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Sampling for Volatile Organic Compounds in Soil by EPA Method 5035/5035A

Methods 5035 and the more recent 5035A outline procedures for sample collection and analysis of soils and solid wastes for volatile organic compounds (VOCs). The original method was published in Update III to the Third Edition of SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods." Method 5035A has been released on the EPA Website for SW-846 methods. This procedure is required for analytical methods using purge-and-trap analysis (8021, 8015 and 8260).

Method 5035 lists two preservatives—sodium bisulfate and methanol. Since the method was promulgated (June 13, 1997), the EPA has expanded guidance in Method 5035A and allowed for freezing samples as a substitute for chemical preservation. This 5035A update gives more instruction and options on the preservation of soil samples.

The analysis for VOCs is performed on two ranges: low level (approximately 5-500 µg/kg for GC/MS) and high level (>250 µg/kg for GC/MS and >25 µg/kg for GC/PID).

The choice of low or high level is determined by the requirements of the project. However, since the low-level method is only valid for a certain concentration range, a sample for analysis by the high-level method must also be collected to ensure quantification of all target analytes.

The low-level method uses one or more of these options for the sampling/preservation of soils:

- Soil sampled into a vial with a sodium bisulfate ($NaHSO_4$) solution.
- Soil collected in an EnCore sampler and immediately shipped to the laboratory for further preservation (within 48 hours).
- Soil collected in a vial with water, sealed in the field and shipped to the laboratory immediately in order to meet the method preservation requirement to freeze within 48 hours of collection.

The high-level method uses one of these options for sampling/preservation of soils:

- Soil sampled into a vial with methanol.
- Soil collected in an EnCore sampler and shipped to the laboratory immediately in order to meet the method requirement of preserving in methanol within 48 hours of collection.

The containers (vials or EnCore samplers) must be kept cool (on ice) during shipment. Because $NaHSO_4$ is corrosive and methanol is flammable, packaging for shipment must comply with DOT regulations. Based on project-specific requirements, travel blanks may be requested (see the chart on other side for the appropriate matrix). Please discuss this with your client services representative.

Low-Level Method

Option A - Direct sampling into EnCore samplers

- Three 5-g size EnCore samplers for each sample.
- One nonpreserved container for moisture determination.

Option B - Direct sampling into vial with chemical preservative

- Three 5-g size cores are added to vials (two with sodium bisulfate, one with methanol) for each sample plus a TerraCore or other coring sampler.
- One nonpreserved container for moisture determination.

Option C - Direct sampling into vial with water (or methanol) to be frozen at the laboratory

- Three 5-g size cores are added to vials (two with water, one with methanol) for each sample plus a TerraCore or other coring sampler. Once sealed in the field these are not opened in the laboratory.
- One nonpreserved container for moisture determination.

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High-Level Method

Option D - Direct sampling into EnCore samplers

- One 5-g size EnCore sampler for each sample.
- One nonpreserved container for moisture determination.

Option E - Direct sampling into a methanol-preserved vial

- One methanol-preserved vial (plus coring device) for each sample.
- One nonpreserved container for moisture determination.

Soils that Effervesce with $NaHSO^4$

For the low-level method, the use of $NaHSO^4$ creates problems with soils that contain carbonate. These soils effervesce on contact with the $NaHSO^4$, which interferes with the analysis. In this case, the high-level technique may be used. The lab can freeze the soil from EnCore samplers with lab grade water for analysis within 14 days of collection. Alternatively, only the high-level analysis may be used.

QA/QC

Because only one analysis will come from each container, additional samples must be collected for matrix spike/matrix spike duplicate analyses or other quality control analyses. The number of containers for MS/MSD samples is listed below.

Sample Containers

Analysis Level	Option #	EnCore Sampler	Sodium Bisulfate-Preserved Vial	Sealed Vial with Water "Preservative"	Methanol-Preserved Vial	Unpreserved Container for Moisture	Travel Blank*
Low	A	3 (7**)	—		—	1	1
Low	B		2 (4**)		1 (3**)	1	1
Low	C			2 (4**)	1 (3**)	1	1
High	D	1 (3**)			—	1	1
High	E	—	—		1 (3**)	1	1

*Based on project-specific requirements, travel blank matrix may vary.

**Total number of sampled containers needed for background/MS/MSD analyses.

Quotation Limits

Method	Low Level		High Level	
	LOQ (µg/kg)*	MDL (µg/kg)*	LOQ (µg/kg)*	MDL (µg/kg)
8260B*	5	1-2	250	50-100

*This is the typical LOQ or MDL for most analytes. Some compounds are detected or quantified at greater concentrations. Contact our Client Services Group for more details and for sampling instructions and materials.

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Method Development
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Organic Acids
Aldehydes
1,4-Dioxane (low level)
Low-Level Mercury
PMI
Method 25D

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