



Designation: D 4547 – 98

Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds¹

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1. Scope

1.1 This guide describes recommended procedures for the collection, handling, and preparation of solid waste, soil, and sediment samples for subsequent determination of volatile organic compounds (VOCs). This class of compounds includes low molecular weight aromatics, hydrocarbons, halogenated hydrocarbons, ketones, acetates, nitriles, acrylates, ethers, and sulfides with boiling points below 200° Celsius (C) that are insoluble or slightly soluble in water.

1.2 Methods of sample collection, handling, and preparation for analysis are described.

1.3 This guide does not cover the details of sampling design, laboratory preparation of containers, and the analysis of the samples.

1.4 It is recommended that this guide be used in conjunction with Guide D 4687.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 3550 Practice for Ring-Lined Barrel Sampling of Soils²
- D 4687 Guide for General Planning of Waste Sampling³
- D 4700 Guide for Soil Sampling from the Vadose Zone²
- D 5058 Test Methods for Compatibility of Screening Analysis of Waste³
- D 5792 Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives³
- D 6051 Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities³
- E 856 Definitions of Terms and Abbreviations Relating to Physical and Chemical Characteristics of Refuse-Derived Fuel³

2.2 Federal Standard:

Title 49 Transportation, Code of Federal Regulations (CFR), Part 172, List of Hazardous Substances and Reportable Quantities⁴

3. Terminology

3.1 *sample, n*—a portion of material taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity. (E 856)

3.2 *subsample, n*—a portion of a sample taken for the purpose of estimating properties or composition of the whole sample. (D 6051)

3.2.1 *Discussion*—A subsample, by definition, is also a sample.

4. Summary of Guide

4.1 This guide addresses the use of tools for sample collection and transfer, conditions for sample storage, sample preservation, and two common means of sample preparation for analysis. Special attention is given to each step from sample collection to analysis to limit the loss of VOCs by volatilization of biodegradation. The sample collected and analyzed should be representative of the parent material sampled. The two methods cited for the preparation of samples for VOC analysis are methanol extraction and vapor partitioning (that is, purge-and-trap and headspace). The method of sample preparation for VOC analysis is dependent on the data quality objectives (see Practice D 5792). Although the equilibrium-driven proportions of VOCs in the different phases change during sample preparation and analysis, the overall intent of the methods in this guide are to minimize VOC losses.

5. Significance and Use

5.1 This guide describes sample collection and handling procedures designed to minimize losses of VOCs. The principal mechanisms for the loss of VOCs from materials during collection and handling are volatilization and biodegradation. Susceptibility of various VOCs to these two loss mechanisms is both compound and matrix specific. In general, compounds with higher vapor pressures are more susceptible to volatilization than compounds with lower vapor pressures. Also, aerobically degradable compounds are generally more susceptible to biodegradation than anaerobically degradable compounds. In some cases, the formation of other compounds not originally

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² *Annual Book of ASTM Standards*, Vol 04.08.

³ *Annual Book of ASTM Standards*, Vol 11.04.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

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present in the material can occur. Loss or gain of VOCs leads to analytical results that are both unrepresentative of the field conditions and ambiguous.

5.2 Ancillary information concerning sample collection and handling for VOC analysis is provided in Appendix X1 and Appendix X2. These appendixes and cited references are recommended reading for those unfamiliar with many challenges presented during the collection and handling of samples for VOC analysis.

6. Selection of Sample Preparation Method for VOC Analysis

6.1 Introduction:

6.1.1 Sample collection, handling, and preservation methods should be compatible with the method used to prepare the sample for VOC analysis, and meet the project's data quality objectives (see Practice D 5792). Preparation of a sample for instrumental analysis can be initiated either in the field or laboratory. In either case, the sample should be placed into a tared volatile organic analysis (VOA) vial or bottle meeting the specifications given in 7.2.2. When working with an uncharacterized solid waste, it is advisable to perform compatibility tests (see Test Methods D 5058) between the sample material and the solution (see 7.2.2.1 and 7.2.2.2) into which it will be transferred in preparation for analysis. For instance, when collecting highly contaminated soils or waste of unknown composition, or both, it is strongly recommended that prelimi-

nary testing be performed to adequately characterize the waste materials so that when the user applies the procedures cited in this guide, there will be no chemical reaction which may jeopardize the user's safety.

6.1.2 Figs. 1 and 2 are flow diagrams showing some different options for combining sample collection, handling and preparation methods for instrumental analysis.

6.2 Methanol Extraction:

6.2.1 This method involves the extraction of VOCs from a sample with methanol and the subsequent transfer of an aliquot of the extract to water for either purge-and-trap or headspace analysis. Advantages of methanol extraction are (1) large samples or composite samples, or both, can be collected to enhance representatives (see Guide D 6051), (2) biodegradation is inhibited, (3) an efficient extraction of VOCs from the matrix materials can be achieved with methanol due to its strong affinity for these compounds and favorable wetting properties, (4) a subsample can be analyzed several times, and (5) sample extracts can be archived (held up to two months, and perhaps longer, if verified that VOC losses have not occurred (see 8.2.1.2)).

6.2.2 The primary disadvantages of methanol extraction are (1) samples may have to be shipped as a flammable liquid depending on the amount of methanol present (U.S. DOT reg. 49CFR§172.101), (2) hazards to personnel due to methanol's toxicity and flammability, (3) detection limits are elevated due to analyte dilution, (4) possible interference of the methanol

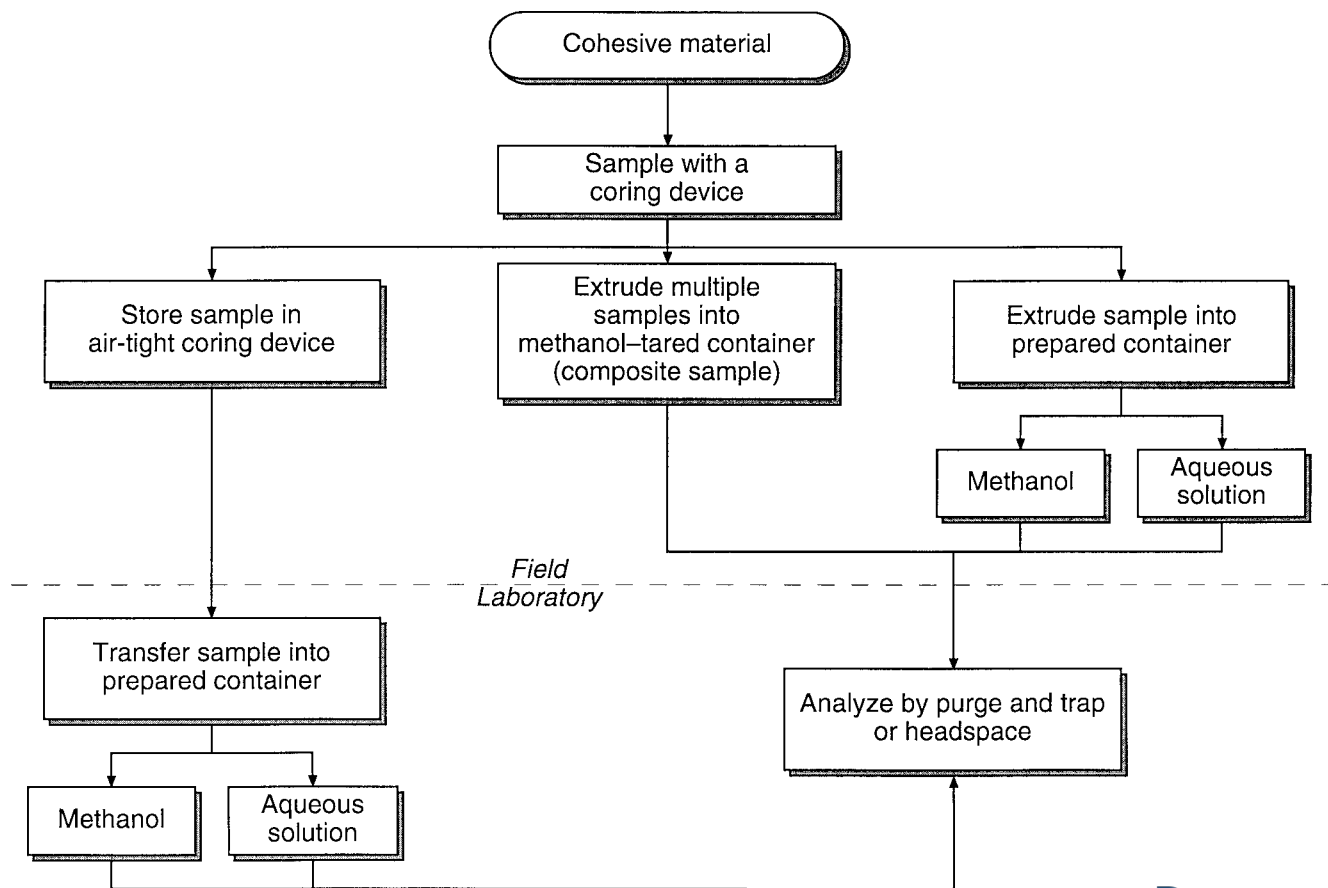


FIG. 1 Sample Handling Options for Cohesive Materials

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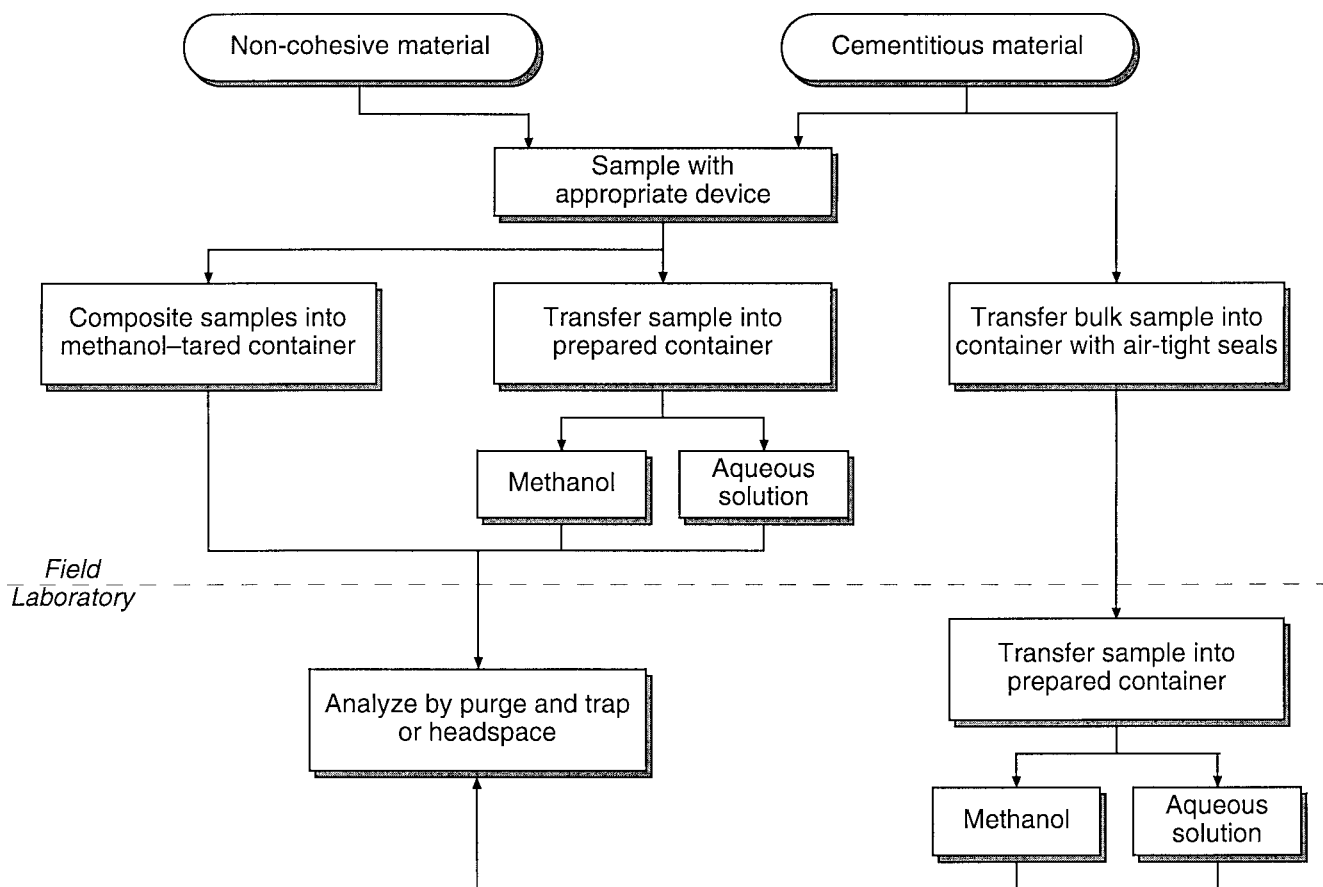


FIG. 2 Sample Handling Options for Non-Cohesive and Cementitious Materials

peak with VOCs of interest, (5) potential adverse impact of methanol on the performance of certain gas chromatograph/detector systems, and (6) in the United States of America, samples extracted with methanol must be disposed of as a regulated waste.

6.2.3 Some of these logistical challenges can be overcome by extracting samples with methanol once they have been received in a laboratory, provided that the samples are transported intact and undisturbed in an airtight container (see 8.1.1). Furthermore, if VOC levels are unknown, a replicate sample can be obtained and screened to determine if methanol extraction is appropriate for the expected contaminant concentrations.

6.3 Vapor Partitioning:

6.3.1 Vapor partitioning involves the direct analysis of a sample by either purge-and-trap or headspace. In both cases, the sample is placed into a tared volatile organic analysis (VOA) vial from which the vapor is removed for analysis without the container being opened. Heat and water are usually used to assist with the partitioning of the VOCs from the sample. The principal advantages of this method are (1) it can offer lower detection limits than methanol extraction because no dilution is involved, (2) there are no organic solvent interferences, and (3) there is no use of regulated organic solvents, which may require special shipment, disposal, and field handling practices.

6.3.2 The disadvantages associated with vapor partitioning are (1) the VOA vial (VOA vials are different sizes for automated purge-and-trap and headspace instrumentation) or

adapter used in conjunction with a VOA vial, or both, often are instrument specific, (2) sample size is limited (<10 g) by automated systems, (3) a matrix-appropriate method of preservation may be necessary, (4) vapor partitioning is less efficient at recovering VOCs from some materials than methanol extraction, and (5) when using purge-and-trap, only a single analysis of the same sample can be made; similarly only a single analysis may be possible with headspace analysis unless concentrations allow for the use of a small injection volume.

6.3.2.1 Limitations imposed by vapor phase partitioning methods with regard to number of analyses that can be performed on a single sample can be addressed by taking replicate samples.

6.3.3 When employing vapor phase partitioning methods, the logistical challenges of performing sample preparation in the field (see 7.2.2.2) can be avoided by performing the preparation step in the laboratory, so long as the sample is transported to the laboratory intact and undisturbed, in an airtight container (see 8.1.1). If VOC levels are unknown, a replicate sample can be obtained and screened to determine if it is appropriate to use of vapor partitioning method of sample preparation.

7. Sample Collection

7.1 This section provides general sampling guidelines for a variety of materials and conditions. The guidelines are intended to allow flexibility in the following: