



## INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT

**Sampling Soil and Waste for  
Volatile Organic Compounds (VOCs)**

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**Notice**

Mention of trade names or commercial products does not constitute endorsement or recommendation by the IDEM for use. This guidance was formerly titled "Supplemental Guidance for Sampling Soil and Waste Samples for Volatile Organic Compounds (VOCs)".

**Related Guidance**

Method 5035A, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," in *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846, Third Edition, Final Update III; updated method: July 2002. [Method 5035A]

**Scope**

This technical guidance is intended to provide additional information regarding the collection and preservation of volatile organic compounds (VOCs) within soil and solid waste samples to be analyzed at a stationary, off-site laboratory. Pre-sampling planning is emphasized as essential for meeting sampling objectives. Method 5035A contains critical information on sampling and analytical requirements and thus should be consulted alongside this document when developing site-specific sampling plans.

The techniques described in Method 5035A represent a significant departure from those traditionally used in the environmental field for sampling of VOCs in solid materials. Traditional sampling methods have been shown to result in losses of VOCs that are significant to certain project objectives. The Method 5035A procedures are designed to minimize losses of VOCs due to volatilization and biodegradation. This guidance will identify instances where it is critical to have the increased accuracy provided by Method 5035A.

**Applicability**

This supplemental guidance applies to all Office of Land Quality programs that require the laboratory analysis of VOCs in soil or solid waste.

## **General Guidance**

Method 5035A does not provide detailed guidance regarding systematic planning, sampling design, retrieval of sub-surface soil cores, or the actual steps for determining VOCs in the laboratory. These and other aspects of a sampling project should be adjusted as needed to assure losses of sample VOCs are minimized.

Several options for the collection, preservation, and storage of samples for VOC analysis are described in Appendix A of SW-846 Method 5035A. Special considerations for samples to be extracted using the Synthetic Precipitation Leaching Procedure (SPLP) prior to analysis are described below. The necessity for representative sampling demands that sample collection and preservation options are selected based on the requirements of the data quality objectives (DQOs) for the project. The selection of a specific Method 5035A sampling technique for a project is not solely based upon the availability of sample containers, the convenience or simplicity of use, or the lowest price.

To minimize loss of VOCs, sub-surface soils are retrieved and sub-sampled as quickly as possible, taking special care to limit exposure and disaggregation of the soil's physical structure. Once a core barrel or sampling device has been opened, the collection of sub-samples is the first activity performed (i.e., pre-empting even the logging of sub-surface core sample recovery and other soil characteristics). If a second core is obtained before the first core has been logged, the logging of the first core should be set aside until the second core has been sampled. In some cases, this is a change in the order in which work is done. The use of intermediate storage containers (e.g., core barrel liners, plastic bags, large glass jars) to hold soil for extended periods prior to sub-sampling by Method 5035A defeats the project intent and is to be avoided. The field log or boring log should show the time the sub-surface core is retrieved and the time sub-samples are collected.

Sampling personnel should be ready to take sub-samples immediately after the sub-surface core sample is retrieved. If the surface of the core remains exposed while field screening the core to determine the interval for sub-sampling, a fresh surface should be created prior to collecting each sub-sample by Method 5035A. Also, if the surface of a soil core has been exposed for more than a minute or two due to an unforeseen delay, a fresh surface should be created just prior to collecting the sub-sample. Drill rig personnel should adjust their pace accordingly to allow collection of sub-samples.

Sampling techniques and equipment will vary slightly depending on the type of soil (cohesive, non-cohesive, cemented, oily, etc.) or solid waste to be collected. Typically, a disposable coring device such as a Terra Core<sup>®</sup> Sampler is used to collect approximately 5 grams of soil or solid waste, and this sub-sample is immediately extruded into an empty sealed vial and preserved (see "Sample Preservation" below). Additional information regarding this issue may be found in Appendix A, Section 7.1 of SW-846 Method 5035A.

In order to provide the laboratory with enough sample volume to account for potential re-analysis and to meet applicable quality assurance and quality control (QA/QC) requirements, it may be necessary to collect several (typically 3-5) sub-samples from each interval sampled within each sub-surface core. One additional sub-sample is also needed from each core to make a dry weight determination. Sampling personnel should plan accordingly prior to site mobilization to assure that sufficient equipment and containers are available at the time of sampling.

Based on field screening information, sampling personnel may determine that the sub-samples collected from a particular sub-surface core interval or solid waste sampling location do not need to be sent to the laboratory for analysis and thus may be discarded. The time and equipment needed for collecting these discarded sub-samples should be factored into the overall cost for the sampling event. Strategic planning prior to site mobilization may limit the number of discarded sub-samples (and related costs) while still meeting project DQOs and requirements for representative sampling.

### **Project-Specific Sampling Planning Guidance**

IDEM Remediation Closure Guide (RCG) [currently under revision; see reference below] Section 3.2.4 states:

- Use U.S. EPA SW-846 Method 5035A (as updated) to minimize VOC loss, especially when collecting soil *closure* samples for VOC analysis.
- The use of specialized containers and preservation techniques as described in Method 5035A may be unnecessary for samples collected within areas of known or suspected contamination, if the sampling method meets project objectives.
- IDEM will consider alternatives to the procedures and equipment described in Method 5035A and supplemental IDEM guidance on a site-specific basis.

Similarly, traditional sampling methods may be acceptable for some specific projects initialized before release of the IDEM RCG and utilizing the Risk Integrated System of Closure (RISC) [see note under “References” below] as described below.

- Area Screening – When performing default or non-default Area Screening activities for VOCs (as described in the RISC Technical Guide [see reference below], Chapter 3.4.3.1) or an underground storage tank (UST) closure sampling assessment (under 329 IAC 9-6-2.5 and the RISC User’s Guide [see reference below], Chapter 3), use Method 5035A in most cases. If information collected is intended to obtain a “clean closure,” use Method 5035A to ensure representative results. However, if additional investigations and/or corrective actions are intended, Method 5035A may not be needed for area screening.
- Site Characterization – When conducting investigations to determine the nature and extent of VOC soil contamination, use Method 5035A. If the investigation includes areas of known or suspected contamination, Method 5035A may not be needed as long as the variation in methods agrees with project DQOs. If the leaking underground storage tank (LUST) “Step-Out Procedure” is employed (as described in the RISC User’s Guide, Chapter 3.5.1), use Method 5035A when soil samples collected are believed to be below RISC Residential Default Closure Levels for the contaminants of concern (COCs).

- **Corrective Action** – When conducting soil removal during an UST removal closure or soil excavation, use Method 5035A when the information will be used to verify that the source was removed or there was no release and/or source present (in the case of an UST closure). Collect soil from the excavation at locations prescribed by the IDEM program providing oversight. Generally, remove an additional six inches (6”) of soil to expose soil that is representative of the actual conditions immediately before sampling using Method 5035A.  
Note: The above description does not apply to projects in the Resource Conservation and Recovery Act (RCRA) Corrective Action Program.
- **Closure (No-further-action)** – When collecting soil samples to confirm soil results meet closure standards, use Method 5035A for all samples sent to a stationary, off-site laboratory for analysis.

## **Receptors**

If the source is close to receptors or conduits, take special care to ensure representative results by using Method 5035A. Of particular concern are sources near drinking water wells, wellhead protection areas, or ecologically susceptible areas. In these situations, it is necessary to detect and define very low levels of contamination (typically on the periphery of a plume) prior to demonstrating site characterization.

## **Sample Preservation and Holding Time**

Sample holding times ranging from 48 hours to 14 days are described in Method 5035A. Implementation of alternative procedures and/or chemical preservatives to extend the maximum holding time may be allowed in some cases, if it can be demonstrated that the concentrations of target analytes in the samples will not be significantly affected.

Common chemical preservatives such as methanol and sodium bisulfate may have effects (dilution or reaction with contaminants of concern, raised detection limits above set action levels, false positives and negatives, etc.) on the samples and thus may not be appropriate for some projects. Sample collection, preservation, and storage options should be selected based on the requirements of the DQOs for the project. Additional information regarding this issue may be found in Appendix A of SW-846 Method 5035A.

## **Synthetic Precipitation Leaching Procedure (SPLP, SW-846 Method 1312)**

Section 9.10 of the RCG allows the use of the Synthetic Precipitation Leaching Procedure (SPLP), SW-846 Method 1312 [Method 1312] as one alternative to calculating site-specific migration to ground water levels. Method 1312 was released before Method 5035A, and Method 1312 has not been revised to include Method 5035A procedures to minimize loss of VOCs during sample collection and handling prior to extraction. The following items should be addressed when soil and solid waste samples are collected using Method 5035A and extracted using Method 1312 prior to analysis for VOCs:

- **Sampling Device/Container** – Obtain samples using an appropriate coring device (such as a 25-gram EnCore® type sampler) that is used for collection, transport, and

storage of the samples with minimal loss of VOCs. Method 1312 typically requires approximately 25 grams of sample in order to prevent headspace in the Zero Headspace Extraction (ZHE) vessel used by the laboratory. The volume of soil (5 grams) obtained using some Method 5035A coring devices is insufficient. Likewise, the 40 mL glass vial container options listed in Appendix A of Method 5035A may not be acceptable due to potential loss of VOCs during transfer of the sample from the container to the ZHE vessel.

- Transport, Preservation, and Holding Time – Transport samples on wet ice at 4 degrees C and deliver samples to the laboratory within 48 hours of collection. Chemical preservation is not permitted prior to SPLP extraction; however, samples received within 48 hours of collection may be frozen on arrival at the laboratory to extend the holding time to 14 days from the date of collection to date of extraction. Frozen samples are then maintained at -10 degrees C and the laboratory documents that the samples were frozen. Once a sample is thawed (to 4 degrees C) the SPLP extraction is performed as soon as possible. After the SPLP extraction has been completed, the holding time for analysis of the leachate is seven days if unpreserved and 14 days if acid preserved by the laboratory performing the SPLP extraction.
- Particle Size Reduction – Section 7.3.6 of Method 1312 describes a procedure for reducing the size of the particles of the sample prior to transferring the sample to the ZHE vessel. Any grinding, cutting, or crushing of the sample prior to extraction may cause loss of VOCs due to volatilization and should be avoided. Samples should be transferred directly from the collection/transport device to the ZHE vessel with as little disturbance as possible.
- Detection Limits – SPLP is not a part of the analytical procedure, and as such is not included in the determination of detection limits. The laboratory maintains the ratio of extraction fluid to sample volume/weight during the SPLP extraction so that established detection limits for the VOC analysis may be maintained. Typically, a 20:1 ratio (500 mL fluid to 25 g sample) is employed.
- Laboratory Equipment and Procedures – When planning for sampling, confirm that the laboratory has the required extraction equipment (ZHE device) available and can document the procedures described in Method 1312.

### **Further Information**

If you have any additional information regarding this technology or any questions about the evaluation, please contact Office of Land Quality, Science Services Branch at (317) 233-6593. This technical guidance document will be updated as new information is acquired.

## **References**

“Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds,” ASTM Standard D 4547-06.

“Standard Practice for Collection and Handling of Soils Obtained in Core Barrel Samplers for Environmental Investigations,” ASTM Standard D 6640-01(2005).

*Note that historical versions ASTM D 4547-06 and ASTM D 6640-01(2005) were consulted for the original (2008) version of this technical guidance document. Updated versions (currently 2015) of the two ASTM Standards are available at:*

*<https://www.astm.org/>.*

“Remediation Closure Guide,” Indiana Department of Environmental Management, March 22, 2012, and updates. [RCG]

*Note that the RCG is currently under revision. References to “RCG” in this technical guidance document will be updated as needed when the revised RCG is final.*

“Risk Integrated System of Closure: Technical Resource Guidance Document,” Indiana Department of Environmental Management, February 15, 2001, and updates. [RISC Technical Guide]

“Risk Integrated System of Closure: User’s Guide,” Indiana Department of Environmental Management, February 15, 2001, and updates. [RISC User’s Guide]

*Note that technical information that appears in the RISC Technical Guide and RISC User’s Guide has been superseded by newer guidance. However, some responsible parties may be eligible to use superseded guidance when addressing releases.*

Method 1312, “Synthetic Precipitation Leaching Procedure” in *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846, Third Edition, Final Update II, September 1994.

Method 3815, “Screening Solid Samples for Volatile Organics” in *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846, Third Edition, Final Update IV; February 2007.