

**Arizona Department of Environmental Quality UST Program
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APPENDIX E INVESTIGATIVE SUBSURFACE SOIL SAMPLING

The UST Program recommends that soil samples be collected using appropriate soil boring methodologies . Soils from excavations and from drill cuttings are disturbed soils and, therefore, analytical results for volatile chemicals of concern are not likely to be representative . The following sampling guidelines apply to all soil samples collected below ground surface to determine the extent of contamination during site characterizations. All soil borings should be abandoned in accordance with ADWR guidelines or ASTM Standard D 5299-92.

- C To describe consolidated and unconsolidated geologic materials hereby referred to as lithology [American Society for Testing and Materials Publication D2488, “Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)”, most recent publication date], soil samples should be collected every five to ten feet during drilling or at significant changes in lithology. The collection of soil samples beneath saturated conditions or within the capillary fringe during drilling activities is NOT recommended for chemical analytical purposes, but may be appropriate for lithologic or stratigraphic classification associated with groundwater investigation. When drilling through contaminated soil, special care should be taken to prevent the introduction of contaminated soils into groundwater and the resulting inadvertent contamination of groundwater (i.e., creation of false positives). Special care should also be taken to ensure that investigators do not unknowingly drill through an aquitard and risk cross-contaminating vertically separated aquifers, in violation of the ADWR requirements [AAC R12-15-812(B)].

- C To determine the vertical extent and magnitude of contamination, soil samples for laboratory analysis should be collected at least every ten feet of drilling, but usually not more than every five feet within the contaminated zone unless a significant change in lithology has occurred. Other samples should be submitted for analysis, based on professional judgement, if additional site information is necessary to design appropriate remedial systems or to determine potential exposure pathways.

- C Samples should be collected from undisturbed soils by pushing or driving a clean split-spoon type sampler lined with clean sleeves composed of an inert material such as Teflon, stainless steel, or brass. Sample collection should be conducted in accordance with industry standards and available ADEQ guidance.

- C If a drilling method is used which produces a soil core, then samples should be collected by pushing a sleeve into the core immediately after the core is brought to the surface. If the core is too hot to handle, then it is inappropriate to test the soil for VOCs.

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- C Soil samples collected from auger cuttings or from the cyclone of air-method drilling rigs are not acceptable for submittal for laboratory analysis.

- C Completely filled sleeves should be immediately sealed by completely covering the ends with a Teflon patch, covering the Teflon patch with a foil patch, covering the patches with tight-fitting plastic caps, and sealing the caps by wrapping custody seals or non-contaminating tape around the sleeve, overlapping the lower edge of the cap. Alternative sampling methods may be used if approved by the ADEQ. The sample should be submitted to a fixed-base or mobile analytical laboratory certified by the ADHS to perform the appropriate analysis. A properly completed chain-of-custody document should accompany all samples.

- C The soil sample preservation and extraction for VOCs should be conducted in accordance with an ADHS approved method . If the owner/operator so chooses, extraction and preservation may be conducted in accordance with the ADEQ 5035 Policy [0170.000] attached with this appendix. This policy must be followed by state contractors performing corrective actions under the UST Program State Lead Unit. Otherwise, samples should be preserved and extracted as soon as possible, but no later than 48 hours after sample collection.

- C The sample should be immediately labeled, immediately placed in a sealable plastic bag, and immediately stored in a cooler on ice. Blue ice, *i.e.*, sealed artificial coolant, should not be used unless required for shipping purposes.

- C Field measurements and the lithologic descriptions should be completed using the remainder of the recovered sample.

- C Composite soil samples are not acceptable for investigation or remediation confirmation. However, these samples are acceptable for stockpiled soil.

- C UST chemicals of concern and discussion of analytical methods are provided in Sections 4 and 6 of this guidance document.

- All samples must be representative, and data quality preserved to the extent practicable. In meeting the data quality objectives for sampling and analyses conducted during site investigation, remediation and compliance , additional information is available in publications issued by ASTM and the U.S. EPA.

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**0170.000 IMPLEMENTATION OF EPA METHOD 5035 - SOIL PREPARATION
FOR EPA METHODS 8015B, 8021B AND 8260B.**

LEVEL TWO Arizona Department of Environmental Quality

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I. PURPOSE

The EPA Office of Solid Waste promulgated Method 5035, Closed-**System Purge-and-Trap Extraction for Volatile Organics in Soil and Waste Samples** (Attachment 1), in June 1997 in SW-846, Update III. The Arizona Department of Health Services (ADHS) Office of Laboratory Licensure, Certification and Training adopted Method 5035 in May 1998 and Method 5035 became enforceable on March 1, 1999 in Arizona. The collection and analytical procedures for the approved method are flexible and, without further guidance, could result in multiple interpretations.

This policy establishes the sampling options and the preservation holding time requirements for individual programs within the ADEQ's Waste Programs Division. This policy is necessary to provide an understanding of the options set forth by the method and the limitations imposed on specific field sampling requirements. This policy does not eliminate the need to read and understand EPA Method 5035. The method, in conjunction with this policy, will provide a technically defensible and consistent approach to sampling for Volatile Organic Compounds (VOCs) in soils.

II DEFINITIONS: (FOR PURPOSES OF EPA METHOD 5035 ONLY).

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1. Sample Preservation: The addition of methanol or sodium bisulfate to an unpreserved sample in the field or in the laboratory.
2. Sample Extraction: The addition of methanol to an unpreserved sample in the laboratory. After extraction, the methanol is transferred to a vial and can be stored at 4°C(± 2°C) until analysis.
3. Hermetically Sealed: For the purposes of this policy a hermetically sealed container shall be defined as a sample storage device that consistently shows less than 10% loss from volatilization over the intended storage holding time (usually 14 days) or a minimum of 48 hours for the compounds of concern at a given site.
4. Sample Freezing: A preservation technique in which the sample is frozen and stored at 0°C (32°F), or lower upon receipt at the laboratory. Blue ice is unacceptable.
5. Calcareous Soil: A soil whose content of carbonate is sufficient to cause effervescence when tested with hydrochloric acid. (Reference: Bates R. L. and Jackson J. A.. (1987). Glossary of Geology. (3rd ed.)Alexandria: American Geological Institute.)

III POLICY

Method 5035 is structured as a 2-tier approach for low and high concentration sampling¹. Preservation is recommended for both low and high contaminant concentrations as stated in the Method. Based upon program requirements, preservation can be conducted in the field or subsampled in an EnCore™ Sampler and the sample preserved in accordance with sample handling.

A. Sample collection options for low reporting limits (<200 µg/kg)

- i. Methanol Preservation

¹Refer to EPA Method 5035 and Regional Interim Policy for Determination of Volatile Organic Compound (VOC) Concentrations in Soil and Solid Matrices, June 23, 1999.

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EPA has permitted the use of methanol preservation for low level analysis if the target analyte(s) can be quantitated below 200 µg/kg. As a result, laboratories must demonstrate their ability to detect below 200 µg/kg to the client and ADHS. Samples preserved in the field with methanol using a 40 mL glass VOA vial with a plastic screw cap and a Teflon septa must be analyzed within 14 days from the time of sample collection.

ii EnCore™ Sampler

The sample can be collected using either a 5-gram or 25-gram EnCore™ Sampler. The sample must be stored at 4°C (±2°C) and preserved or extracted within 48 hours if not preserved. Approved preservatives include either methanol or sodium bisulfate. Once preserved, the sample must be analyzed within 14 days from the time of sample collection. The EnCore™ Sampler 48-hour preservation hold time as required in the method applies only to the EnCore™ Sampler option and is based on manufacturers' studies. Freezing the unpreserved sample in the EnCore™ Sampling device can extend the holding time up to seven days (e.g., 48 hours unfrozen and 5 days frozen).

iii Sodium Bisulfate Preservation

Samples preserved in the field with sodium bisulfate must be analyzed 14 days from the time of sample collection. This technique should be used if detection limits in the range of 2 - 5 µg/kg are desired. Calcareous samples, however, may effervesce upon contact with the sodium bisulfate preservative solution (thereby liberating the volatile gases) and compromise the integrity of the sample. In these instances, sodium bisulfate preservative solution cannot be utilized to attain the lower reporting levels and one of three alternative sample collection methods must be employed.

- a) The sample can be collected in a VOA vial containing 10 mL of reagent grade water, sealed with a plastic screw cap containing a Teflon septa and stored at 4°C (± 2°C). This sample must be analyzed within 48 hours from the time of sampling using a closed system purge and trap.
- b) The sample can be collected in a dry VOA vial, sealed with a plastic screw cap containing a Teflon septa and stored at 4°C (± 2°C). Once at the lab, water must be introduced through the

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septa and analyzed by closed purge and trap within 48 hours from the time of sample collection. Freezing the unpreserved sample can extend the holding time an additional 5 days for a total of 7 days from the time of sample collection.

- c) The sample can be collected in an EnCore™ Sampler, stored at 4°C (± 2°C) and analyzed within 48 hours from the time of sample collection. Freezing the unpreserved sample can extend the holding time up to seven days.

iv Bulk Sampling

The rationale for the collection of bulk samples must be clearly documented and approved by the appropriate program in a work or sampling plan or other written communication with ADEQ. If samples are not preserved in the field, the reasons for not preserving must be clearly documented and approved by the relevant program.

ADHS rules require laboratories to flag data generated from samples that have not been preserved in the field or have not been collected in recommended containers if the reporting levels are below 200 µg/kg.

B. Sample collection options for high reporting limits (>200 µg/kg):

i. Methanol Preservation

This technique may be used if the reporting limits are above 200 µg/kg. Samples preserved in the field with methanol using a 40 ml glass VOA vial with a plastic screw cap and a Teflon septa must be analyzed within 14 days from the time of sample collection.

ii. EnCore™ Sampler

The sample can be collected using an EnCore™ Sampler. Methanol must be added within the 48-hour period immediately following sample collection. The EnCore™ Sampler 48-hour preservation hold time as required in the method is applicable specifically only to the EnCore™ subcoring device and is based on the manufacturers' studies. After collection the sample must be stored on ice at 4°C (±2°C) until analyzed. Freezing the unpreserved sample in the EnCore™ sampling device can extend the holding time up to seven days (e.g., 48 hours unfrozen and 5 days frozen.) Once the sample is preserved, it must be analyzed within 14 days from the time of sample

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collection.

iii Bulk Sampling

The rationale for collection of bulk samples must be clearly documented and approved by the appropriate program in a work or sampling plan or other written communication with ADEQ. If samples are not preserved in the field or subsampled in EnCore™ Samplers, the reasons for not preserving must be clearly documented and approved by the relevant program.

Significant volatile loss occurs when samples are collected in glass jars and transported to a laboratory for analysis². Therefore, **glass jars with Teflon™-lined lids containing no preservative ARE NOT ACCEPTABLE** for the collection of soil for VOC analysis, unless otherwise specified in this policy (Program Specific Requirements) or prior approval has been received from the appropriate program.

III. Program Specific Requirements³:

1. WQARF, Hazardous Waste Compliance, Solid Waste Programs

When utilizing the field preservation option of the 5035 method, samples must be preserved immediately after collection with minimal handling to be considered reliable compliance samples. Samples may be collected and held on ice at 4°C (±2°C) for a maximum of 2 hours before preserving or analyzing the sample. This option of holding samples on ice for up to 2 hours is accepted, but not encouraged, due to the known volatile loss over time.

Samples collected and preserved or analyzed after 2 hours will be considered bulk samples and not suitable for compliance purposes. Data

²Siegrist, R.L., and P.D. Jennsen, 1990. Evaluation of Sampling Method Effects of Volatile Organic Compound Measurements in Contaminated Soil, *Environmental Science and Technology*, Vol.24, pp. 1387-1392.

³For specific programs, a sample collected in a brass/steel sleeve is acceptable under the conditions noted in Section IV. The brass or steel sleeves must have each end covered with a sheet of Teflon, aluminum foil(aluminum is optional, but preferred) and sealed with a plastic cap. The plastic caps must be secured and the capped sleeve should be placed in a plastic ziplock bag which is then taped to ensure the caps are secure. The use of tape to bind the cap to the end of the sleeve is discouraged. The length of time a sample can be held in this container is finite and subject to specific program requirements set forth in Section V.

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generated from samples collected and transported to a laboratory in this manner has limited compliance value and may not be accepted by the above referenced programs.

2. Hazardous Waste Inspections and Emergency Response Programs

For planned field sampling events, samples must be preserved immediately after collection, with minimal handling, to be considered compliance samples. The sample may be held on ice at 4°C ($\pm 2^\circ\text{C}$) for a maximum of 2 hours before preserving or analyzing the sample.

For unanticipated sampling events, where significant difficulties exist for preserving samples onsite, bulk soil samples may be collected and stored at 4°C ($\pm 2^\circ\text{C}$) but must be preserved within 72 hours with the approval of the program.

3. UST Program

When site-specific sampling conditions prevent the use of appropriate sample collection and preservation techniques as defined in Section I or Section II, samples may be submitted in properly sealed brass sleeve containers maintained at 4°C ($\pm 2^\circ\text{C}$) for laboratory analysis of VOCs. The laboratory must document sample holding time and flag the associated analytical results if sample preservation or extraction exceeds 48 hours, regardless of the reporting limit. Reasons for lack of field preservation within the 48 hour period and submittal of bulk samples for laboratory analysis must be clearly documented.

IV. Quality Control for unpreserved samples:

Unpreserved samples submitted to the laboratory should have matrix spikes and surrogates added directly to an aliquot of the sample before extraction. The laboratory should be requested to provide a narrative describing the procedures for sample spiking and flag all data in which the matrix was not directly spiked prior to extraction.

III. Example of Holding Time Calculations for Frozen Samples:

Example 1 Sample is placed in a vial without chemical preservative in the field and stored at 4°C ($\pm 2^\circ\text{C}$).

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The sample must be analyzed within 48 hours of collection.

Example 2 The sample is collected in a hermetically sealed subcoring and storage device in the field, stored at 4°C (±2°C) and transferred into a vial without chemical preservative in the laboratory.

The sample must be analyzed within 48 hours of collection.

Example 3 The sample is collected in a hermetically sealed subcoring and storage device, transported/stored at 4°C (±2°C), frozen at the laboratory 18 hours after collection, thawed (at ambient temperature) after 4 days and transferred into a vial without a chemical preservative in the laboratory.

The sample must be analyzed within 30 hours from the time the sample is defrosted to 4°C (±2°C).

48 hours allowed before analysis - 18 hours before freezing = 30 hours allowed from thawing (at ambient temperature) to analysis.

Freezing can only extend the holding times for unpreserved samples. Freezing is an alternative to preserving samples in the field. Freezing can never extend the holding times of samples beyond the analytical methods required holding time (e.g., Freezing cannot extend the holding time from 14 days to 19 days).

IV. RESPONSIBILITY

All staff in the respective Waste Programs Division programs are responsible for knowledge and implementation of this policy. Supervisors are responsible for ensuring that the information contained in this policy is consistently and equitably applied by all staff. It is the responsibility of the sampler to inform the laboratory receiving personnel which program requirements are appropriate for the sample.

APPROVED BY

David Esposito,
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Date 4/20/00

Mark Santana,
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Office of Administrative Counsel

Date 4/21/00

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The Policy Review Committee has posted, reviewed and accepted this policy by motion as of April 19, 2000.

Juanita Guidry Copeland,
Acting Policy Coordinator

Date 4/21/00