Sampling and Analysis for Volatiles in Soil Using EPA Method 5035

The procedure for sampling and analysis of soils for volatile organic compounds by GC/MS EPA 8260, TPH GRO by EPA 8015, and BTEX, MTBE & naphthalene by GC EPA 8021 was updated in June, 1997 to Method 5035. This update eliminated Method 5030 (using a soil packed jar) for soil volatile organic analyses because studies had shown that the results for many target analytes were biased low. To minimize this bias, the USEPA developed Method 5035, incorporating changes that greatly reduced the handling of soil samples in open air in the field by samplers and by analysts.

Method 5035 gives a number of options for collecting soil samples – Terra Core kits and EnCore® Sampling Devices are the most commonly used options.

Use of Terra Core Soil Sampler® Sampling Device


The Terra Core Soil Sampler is used for soil sampling for low and high level analyses. The sample is collected using the syringe like coring device pictured here and then extruded into pre-preserved vials. Each kit consists of a foam block containing a total of three vials and a 2 oz plastic jar in addition to the coring device. The 2 oz jar is to be filled for laboratory use for percent solids analysis to allow the results to be reported on a dry weight basis. Two to four vials with sodium bisulfate will be filled for low level analysis and the third vial with methanol for a high level analysis.

Click on the link to find a video demonstrating the use of the Terra Core Sampling kit [link to video]

Low level samples: The two to four vials that are preserved with sodium bisulfate sulfuric acid are used for low level samples. Having two vials with sodium bisulfate enables the laboratory to perform the low level analysis twice in situations where a reanalysis is required.

The low level analysis technique utilizes a closed system purge and trap procedure. In the low level procedure, the volatiles are liberated from the soil by agitation and purging with an inert gas into the "headspace" or gaseous layer in the vial. A special instrument is utilized that punctures the septum on the vial, and extracts an aliquot of the gaseous layer above the sample. Therefore, only one analysis can be performed on each vial. For this reason, a second vial must be collected in case reanalysis is necessary.

Caution must be exercised with carbonate mineral soils as they may react with the acid preservative creating gas bubbles. The gas formation could cause the container to explode after capping. The easiest way to prevent this situation is to test each soil type collected with sulfuric acid to determine if they effervesce. If introduction of the sample into the sodium bisulfate preserved vial results in effervescence, vials with organic free reagent water should be used instead.
Because the vials are not opened again after the sample is injected, loss of analytes due to volatilization is minimized. In addition, the preservation in the vials minimizes biodegradation. The holding time for the preserved samples is 14 days.

**High level samples:** Additionally, a third vial that preserved with methanol is required in case the sample must be reanalyzed as a high level sample. An aliquot of the methanol containing the contaminants of concern is introduced to the instrument. For high level samples only one vial is required as multiple analyses can be made from that vial.

**Multiple Volatiles Analyses on a sample:** Phase Separation Science keeps two forms of Terra Core kits in stock. If there is only one volatile analysis needed (just GRO for example) then a kit comprised of just three vials (two preserved with sodium bisulfate and one preserved with methanol), the 2 oz plastic jar and the coring device is to be used.

However, if your site requires volatiles by GC/MS and GRO then the lab needs a total of four vials preserved with sodium bisulfate and one vial preserved with methanol along with the 2oz plastic jar. Phase puts together this extended kit for client use which is more cost effective than using two complete Terra Core kits. When you call for bottles, discuss with your laboratory contact what your project requirements are and the appropriate kit can be selected.

There are a couple of very important considerations to observe when using these kits. First, there is a measured amount of a preservative in each vial, if any is lost through spillage the vial is unusable as it will yield inaccurate results. Second, the coring device is calibrated to give you the proper amount of sample as long as the inner plunger remains in the “detent” position. If the inner plunger protrude from the handle, too much sample will be collected and the laboratory will not be able process the sample on the instrument.

**Use of En Core® Sampling Device**


The En Core® Sampling Device can be used for low or high level analysis: Method 5035 also contains an option with a proprietary device called an En Core® sampler. This is a special, one use, EPA approved sampling device that allows the sampler to collect and transport unpreserved samples to the laboratory.

The En Core® sampler is a device made of an inert composite polymer, designed to collect, store and deliver soil in a sealed, headspace-free state. In order to collect the sample, the En Core® sampler is attached to a reusable metal T-handle that serves to assist in pushing the sampler in the soil. An airtight sealing cap is then attached creating a self-contained package as shown in the picture.

Similarly, three En Core® samplers are required for each sample providing the laboratory with two for low level analysis and one for high level analysis. The devices are transported to the laboratory on ice where the cap is opened and the undisturbed sample is immediately placed in the appropriate preservation fluid, methanol or sodium bisulfate. This preservation step must take place within 48 hours of sample collection. The Terra Core sampling kit is therefore
advantageous over the En Core® sampling devices since the samples arrive preserved and with a 14 day holding time. The Terra Core kits are also more cost effective than the En Core® samplers.

Phase does not encourage the use of or stock En Core® sampling devices. We do maintain a supply of Terra Core Sampling kits for use with method 5035.

Note: There are situations where the matrix is not suited for Terra Core kits or En Core® Sampling Devices. If you are sampling sand or gravel or very wet sediments or even a muddy substrate then the best available option is a 4 oz jar for sample collection.

<table>
<thead>
<tr>
<th></th>
<th>Previous 5030</th>
<th>Current 5035</th>
<th>Current 5035</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Low Level</td>
<td>Low Level</td>
<td>High Level</td>
</tr>
<tr>
<td>VOC 8260</td>
<td>5 ug/kg</td>
<td>5 ug/kg</td>
<td>500 ug/kg</td>
</tr>
<tr>
<td>BTEX 8021</td>
<td>1 ug/kg</td>
<td>1 ug/kg</td>
<td>100 ug/kg</td>
</tr>
<tr>
<td>TPH GRO 8015B</td>
<td>100 ug/kg</td>
<td>100 ug/kg</td>
<td>10,000 ug/kg</td>
</tr>
</tbody>
</table>