



## Understanding Oil and Grease/Hexane Extractable Material (O&G or HEM)

**Introduction** – Oil and Grease (O&G) is the most method defined of all method defined parameters. For reasons that we will explore later, a better name for this particular test would be HEM (Hexane Extractable Material). While it is somewhat easy to say “oil and grease” and have a good idea of what is meant, it is quite difficult to come up with a good scientific definition and even harder to design a selective test. Depending on the specific origin of the material they can foul up water treatment processes, impart unpleasant odors or tastes to the water, or look unpalatable.

**Approved Methods** – There are only two, EPA 1664 and SM5520 B/F.

**Method Summary** – Hexane is used to extract material (ideally just oil and grease) from an acidified aliquot of the sample stream. The hexane is evaporated and the entire amount of residue left behind is defined as oil and grease.

**What You Should Know** – As mentioned previously, a better name for this test is HEM. This is because, while the common perception of oil and grease will be recovered by this method, other materials which are not oil or grease will also be detected. To make matters worse, some materials that you would think should be included are not. Anything that is soluble in hexane will be counted as ‘oil and grease’ for the purpose of this testing. This includes some dyes, sulfur, and substituted hydrocarbons but does not include some heavier petroleum residuals.

There are two main components of oil and grease, petroleum based hydrocarbons (now referred to as ‘non-polar materials’) and fatty compounds of animal or vegetable origin. They can either be determined as a whole or as just the non-polar material, with the animal/vegetable material being the difference between the two. The differentiation between the two is done by adding an extra step involving silica gel. Once again, these two categories are purely method defined. Anything left over after silica gel treatment is a non-polar material.

Method 1664 specifically deals with a liquid-liquid extraction procedure. This involves transferring your sample into large separatory funnels and adding 30 mL of hexane. The sample and solvent are agitated and then allowed to separate. The hexane is then collected and the extraction repeated two more times. Because hexane is lighter than water, the entire sample volume must be drained from the funnel, usually back into the sample container, before the hexane can be collected. Care must be taken to carefully vent during the agitation to avoid potentially dangerous build up of pressure from the fumes. Often during the agitation process an emulsion will form between the organic and aqueous layers. This can cause significant delays in the extraction process. The method does allow alternative extraction methods provided they adhere to the method definition. Solid phase extraction (SPE) is the most widely known and used of these alternatives. SPE is accomplished by passing the sample through a material which retains the HEM components. The HEM is then extracted by rinsing with solvent to carry the HEM into a collection dish or flask. This method has the advantage of reduced solvent usage (often more than 50% less), no dangerous pressure build up, and avoiding the formation of emulsions.

Because this is a method defined parameter there are certain requirements to which one must adhere in order to stay within that definition. The most important one is the solvent of choice. Whether you choose liquid-liquid, Soxhlet, or some sort of SPE method you must use n-hexane and

**ENVIRONMENTAL EXPRESS**

Call 800.343.5319 or 843.881.6560 • [www.environmentalexpress.com](http://www.environmentalexpress.com)





## Lab Matters

David Smith  
Environmental Express  
Technical Specialist

only n-hexane as the active solvent. There are certain *Standard Methods* that are not allowed because they use a mix of solvents in the procedure. If using solid phase discs that require activation (typically with methanol) before the sample is filtered, all traces of that solvent must be removed before the sample is eluted from the disc. Another crucial step is the evaporation temperature. *Standard Methods* recommends 85 °C while 1664A says  $70 \pm 2$  °C. 1664B simply says to adjust the temperature as appropriate. The lower temperature lends itself to better standard recovery as you have less of a chance of volatilizing some of the lighter weight organics. Still lower evaporation temperatures would allow for losing less material to volatilization at the expense of longer evaporating times.

When performing an analysis for HEM you must always use whole volume samples. Because the HEM constituents are typically not water soluble, the sample is not homogenous with respect to the analyte of interest. Almost all components of HEM will be found either on the surface of the sample or adhering to the walls of the container. Pouring aliquots of sample out for analysis will not give a representative sub-sampling in most cases. One final step to keep in mind is to always acidify your blanks and other standards. Failure to do so will result in incomplete recovery of your standards.



## Method Procedure

**Note** – This is not intended to be a standalone method and does not address all safety or quality control aspects that may be required. Please consult your local regulations to comply with all requirements.

This section will only focus on the use of solid phase extraction as I do not wish to encourage anyone to use the colossal time sink that is liquid-liquid extraction.

### Solid Phase Extraction StepSaver

1. Collect your sample in a [1 liter glass jar](#) and preserve with [hydrochloric acid](#).
2. Preweigh your collection flasks and label them appropriately.
3. Assemble your [StepSaver system](#) with the appropriate number of glass or disposable funnels.
4. Follow the StepSaver directions carefully to prep your [solid phase filters](#) with [methanol](#) followed by reagent water.
5. Mark or weigh your sample bottles (for later sample volume determination) and [vacuum filter](#) with the stopcock directed to [waste](#).
6. Redirect the stopcock to the collection flask and elute the filter discs with [n-Hexane](#) through [sodium sulfate cartridges](#).
7. Place collection flasks in the [QuikVap](#) and follow the instructions to evaporate the hexane.
8. Cool to room temperature in a [desiccator](#).
9. Weigh the flask on a [balance](#). Repeat steps 7-9 until successive weighings show a weight change of less than 0.5 mg or 4%, whichever is the smaller quantity.
10. Determine your sample volume by filling the collection jar to the marked line and measure the volume in a 1000 mL graduated cylinder, or weigh the empty jar and assume a density of 1.00 g/cm<sup>3</sup> to calculate the volume of sample.
11. Calculate HEM with the following formula

$$HEM \left( \frac{mg}{L} \right) = \frac{Mass_{extract} (mg)}{Volume_{sample} (L)}$$

12. If a silica gel treatment is desired to determine petroleum hydrocarbons, calculate the amount of silica gel needed for the total HEM (3 g silica gel/100 mg HEM, always round up to next whole gram, 30 g silica



- gel maximum). Add enough hexane to redissolve the HEM and swirl to mix. Some slight heating may be necessary to dissolve the entire amount of residue.
13. Add the appropriate amount of silica gel and mix for 5 minutes. Filter the mixture through filter paper to trap the silica gel and collect the hexane in a pre-weighed collection flask.
  14. Repeat steps 7-9 to obtain the SGT residue. Use the formula in step 11, substituting the SGT residue for the mass of the extract.

## SPE-XPress

1. Collect your sample in a [1 liter glass jar](#) and preserve with [hydrochloric acid](#).
2. Preweigh your [drying pans](#) and label them appropriately.
3. Verify appropriate volume of [n-Hexane](#), [methanol](#), and reagent water are in the reagent caddy. This is automatically monitored if using the Smart Bottle Rack.
4. Mark or weigh your sample bottles (for later sample volume determination) and follow the instructions in the [SPE-XPress](#) to initiate a test run. Consumables at this stage are [filter assemblies](#) and [sodium sulfate cartridges](#).
5. Once evaporation is complete weigh the pan on a [balance](#).
6. Calculate HEM with the following formula.

$$HEM \left( \frac{mg}{L} \right) = \frac{Mass_{extract} (mg)}{Volume_{sample} (L)}$$

7. If a silica gel treatment is desired to determine petroleum hydrocarbons, calculate the amount of silica gel needed for the total HEM (3 g silica gel/100 mg HEM, always round up to next whole gram, 30 g silica gel maximum). Add enough hexane to redissolve the HEM and swirl to mix. Some slight heating may be necessary to dissolve the entire amount of residue.
8. Add the appropriate amount of silica gel and mix for 5 minutes. Filter the mixture through filter paper to trap the silica gel and collect the hexane in a pre-weighed collection flask or drying pan.
9. Use the manual control options on the SPE-XPress to control the heating elements and evaporate the n-Hexane.
10. Weigh the drying pan to obtain the mass of the SGT-HEM.



## Lab Matters

David Smith  
Environmental Express  
Technical Specialist

11. Use the formula in step 6, substituting the SGT residue for the mass of the extract.

Don't forget your [standards](#) for use in either method.

*We all like things that make life easier. Was this document helpful? Or do you...disagree with something?*

*Have something to add? Contact me at [DavidS@envexp.com](mailto:DavidS@envexp.com) to let me know what you think.*