



# Standard Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method<sup>1</sup>

This standard is issued under the fixed designation D 3687; 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 the applications of methods for the extraction and gas chromatographic determination of organic vapors that have been adsorbed from air in sampling tubes packed with activated charcoal.

1.2 This practice is complementary to Practice D 3686.

1.3 This practice is applicable for analysis of samples taken from workplace or other atmospheres provided that the contaminant adsorbs onto charcoal, that it can be adequately extracted from the charcoal, and that it can be analyzed by gas chromatography (GC). Other adsorbents and other extraction techniques are described in Practice D 6196.

1.4 Organic compounds of multicomponent samples may mutually interfere during analysis. Methods to resolve interferences are given in Section 6.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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.* Specific precautions are given in 8.4, 9.2, and in A1.2.3.

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres

D 3686 Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)

D 6196 Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air

E 355 Practice for Gas Chromatography Terms and Relationships

2.2 *NIOSH Standards:*

CDC-99-74-45 Documentation of NIOSH Validation Tests<sup>3</sup>

NIOSH Manual of Analytical Methods, 4<sup>th</sup> Ed.<sup>4</sup>

2.3 *OSHA Standards:*

29 CFR 1910 Code of Federal Regulations, Regulations Relating to Labor, Occupational Safety and Health Administration, Department of Labor<sup>5</sup>

OSHA Sampling and Analytical Methods<sup>6</sup>

2.4 *UK Health and Safety Executive (HSE):*<sup>7</sup>

Methods for the Determination of Hazardous Substances (MDHS)

2.5 *Berufsgenossenschaftliches Institut für Arbeitsschulz (BGIA):*<sup>8</sup>

GESTIS Analytical Methods

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this practice, refer to the terminology specified in D 1356 and E 355.

## 4. Summary of Practice

4.1 Organic vapors that have been collected on activated charcoal are extracted with carbon disulfide or another appropriate solvent and are determined by GC using a flame ionization detector (FID). Carbon disulfide is a relatively small molecule that can penetrate the “ink-bottle” shaped pores of activated charcoal, it has a high heat of adsorption on activated charcoal which helps in displacing other adsorbed molecules, and it is a reasonably good solvent for most, especially

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from the U.S. Department of Commerce, National Technical Information Service, Port Royal Road, Springfield, VA 22161.

<sup>4</sup> NIOSH Manual of Analytical Methods (NMAM). <http://www.cdc.gov/niosh/nmam/> (accessed 1/2007).

<sup>5</sup> Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

<sup>6</sup> OSHA Sampling and Analytical Methods, <http://www.osha.gov/dts/sltc/methods/index.html> (accessed 1/2007).

<sup>7</sup> HSE Methods for the Determination of Hazardous Substances (MDHS). <http://www.hse.gov.uk/pubns/mdhs/index.htm#> (accessed 1/2007).

<sup>8</sup> GESTIS Analytical Methods. [http://www.hvbg.de/e/bia/gestis/analytical\\_methods/index.html](http://www.hvbg.de/e/bia/gestis/analytical_methods/index.html) (accessed 1/2007).

non-polar, organic molecules. Polar modifiers (such as *N,N*-dimethylformamide) are frequently added to enhance the recovery of polar organic compounds. Other advantages to using carbon disulfide include an early elution time on most GC columns and a small FID response.

4.2 Interferences resulting from the analytes having similar retention times during GC analysis are resolved by changing the GC column, by changing the operating parameters, or by fractionating the sample using solvent extraction as described in Section A1.1.

4.3 Peak purity and identity can be confirmed using techniques such as GC/MS.

## 5. Significance and Use

5.1 Promulgations by the Federal Occupational Safety and Health Administration (OSHA) in 29 CFR 1910 designate that certain organic compounds must not be present in workplace atmospheres at concentrations above specified values.

5.2 This practice, when used in conjunction with Practice D 3686, will promote needed accuracy and precision in the determination of airborne concentrations of many of the organic chemicals given in 29 CFR 1910, CDC-99-74-45, NIOSH Manual of Analytical Methods, OSHA Sampling and Analytical Methods, HSE Methods for the Determination of Hazardous Substances, and BGIA GESTIS Analytical Methods. It can be used to determine worker exposures to these chemicals, provided appropriate sampling periods are used.

5.3 Most laboratories are equipped with apparatus similar to that described in Section 7. Other apparatus can be used when analytical procedures suitable for that equipment are employed. The analytical techniques (or variations thereof) described in Sections 9-11 are in general use to analyze volatile organic compounds extracted from charcoal. Other procedures can be used when appropriate.

## 6. Interferences

6.1 Any chemical that produces an FID response and has a similar retention time as the analyte is a potential interference. If potential interferences were reported when the samples were received they should be considered before the samples are extracted. Generally, gas chromatographic conditions such as the type of GC column (phase) or operating parameters can be changed to resolve interferences.

6.2 Selective solvent stripping techniques have been used successfully to make clean and fast separations of polar, nonpolar and oxygenated compounds. A general guideline is given in A1.1 and detailed procedures are given in Refs<sup>9</sup> (1 and 2).

6.3 When necessary, the identity or purity of an analyte peak can be confirmed by GC/mass spectrometry.

6.4 The presence of co-adsorbed chemicals can affect the recovery (extraction efficiency) of a particular analyte. Suspected effects can be tested by spiking the analytes on charcoal as in Section 11.

## 7. Apparatus

7.1 *Gas chromatograph*, equipped with a flame ionization detector (FID), a temperature-programmable oven, and an automatic sample injector (autosampler). Sample injection may be performed manually if necessary. Other detectors (such as electron capture, flame photometric, nitrogen phosphorous detectors) can be used when appropriate but the extraction solvent may have to be modified.

7.2 *Electronic data system*, or other suitable means to record and measure detector response, to prepare calibration curves, and to process sample results.

7.3 *GC columns*, required to separate the complex mixture of possible organic chemicals. Examples of the most common and useful GC columns are 60-m long, 0.32-mm i.d. fused silica capillary GC columns with 0.1 to 1- $\mu$ m thick (df) phases such as 100 % dimethyl polysiloxane, 95 % dimethyl-5% diphenyl polysiloxane, and polyethylene glycol.

7.4 *Laboratory glassware*, calibrated syringes, calibrated solvent dispensers, assorted Class A pipets and volumetric flasks and glass automatic sample injector (autosampler) vials with PTFE septum caps to contain analytical standards and samples.

## 8. Reagents

8.1 *Analytical standards*, reagent grade or better, typically 97-99+%.

8.2 *Carbon disulfide*, reagent grade or better, typically 99.9 % with low benzene content.

8.3 *Internal standard*, reagent grade or better, typically 99+%, *p*-cymene and 1-phenyl hexane are often used. Other internal standard reagents can be used providing that they not appear in air samples and that they are fully tested.

8.4 *Extraction solvent*, usually consists of 0.25 microlitres of internal standard per milliliter of carbon disulfide. Other extraction solvents can be used provided they are fully tested. (**Warning**—Carbon disulfide is toxic and extremely flammable, as are many of the organic chemicals to be analyzed. Work with these chemicals in a properly functioning laboratory hood.)

## 9. Calibration

9.1 In general, follow the manufacturer's manual and safety instructions to set up the gas chromatograph. Always use high purity gases and high quality gas purifiers.

9.2 Install the selected GC column and set the linear velocity of the carrier gas following manufacturer's instructions. Set the injector split ratio at 10:1 or at some other appropriate ratio. The most commonly used capillary GC carrier gas is hydrogen. Set the injector, detector, and column oven temperatures appropriate for the selected GC column. It is often useful to heat the GC column at 10-20°C below the expected maximum operating temperature of the column for about two hours before any analysis is performed. Before analyzing standards or samples, place a fresh septum into the injection port of the chromatograph. Replace the septum daily or when necessary. Septum failure is a frequent cause of inconsistent FID response and changes in chromatography.

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