



Designation: D3686 – 20

Standard Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)¹

This standard is issued under the fixed designation D3686; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a method for the sampling of atmospheres to determine the presence of certain organic vapors by means of adsorption on activated charcoal using a charcoal tube and a small portable sampling pump worn by a worker. A list of some of the organic chemical vapors that can be sampled by this practice is provided in **Annex A1**. This list is presented as an information guide and should not be considered as absolute or complete.

1.2 This practice does not cover any method of sampling that requires special impregnation of activated charcoal or other adsorption media.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* A specific safety precaution is given in **9.4**.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

¹ This practice is under the jurisdiction of ASTM Committee **D22** on Air Quality and is the direct responsibility of Subcommittee **D22.04** on Workplace Air Quality. Current edition approved March 1, 2020. Published June 2020. Originally approved in 1978. Last previous edition approved in 2013 as D3686 – 13. DOI: 10.1520/D3686-20.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D3687 Test Method for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method

D4840 Guide for Sample Chain-of-Custody Procedures

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

2.2 NIOSH Standards:

CDC-99-74-45 Documentation of NIOSH Validation Tests³

HSM-99-71-31 Personal Sampler Pump for Charcoal Tubes; Final Report³

NIOSH Manual of Analytical Methods⁴

2.3 OSHA Standards:

29 CFR 1910 U.S. Code of Federal Regulations Relating to Labor, U.S. Occupational Safety and Health Administration, Department of Labor⁵

OSHA Chemical Sampling Information⁶
OSHA Sampling and Analytical Methods⁷

2.4 Other Documents:

ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories⁸

3. Terminology

3.1 **Definitions**—For definitions of terms used in this method, refer to Terminology **D1356**.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 **activated charcoal, n**—charcoal that has been heated or otherwise treated to increase its adsorptive power.

3.2.1.1 **Discussion**—A common procedure is to increase the inherent porosity of charcoal derived from biological precursors (for example, nut shells) by steam activation. Prior to use

³ Available from National Technical Information Service (NTIS), 5285 Port Royal Rd., Springfield, VA 22161, <http://www.ntis.gov>.

⁴ NIOSH Manual of Analytical Methods, <http://www.cdc.gov/niosh/nmam>.

⁵ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

⁶ OSHA Chemical Sampling Information, http://osha.gov/dts/chemicalsampling/toc/toc_chemsamp.html.

⁷ OSHA Sampling and Analytical Methods, <http://osha.gov/dts/sltc/methods/toc.html>.

⁸ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

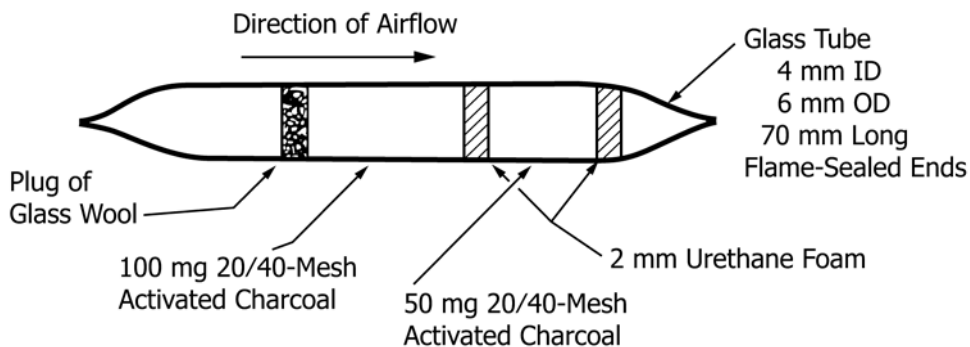


FIG. 1 Activated Charcoal Adsorption Sampling Tube

in this application, activated charcoals may need to be further treated to remove remnant organic compounds.

4. Summary of Practice

4.1 Air samples are collected for organic vapor analysis by aspirating air at a known rate and for an appropriate time through sampling tubes containing activated charcoal.

4.2 Instructions are given to enable assembly of charcoal tubes suitable for sampling purposes.

4.3 Information on the correct use of the charcoal tube sampling device is presented.

4.4 Test Method D3687 describes a method for the analysis of these samples.

5. Significance and Use

5.1 Promulgations by the U.S. Occupational Safety and Health Administration (OSHA) in 29 CFR 1910.1000 designate that certain organic compounds must not be present in workplace atmospheres at concentrations above specific values.

5.2 This practice, when used in conjunction with Test Method D3687, will provide the needed accuracy and precision for the determination of airborne time-weighted average concentrations of many of the organic chemicals including but not limited to CDC-99-74-45, HSM-99-71-31, NIOSH Manual of Analytical Methods, 29 CFR 1910.1000, OSHA Chemical Sampling Information, OSHA Sampling and Analytical Methods, and HSE Methods for the Determination of Hazardous Substances.

5.3 A partial list of chemicals for which this method is applicable is given in Annex A1, along with their OSHA permissible exposure limits.

6. Interferences

6.1 Water mist and vapor can interfere with the collection of organic compound vapors. Humidity greater than 60 % can reduce the adsorptive capacity of activated charcoal up to 50 % for some chemicals (1).⁹ Presence of condensed water droplets in the sample tube will indicate a suspect sample. Water vapor co-collected by the sorbent can lead to poor recovery of polar

and reactive chemicals, for example, acetone, although it should not affect most chemicals listed in Annex A1. Alternative sorbents are available for sampling polar and reactive compounds at high humidity. Users are advised to consult their analytical laboratory, method documentation, or associated literature for more information.

6.2 High levels of organic vapors can interfere by reducing the amount of time a workplace can be sampled before the charcoal sampler becomes saturated.

7. Apparatus

7.1 Charcoal Tube:

7.1.1 A sampling tube consists of a length of glass tubing usually containing two sections of activated charcoal that are held in place and separated by nonadsorbent material. The tube is sealed at each end.

7.1.1.1 Sampling tubes are commercially available. The tubes are usually divided into two sections with the front section containing 100 to 800 mg of activated charcoal and the back section containing 50 to 400 mg of activated charcoal. The 100/50-mg tube ((2-4) and Fig. 1) is the one most frequently used, it consists of a glass tube that is 70-mm long, 6-mm outside diameter, 4-mm inside diameter, and contains two sections of 20/40 mesh-activated coconut-shell charcoal separated by a 2-mm section of urethane foam. The front section of 100 mg is retained by a plug of clean glass wool, and the back section of 50 mg is retained by either a second 2-mm portion of urethane foam or by a plug of clean glass wool. Both ends of the tube are usually flame-sealed.

NOTE 1—Urethane foam is known to adsorb certain pesticides (5). Contaminated urethane foam should not be used for this practice.

7.1.1.2 When it is desirable to sample highly volatile compounds for extended periods, or at a high volume flow rate, a larger device capable of efficient collection can be used, provided the proportions of the tube and its charcoal contents are scaled similarly to the base dimensions to provide nominally the same linear flow rate and contact time with the charcoal bed.

7.1.2 The back portion of the sampler tube usually contains 50 % of the mass of activated charcoal present in the front section. The back section adsorbs vapors that penetrate the front section and serves as a warning that breakthrough may have occurred. (Annex A1 gives recommended maximum tube loading information for many chemicals.)

⁹ The boldface numbers in parentheses refer to the list of references at the end of this standard.

7.1.3 The adsorptive capacity and extraction efficiency (also called desorption efficiency) of different batches of activated charcoal can vary. Commercial tubes, if used, should be purchased from the same batch and in sufficient number to provide sampling capability for an adequate period of time. *Care must be taken to have enough tubes from the same batch for a given study.*

7.1.4 Pressure drop across the sampling tube should be less than 3.3 kPa (25 mm Hg) at a flow rate of 1000 mL/min and less than 0.61 kPa (4.6 mm Hg) at a flow rate of 200 mL/min.

7.1.5 Charcoal sampling tubes prepared in accordance with this practice and with sealed glass ends can be stored indefinitely. Accrediting bodies, such as American Industrial Hygiene Association, may require that sampling media have an expiration date. Accredited facilities must abide by this date.

7.2 Sampling Pumps:

7.2.1 Any pump with a flow rate that can be accurately determined, that can be set at the desired sampling rate, and that can maintain the desired sampling rate for a sufficient time is suitable. Primarily though, this practice is intended for use with small personal sampling pumps.

7.2.2 Pumps having stable low flow rates (10 to 200 mL/min) are preferable for long period sampling (up to 8 h) or when the concentration of organic vapors is expected to be high. Reduced sample volumes will avoid exceeding the adsorptive capacity of the charcoal tubes. (Suggested flow rates and sampling times are given in **Annex A1** for anticipated concentration ranges.) (Sample volumes are discussed in 9.5.)

7.2.3 Pumps are available that will provide stable flow rates between $\pm 5\%$ of the desired flow rate. Flow rate through the charcoal tubes should be measured before and after sampling with an instrument calibrated for the purpose.

7.2.4 A sampling tube holder with flexible tubing is used to connect the sampling tube to the sampling pump. The sampling tube holder is used to protect the worker from the sharp end of the sampling tube.

7.3 Flowmeter:

7.3.1 Flowmeter, portable, with an accuracy that is sufficient to enable the volumetric flow rate to be measured to within $\pm 5\%$. The flowmeter calibration by a provider accredited to ISO/IEC 17025 for such calibrations shall be traceable to national or international standards. Retain the calibration certificate, including the pressure and temperature at which the calibration was performed, and identifying and performance documentation for the flowmeter.

8. Reagents

8.1 *Activated Charcoal*—Prior to being used to make sampling devices the charcoal should be heated in an inert gas at an appropriate temperature for a sufficient time to ensure background of organic compounds sufficiently low as to not interfere with the analysis of low concentrations of volatile organic compounds (VOC). Commercially available coconut-shell charcoal (20/40 mesh) has been found to have adequate adsorption capacity for many volatile chemicals. Other charcoals, such as petroleum-based charcoal and proprietary charcoals, can be used for appropriate applications.

9. Sampling with Activated Charcoal Samplers

9.1 *Setting the Flow-rate of the Sampling System*—The sampling system consists of a pump, flow regulator, tubing to be used, and a representative charcoal tube (or an equivalent induced resistance). The air flow through the sampling system should be adjusted and set at the desired rate by means of a flowmeter calibrated traceably to national or international standards (see **D5337**). The calibration of the flow meter must be performed by an organization accredited for the purpose according to ISO 17205. The flow through the sampling system shall be measured in a clean location with similar temperature and barometric pressure as the sampling site. Normally, pump flow rates or sample air volumes are not corrected for temperature or barometric pressure where concentrations are calculated for normal temperature and pressure.

9.2 Break open both ends of the charcoal tube to be used for sampling, ensuring that each opening is at least one half the inside diameter of the tube.

9.3 Insert the charcoal tube into the connective flexible tubing, placing the back-up section nearest to the pump. At no time should there be any tubing ahead of the sampling tubes. Use a sampling tube holder to protect the worker from the sharp end of the sampling tube.

9.4 For a breathing zone sample, fasten the sampling pump to the worker, and attach the sampling tube as close to the worker's breathing zone as possible. Position the tube in a vertical position to avoid channeling of air through the charcoal sections. (**Warning**—Assure that the presence of the sampling equipment is not a safety hazard to the worker and that the equipment will not interfere with the worker's duties.)

9.4.1 Turn on the pump.

9.4.2 Record the flow rate, the starting time, and depending on the make of pump used, the register reading.

9.5 *Sampling Volumes*—The minimum sample volume will be based on the detection limit of the analytical method, and the maximum sample volume will be based on either the adsorptive capacity of the charcoal or limitations of the pump and battery.

9.5.1 One method of calculating required sample volumes is to determine first the concentration range, over which it is important to report an exact number, for example from 0.2 to 2 times the permissible exposure concentration, and then calculate the sample volumes as follows:

$$\text{Minimum sample volume, m}^3 = \quad (1)$$

$$\frac{\text{minimum detection limit, mg}}{0.2 \times \text{permissible exposure limit, mg/m}^3}$$

$$\text{Maximum sample volume, m}^3 = \quad (2)$$

$$\frac{\text{tube capacity for vapors, mg}}{2 \times \text{permissible exposure limit, mg/m}^3}$$

9.5.2 Select a sampling rate that, in the sampling time desired, will result in a sample volume between the minimum and maximum calculated in 9.5.1.

9.5.2.1 Generally a long sampling time at a low flow rate is preferable to short-term, high-volume sampling. This is consistent with the fact that most health standards are based on