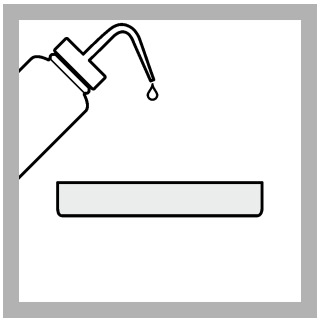
Example:
A = 6.2394 g

B = 6.2318 g

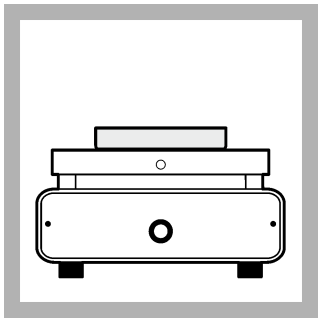
Sample volume = 0.950 L

[(6.2394–6.2318) ÷ 0.950] × 1000 = 8.0 mg/L HEM

Test procedure—SGT-HEM (< 1000 mg/L)



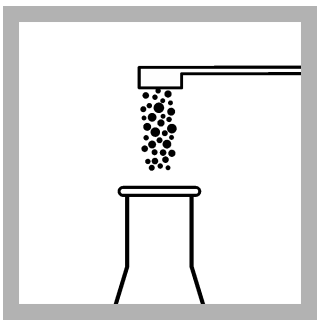
1. Add approximately 60 mL of n-hexane to the residue in the aluminum dish from the HEM test.



2. Use a hot plate to apply low heat to the dish and dissolve all of the residue.

3. Pour the mixture into a 125-mL Erlenmeyer flask. Rinse the pan and funnel several times with n-hexane. Add the mixture to the flask.

4. Add n-hexane to approximately the 100-mL mark.

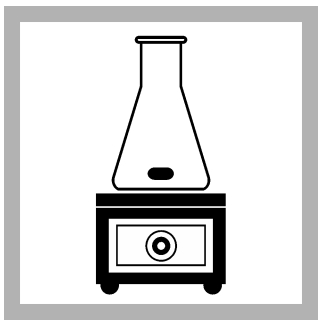


5. Add 3 (± 0.3) g of silica gel to each 100 mg of HEM:

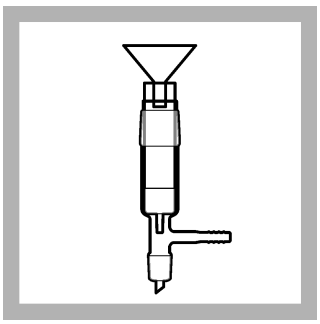
(3 × mg/L HEM) ÷ 100 = silica gel (g)

Example: If the HEM value is 735 mg, round 7.35 g up to the next whole gram increment (8 g or 800 mg) and add 3 × 800/100 = 24 g of silica gel.

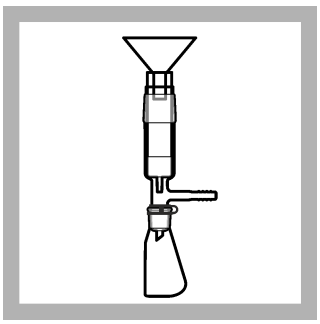
Do not add more than 30 g of silica gel.



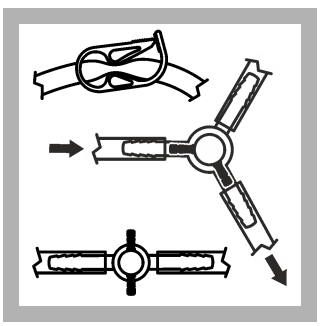
6. Add a stir bar to the flask. Stir for 5 minutes.



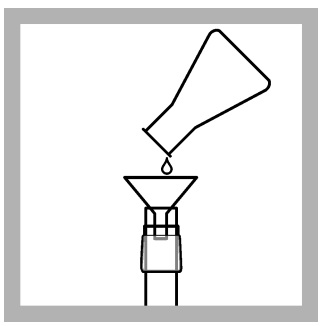
7. Put a new sodium sulfate column into the eluter tube. Put the funnel on the eluter tube.



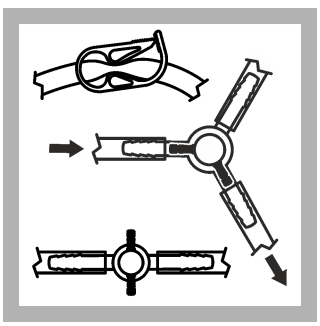
8. Install a clean flat-sided flask.



9. Adjust the valves to apply vacuum from the eluter tube.

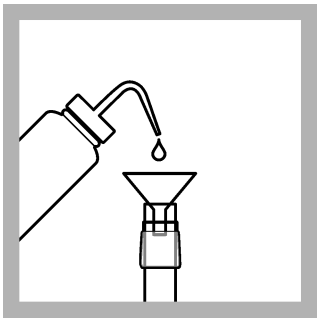


10. Pour the solution from the Erlenmeyer flask into the funnel.

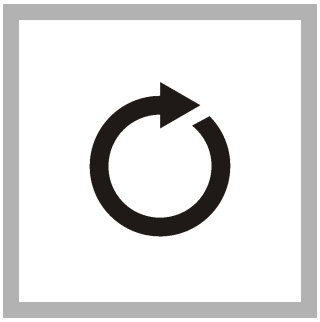


11. Set the vacuum to on and then to off to pull the solution into the flat-sided flask.

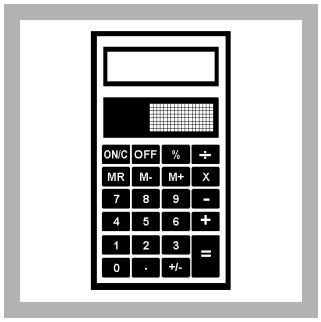
12. Rinse the Erlenmeyer flask with 5 mL of n-hexane. Add the n-hexane to the funnel. Set the vacuum to on and then to off.



13. Rinse the walls of the funnel with approximately 5 mL of n-hexane. Set the vacuum to on and then to off.



14. Complete steps 22–33 of the Test procedure—HEM on page 4 to evaporate the solvent.



15. Calculate the test results:

[(A – B) ÷ Sample volume] × 1000 = mg/L SGT-HEM

Where:
A = weight of dish with residue (g)

B = weight of dish (g)

Example:
A = 6.2360 g

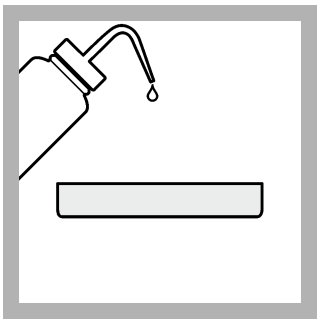
B = 6.2320 g

Sample volume = 0.950 L

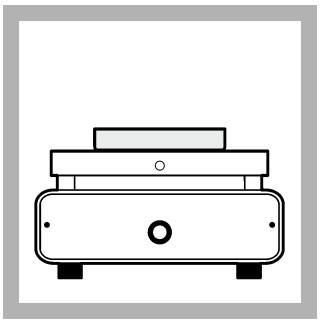
[(6.2360–6.2320) ÷ 0.950] × 1000 = 4.2 SGT-HEM

Report result as ≤ 5 mg/L SGT-HEM

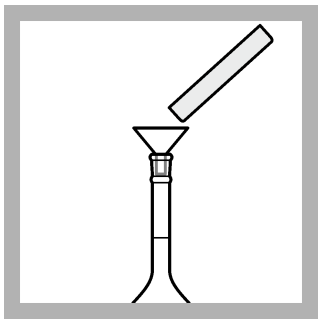
Test procedure—SGT-HEM (> 100 mg/L)



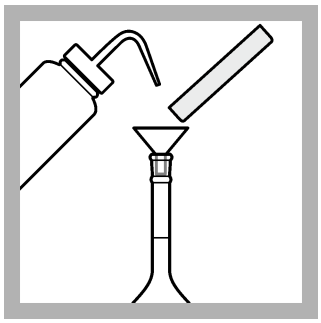
1. Add approximately 60 mL of n-hexane to the residue in the aluminum dish from the HEM test.



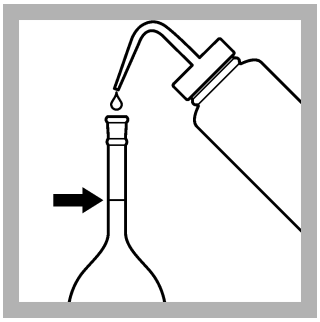
2. Use a hot plate to apply low heat to the dish and dissolve all of the residue.



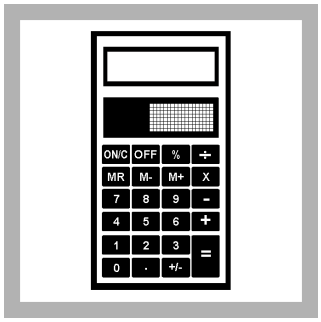
3. Pour the mixture into a 100-mL volumetric flask.



4. Rinse the pan and funnel several times with n-hexane. Add the rinse to the volumetric flask.



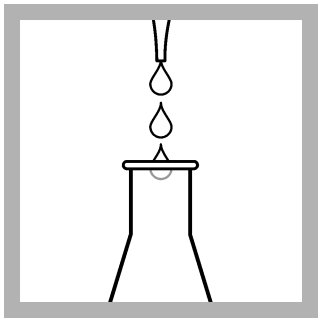
5. Dilute to the mark with n-hexane. Mix well.



6. Calculate the volume to remove for the silica gel treatment:

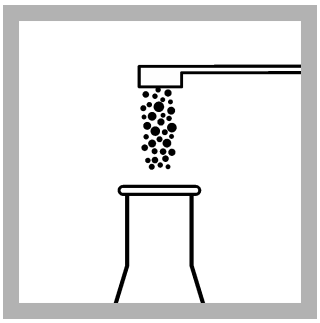
V_{1000} = 100,000 ÷ HEM value

V_{1000} = volume that contains 1000 mg HEM



7. Remove the amount calculated in step 6 from the volumetric flask and add it to a 125-mL Erlenmeyer flask.

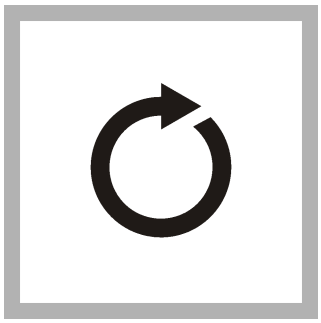
8. Add n-hexane to approximately the 100-mL mark.



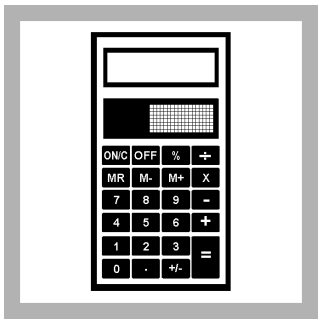
9. Add 30 g of silica gel to the flask. Mix well.

If the volume added in step 7 contained less than 1000 mg HEM, calculate the amount of silica gel to add based on the mg HEM that was added to the flask.

(3 × mg HEM in alliquot) ÷ 100 = silica gel (g)



10. Complete steps 6–15 in Test procedure—SGT-HEM (< 1000 mg/L) on page 7.



11. Correct the test result for the reduced volume that was treated with the silica gel:

$W_d \times (100 \div V_{1000}) = W_c$

Where:

W_d = result from step 15.

V_{1000} = volume removed for silica gel treatment

W_c = corrected SGT-HEM result

Interferences

For samples that are known to contain extremely high levels of oil and grease use a smaller sample volume. Correct the volume difference to give the result as a 1-L sample.

High concentrations of particulates in the water sample can clog the SPE filter or keep high levels of water, which can lower the extraction efficiency. Inorganic particulates are easier to filter than organic particulates. The techniques that follow can help to filter samples that contains high levels of particulates:

- Decanting—Let the sample to settle and pour the top portion into the funnel first. Just before dryness, add the rest of the sample. Remove all possible sediment from the bottle and add it to the SPE filter. Use deionized water to rinse any sediment that remains in the bottle.
- Prefilters or prefilter fibers—Put the prefilter or prefilter fibers into the coupler before the funnel is attached.
- Drying agents—Add magnesium sulfate to the SPE filter or to the prefilter to remove water from the particulates.

- Filtration aids—Add materials such as sodium chloride, sand, diatomaceous earth or glass beads to help speed the complete filtration of samples that contain organic particulates.

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
- 15.0-mL Class A volumetric pipet

1. Put 200 (± 2) mg stearic acid and 200 (± 2) mg hexadecane into a 100-mL volumetric flask.
2. Pour 75–85 mL of acetone and shake vigorously until all material has dissolved.
3. Let the solution temperature decrease to room temperature. Fill to volume with acetone. Mix well. The concentration of this stock solution is 4000 mg/L HEM (2000 mg/L SGT-HEM).
4. Use a volumetric pipet to dilute the stock solution for use in the minimum detection limit (MDL) and the initial precision and recovery (IPR) measurements. Refer to MDL standard solution on page 10 and IPR (OPR) standard solution on page 10.

MDL standard solution

1. Add 15 mL of the stock solution into a clean 100-mL volumetric flask. Dilute to the mark with acetone. Mix well.
2. Use a pipet to add 10 mL for HEM (or 20 mL for SGT-HEM) into a 1-L volumetric flask.
3. Dilute to the mark with deionized water at pH less than 2. Mix well.
   The concentration of this standard solution is 6 mg/L HEM (or 6 mg/L SGT-HEM)

Complete the procedure seven separate times with a 6 mg/L standard solution. Refer to EPA requirements for MDL and IPR on page 10.

IPR (OPR) standard solution

Add 10 mL of the stock solution into 1 liter of deionized water. Mix well.

The concentration of this solution is 40 mg/L HEM (20 mg/L SGT-HEM).

Note: *To verify the concentration, use a pipet to add 10 mL of the IPR standard solution 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 (± 1) mg.*

Complete the procedure for HEM and SGT-HEM (if necessary) four separate times with a 40 mg/L HEM (20 mg/L SGT-HEM) standard solution. Refer to EPA requirements for MDL and IPR on page 10.

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 20 (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 HEM (20 mg/L SGT-HEM)).*

2. **OPR (Ongoing Precision and Recovery)**: Add 5 mL of the standard (40 mg/L HEM (20 mg/L SGT-HEM)) to a 1-liter sample and complete the test. The permitted limits for recovery are:

   - HEM: 78–114%
   - SGT-HEM: 64–132%

   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 10 mL of the standard and measure the concentration after spiking (A).

   Calculate the Percent Recovery (P) as follows:

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

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

   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).

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

   If the RPD for HEM is ≤ 18 and for SGT-HEM ≤34, 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 ± 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 20 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, for Organic Residue Analysis	100–200 mL	1 L	2510253
Methanol, ACS grade	10 mL	500 mL	1446449
Silica gel, 100–200 mesh (for SGT-HEM)	1–30 g	500 g	2665034

Required apparatus

Description	Quantity/test	Unit	Item no.
Balance, analytical, 115 VAC	1	each	2936801
Bottle, wash, 500-mL Teflon, FEP	1	each	2505100
Bottle, wash, 500-mL	1	3/pkg	2927204
Bottle, wide-mouth, 1-L	1	each	2951401
Clamp, swivel	2	each	2503400

Required apparatus (continued)

Description	Quantity/test	Unit	Item no.
Cylinder, graduated, 1-L	1	each	108153
Desiccator	1	each	2088800
Desiccator plate, ceramic	1	each	1428400
Flask, Erlenmeyer, 125-mL	2	each	50543
Hot plate (Thermolyne), 120 VAC, 50 Hz	1	each	2344100
Marker, laboratory	1	each	2092000
Oven, drying, 120 VAC	1	each	1428900
Pump, vacuum, 27 in. Hg, 1.3 CFM	1	each	2824800
SPE Consumables Kit	1	24/pkg	2943800
SPE Starter Kit, EPA Method 1664A	1	each	2943231
SPE Solvent Recovery Kit	1	each	2514300
Stirrer, magnetic, 120 VAC	1	each	2343600
Stir bar, 22.2 x 7.9 mm	1	each	2095350
Support stand	1	each	2504900
Tongs, crucible, 9-inch	1	each	56900

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, for Organic Residue Analysis	1 L	2510153
Flask, volumetric, Class A, 100-mL	each	1457442
Flask, volumetric, Class A, 1-L	each	1457453
pH paper, 0–14 pH units	100/pkg	2601300
Pipet filler, safety bulb	each	1465100
Pipet, serological, 10-mL	each	53238
Pipet, volumetric, Class A, 10 mL	each	1451538
Pipet, volumetric, Class A, 15 mL	each	1451539
Silica gel with indicator (for desiccator)	454 g	1426901

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