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# elements

## the new protocol

for contaminated sites in Ontario cites the use of methanol precharged vials as a best-practice for soil sampling for volatile parameters after a round robin study showed that laboratories can use the methanol procedure and still obtain detection limits suitable for the regulatory limits. The methanol procedure is deemed the best way to analyze VOC compounds because of its ability to minimize volatile losses, and its precision and applicability to many soil types. Sample hold time also increases drastically from 48 hours for samples collected in hermetic containers to 14 days using the methanol procedure.

## Best-Practices for Soil Sampling for Volatile Parameters



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Improper sampling techniques for volatile organics can lead to seriously compromised test results. Volatile parameters are by their very nature volatile – meaning they vaporize easily. Significant vapor losses can occur during both field sampling and in sample transfer steps that occur in the lab if adequate care is not taken. It has been estimated that the losses of volatile organic compounds can be as high as 90% by the time the samples come to the laboratory for some samples if they are merely collected in glass containers with Teflon-lined septa. For this reason, a better approach is needed.

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## sampling options:

The EPA Method (5035A) offers two alternative solutions to address this. One is an option to use in cases where low levels of volatiles are expected, the other in high level cases. Both involve forgoing the use of a soil jar and instead dropping a soil plug directly into a precharged 40 mL vial.



## low-level approach:

In the low level approach, the soil plug is placed in a 40 mL vial containing 5 mL of sodium bisulphate and a metal Teflon-coated stir bar. The solution preserves the sample until it is analyzed. Analysis is performed directly on the vial when water is added to the sample vial through the septa and the solution is purged to strip away the volatiles for analysis. This is an excellent method and it provides low detection limits because all the compounds in the sample are analyzed. However, one very serious limitation found with the low-level sampling kits is that the entire sample is used in the analysis and if the levels of volatiles are too high (out of calibration range) a second analysis must be performed. This means either resampling in the field, or using soil collected from a soil jar (which accompanies the vials and is provided for the required moisture calculation) which implies vapor losses will occur. In addition, this second analysis must be performed within 48 hours of sampling.

## high-level approach:

The high level method involves the use of methanol, whereby 2 soil plugs are dropped into a 40 mL vial precharged with 5 mL of methanol. Once the volatile organic compounds are in contact with the methanol they are quickly solubilized and isolated from the sample. Methanol is capable of readily solubilizing large quantities of volatile organic compounds and quantitatively extracting the compounds from the soil. At the lab, a portion of the methanol is analyzed. For this reason only a fraction of the compounds from the sample are analyzed and the detection limits are higher. Advantages of the methanol procedure include the ability to dilute the sample and use a smaller amount of methanol for the analysis, as well as the longer sample hold time allowed.





## Testmark's Volatile Organic Soil Sampling Kit

In light of the preceding discussion, Testmark recommends the use of our Volatile Organic Soil Sampling Kit for clients wishing to test for F1, BTEX and/or VOC compounds. All tests can be run from one vial. In cases where very low levels are needed, vials containing sodium bisulphate should be used. Table 1 criteria under the Brownfields Regulation can be readily met with the methanol method.

### benefits:

Once the soil plugs are placed in the pre-charged vial, the vial is never opened again to handle the soil sample. Once in the laboratory, a portion of the methanol extract containing the volatiles is removed using a syringe by making a small injection through the septum. This means no vapor loss through sample handling at the lab. The 120mL soil jar also provided in the kit is used simply to determine the moisture content of the sample which is needed to calculate the final results. Kits are available free of charge with the cost of the test.



## Department of Fisheries and Oceans (DFO) Release Updated Species at Risk Maps

DFO has just released updated distribution maps for fish and mussel species at risk in Ontario (within Conservation jurisdiction areas) which may be a useful resource when assessing sites under Ontario's Species at Risk Act. Maps are posted at [www.conservationontario.ca/projects/DFO.html](http://www.conservationontario.ca/projects/DFO.html) and also include critical habitat extents as well as GIS information and color-coded stream segments. DFO has also announced that they are continuing to work at expanding these maps to all areas of Ontario.



### contents of kit:

The kit consists of a disposable Terra Core™ Sampler, two 40mL methanol-charged, pre-weighed vials (second vial is provided simply for backup as a cautionary measure) and a 120mL amber glass jar (to provide soil for a corresponding moisture calculation which is required in volatile analysis).

### sampling procedure:

- Take 2 soil plugs using the Terra Core™ Sampler
- Carefully drop each plug into the methanol preserved 40mL vial
- Ensure no soil is present on the bottle rim
- Quickly place the lid on the vial
- Repeat for second 40mL vial (backup vial)
- Fill 120mL soil jar with soil and close
- Send vials and jar to lab for analysis

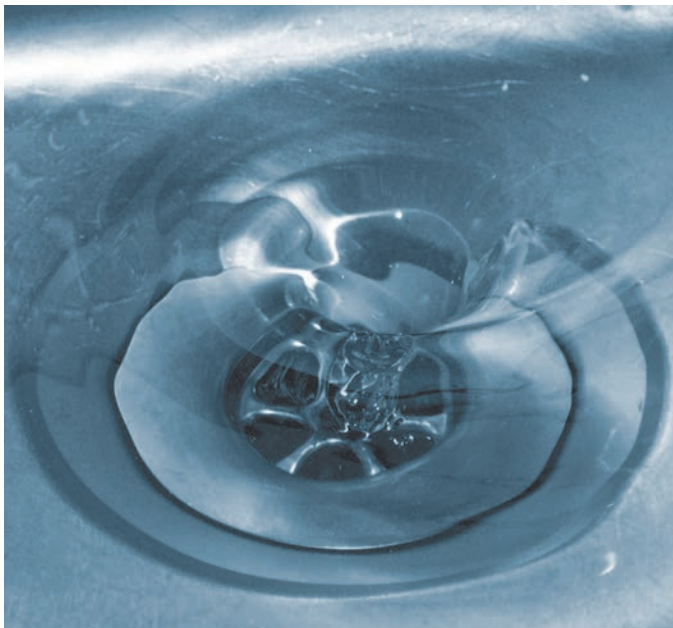


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# The Analysis of Oil and Grease in Water Samples

Oil and grease analysis is often cited as an item of interest in “greywater” studies (investigations into non-sewage effluent from domestic or industrial sources), for example in investigations under Ontario’s Provincial Water Quality Objectives and as hard daily and monthly load limits in many MISA-based operations. Oil and grease as a test includes not only petroleum oils, but also natural vegetable, mineral and animal based oils such as those found in vegetables, humic soils and animal fat. Sources are therefore both anthropogenic (wastewater, oil spills, manufacturing, automotive emissions) as well as natural.



## analytical methodology:

The standard EPA method for oil and grease analysis is Method 1664 Revision A, which outlines the quantitative determination of Hexane Extractable Material (HEM), as well as Silica Gel-Treated Hexane Extractable Material (SGT-HEM) in water. The distinction between the HEM oil and grease and SGT-HEM oil and grease is related to the procedure used to generate the values and to the fact that HEM measures both relatively polar and non-polar material including non-volatile petroleum hydrocarbons, waxes, animal fats, mineral and vegetable oils, soaps, sulfur compounds, organic dyes, chlorophyll and some phenols, where SGT-HEM measures only non-polar oil and grease compounds such as mineral oil.

HEM is simply the weight of material per unit volume extracted with hexane solvent after the solvent isolated from the water and the solvent has evaporated. Prior to the extraction of the sample however, the pH of the solution is adjusted to less than 2. Once the pH is adjusted, compounds that would not normally be extracted from a neutral solution are indeed extracted. This is because compounds with carboxylic acids, like those found in soap, are rendered neutral and can be released from the solution and will be extracted with the hexane used to perform the extraction.

In many cases, it is important to know if the HEM value is due to polar compounds or due to non-polar compounds like those found in mineral oil. The SGT-HEM method will accomplish this. In this method, the material collected from the





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HEM analysis is redissolved in hexane and passed through a silica gel column. The polar compounds are bound to the silica gel through the hydroxyl groups on the silica gel and the non-polar compounds like mineral oil pass through the column and are collected. For a second time, the hexane is evaporated and the weight of the material is determined. Both HEM and SGT-HEM procedures involve evaporation of the solvent. This step however does allow for some extracted compounds to be lost due to evaporation. The majority of gasoline extracted in this procedure is normally lost due to evaporation. For this reason, care must be used in determining the interdependence of analytical data when multiple analytical techniques are used for investigation.

Testmark offers both HEM (commonly referred to as Total Oil & Grease or TOG) and non-polar oil and grease tests often referred to as SGT-HEM.

A common MDL for both methods is 2 mg/L as this is fit for purpose for most environmental applications. To put this in perspective, most EPA and CCME criteria point to a concentration of 10 mg/L of oil and grease as being a limit to consider for the protection of aquatic life - the concentration at which you will notice a sheen on the surface of the water.

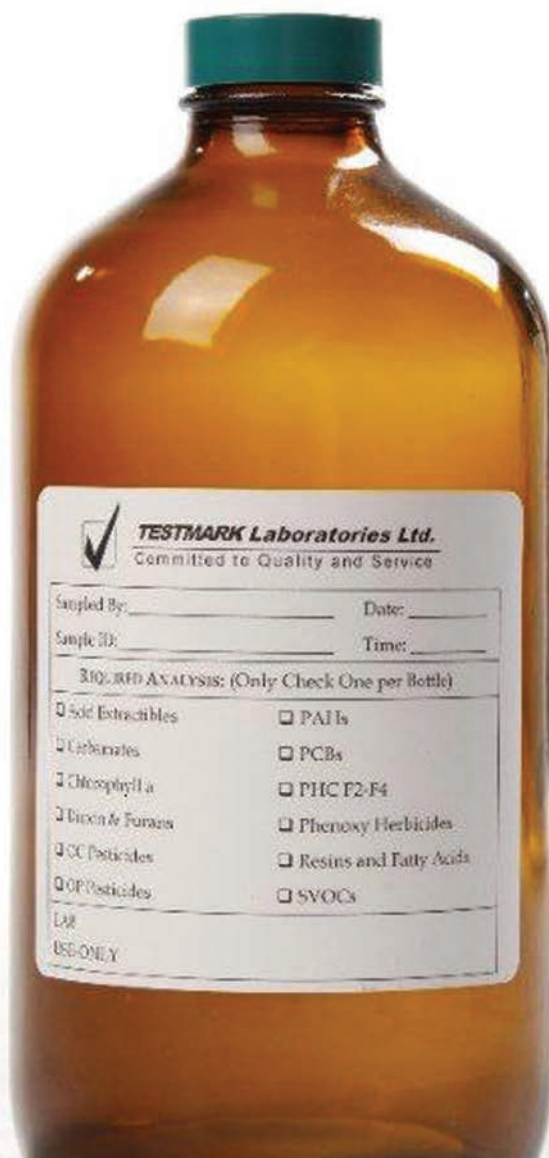
## sampling considerations:

The standard sampling protocol for oil and grease is to use a 1L amber, glass container and preserve with Hydrochloric acid (HCl). Most compounds extracted with hexane won't readily adhere to the sides of the container unless the concentration of the compounds is high. The determination of Oil and Grease involves a procedure known as whole bottle analysis. In whole bottle analysis procedures, the bottle is rinsed with hexane to ensure that any compounds adhered to the bottle interior are isolated in the solvent. The amber glass along with the acid helps deter microbiological growth from developing in the sample and altering the chemistry of the molecules collected and perhaps altering the

values obtained. The standard hold time is 7 days if unpreserved and 28 days if preserved.

Because the majority of oil and grease products and molecules are non-polar and light, they tend to rise to the surface of a water body and often appear as a sheen. Therefore proper sampling technique should account for this by skimming a representative sample along the surface of the water such that the collected sample includes both surface and sub-surface water. Oil and grease compounds can be mechanically or chemically emulsified and these may not migrate readily to the surface due to their small size (<1 micron for chemically emulsified oil) therefore the application

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of the sampling site must be considered by the Qualified Professional. In most cases, it is advisable to review the analytical data collected at a specific sampling point to determine the best practice for collecting samples.

## environmental significance of oil and grease measurements:

Depending on the composition of the oil and grease, at the one end of the spectrum water bodies with high loads of oil and grease with more petroleum-based components can experience high mortality of aquatic and benthic organisms as the oil and grease coats surfaces and creates asphyxiating conditions. Data originating from this type of contamination will often show both appreciable SGT-HEM and HEM values. Oils of animal or vegetable origin however are generally chemically nontoxic to humans and aquatic life but are often targets for aesthetic reasons.

Although it may be associated with petroleum hydrocarbons, it is important to note that this parameter does not correlate well with levels of petroleum hydrocarbons when analyzed through scatter plots and should not be used for this purpose. Likewise, technical studies have noted that oil and grease is an inappropriate measure for considering overall ecological effects (in terms of hazard, toxicity or risk), but it has been found to have some indirect value as a measure of oxygen demand in biological systems.



## need more information?

Call our Customer Service Team  
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