



Designation: D4600 – 22

# Standard Test Method for Determination of Benzene-Soluble Particulate Matter in Workplace Atmospheres<sup>1</sup>

This standard is issued under the fixed designation D4600; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes the sampling and gravimetric determination of benzene-soluble particulate matter that has become airborne as a result of certain industrial processes. This test method can be used to determine the total weight of benzene-soluble materials and to provide a sample that may be used for specific and detailed analyses of the soluble components.

1.2 The limit of detection is 0.05 mg/m<sup>3</sup> by sampling a 1 m<sup>3</sup> volume of air.

NOTE 1—Other volatile organic solvents have been used for this determination and whereas a less toxic solvent for this analysis might be desirable, the substitution of a solvent other than benzene is unwise at this time. A tremendous volume of environmental sampling data based on benzene-soluble determinations has been accumulated over many years in several industries.<sup>2</sup> Some of the determinations have been used in epidemiological studies. Furthermore, the use of benzene is specified in existing United States federal standards.<sup>3</sup> As a result, it appears imprudent to use a different solvent until the qualitative and quantitative relationship of analyses derived from benzene and a substitute solvent is established. With proper care, benzene can be safely used in the laboratory.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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<sup>2</sup> “Benzene-Soluble Compounds in Air, P&CAM 217,” *NIOSH Manual of Analytical Methods*, 2nd Ed., DHEW (NIOSH) Pub. No. 77-157-A, National Institute of Occupational Safety and Health, Cincinnati, OH 1977.

<sup>3</sup> “Appendix B—Industrial Hygiene and Medical Surveillance Guidelines,” 20CFR 1910.1029 Coke Oven Emissions.

*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:<sup>4</sup>

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D4840 Guide for Sample Chain-of-Custody Procedures

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

D8358 Guide for Assessment and Inclusion of Wall Deposits in the Analysis of Single-Stage Samplers for Airborne Particulate Matter

### 2.2 Other Documents:<sup>5</sup>

ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories

## 3. Terminology

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

## 4. Summary of Test Method

4.1 By the use of a personal sampling pump, air is pulled through a glass-fiber filter held in a sampling cassette. The filter is extracted ultrasonically with benzene. After filtration, an aliquot of the total extract is evaporated to dryness and the residue weighed.

## 5. Significance and Use

5.1 This test method provides a means of evaluating exposures to benzene-soluble particulate matter in a concentration range that can be related to occupational exposures.

<sup>4</sup> 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.

<sup>5</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

## 6. Interferences

6.1 This test method is, by definition, free of interferences. However, this test method is nonspecific. It supplies no information on the composition of the soluble material. It measures all those substances in the sample that are soluble in benzene. The composition must be determined by some independent means.

6.2 The greatest errors most likely to be incurred in the use of this test method are associated with the sample collection or high blanks from solvent and filter. Avoid contamination from extraneous material by using high-purity solvents for the final cleaning of all apparatus. The use of plastic containers other than polytetrafluoroethylene (PTFE) for any organic solvent is unacceptable, and the evaporation step should be conducted in a clean hood.

## 7. Apparatus

7.1 *Personal Sampling Pumps*, any pump capable of sampling at a rate of about 2 L/min for 8 h.

7.2 *Flow Measuring Device*, used in accordance with Practice [D5337](#).

7.3 *Flexible Tubing*, of a diameter suitable for making a leak-proof connection from the personal sampling pumps to the samplers.

7.4 *Glass-Fiber Filters*, 37 mm, free of organic binder, held in a two- or three-piece polystyrene cassette having a rigid filter support. Filters should be pre-extracted or preheated at 400 °C for a minimum of 1 h.

7.4.1 There exists the possibility that sample may enter the sampling cassette and be collected on the inner surfaces of the sampler rather than on the filter. The use of conductive plastic cassettes rather than polystyrene should reduce the amount of fine particulate electrostatically attracted to the inner walls of the cassette, but may not eliminate the issue entirely. See Guide [D8358](#) for additional guidance on including non-filter sampler deposits for analysis.

7.5 *Extraction Vessel*, 25 mL Erlenmeyer flask, 8-dram vial or any glass container of 10 mL to 25 mL capacity having a PTFE-lined screw cap (a 50 mL beaker is suitable if entire sample is to be used for the benzene-soluble determination).

7.6 *Ultrasonic Bath*, 100 W to 500 W, sufficient to disperse the particulate without destroying the glass-fiber matrix.

7.7 *Filtration Apparatus*—Align tubes with fine porosity fritted glass, 30 mL; Gelman Acrodisc CR disposable syringe filter (0.45 µm to 1.0 µm pore size) attached to a 5 mL to 10 mL syringe having a Luer tip; or other equivalent suitable media.

NOTE 2—When using a disposable syringe filter, prewash with 10 mL of acetonitrile.

NOTE 3—Placing a pre-extracted filter (37 mm glass fiber) ahead of the fritted glass in the Allihn tube will increase the life of the frit.

7.8 *Microbalance*, capable of weighing with a resolution of 10 µg. (If entire sample is to be used for the benzene-soluble determination, a five-decimal balance is acceptable.)

7.9 *Weighing Cups*, aluminum or PTFE of suitable capacity with a weight not to exceed 500 mg.

7.10 *Vacuum Oven*.

## 8. Reagent

8.1 *Benzene*—The benzene used should be of sufficient purity so that the residue weight following evaporation of 5 mL of the solvent is less than 10 µg.

## 9. Safety Precautions

9.1 To minimize the exposure hazard to benzene, analysts should avoid inhalation of solvent vapors and, if indicated, be monitored to determine the magnitude of their exposures. Transfer and evaporation of the solvent should be carried out in a properly ventilated fume hood (150-fpm face velocity). Protective gloves should be used to minimize the possibility of absorbing benzene through the skin. See safety data sheets (SDS) for additional hazard and handling information.

9.2 The extract must be handled with proper caution because some polycyclic aromatic hydrocarbons are potential carcinogens.

## 10. Sampling

10.1 Refer to Practices [D1357](#) for general information on sampling.

10.2 Adjust the personal sampling pumps to about 2 L/min with an assembled cassette between the pump and the flow-measuring device, using a flexible tubing, in accordance with Practice [D5337](#). After the sampling, perform a post sampling flow rate verification. Pre- and post-sampling flow rates are expected to be within ±5 % of a nominal value, in which case the nominal value can be used, or within ±5 % of each other, when the average value can be used. If the difference between the two measurements is greater than ±5 % then the sample shall be flagged and the two separate results recorded, or the sample is discarded. The smaller value of flow rate can be used to determine an upper bound to the time-weighted average concentration, while the larger value provides a lower bound. The user of the data shall decide how to deal with the results and must justify their choice as appropriate.

10.3 Attach the cassette, in the closed face mode, to the sampling pump using the flexible tubing, and sample in the breathing zone of the employee for the entire shift. The pump should be checked periodically to ensure maintenance of the adjusted flow rate.

10.4 Upon completion of sampling, plug the inlet and outlet of the filter cassette using supplied plastic plugs and send it to the laboratory for analysis. The cassette must be protected from exposure to heat and light. To minimize deterioration of samples prior to analysis, they may be stored in a freezer.

10.5 Field blanks are treated in the same manner as samples. They are opened in the environment to be sampled and immediately closed and placed with the samples to be sent to the laboratory for analysis. An unopened, unused cassette assembly is used as a laboratory blank. At least one laboratory blank and one field blank should be submitted with each set of samples collected.

10.6 Follow sampling chain of custody procedures in accordance with Guide D4840 to ensure sample traceability. Ensure that the documentation that accompanies the samples is suitable for a “chain of custody” to be established.

## 11. Calibration

11.1 *Balance*—Check balance calibration and adjust per manufacturer’s recommendations.

## 12. Procedure

12.1 Remove the filter from the cassette and, using forceps, insert into the extraction vessel. Treat the blanks in the same manner as the samples.

12.2 Pipet 5.0 mL of benzene into the extraction vessel. Seal immediately with a PTFE-lined cap if an aliquot of the sample is to be used for analysis.

12.3 Place the extraction vessel in the ultrasonic bath for 5 min and then allow the vessel to stand for 30 min. If the entire sample is to be used for the benzene-soluble determination, proceed to 12.8.

12.4 Decant the solution into the filter vessel. Immediately pressure or vacuum filter the solution through the filter into a clean, dry collection vessel. The entire 5 mL volume of benzene solution need not be recovered; however, evaporation losses must be minimized. On completion of filtration, seal the vessel immediately.

12.5 Weigh the weighing cup to the nearest microgram on the microbalance. Store the weighed cup in a covered glass petri dish.

12.6 Pipet an aliquot no less than 0.5 mL of the solution from 12.4 into the tared weighing cup. Evaporate the solvent in a preheated vacuum oven at 40 °C and 26.66 kPa and continue drying for a total of 3 h.

NOTE 4—It is recommended to air evaporate benzene to dryness under a hood before transferring to the preheated vacuum oven. This will minimize benzene exposures in the laboratory.

12.7 After cooling, reweigh the cup against the same tare used in 12.5. The difference between the weight obtained and the tare obtained in 12.5 is the residue weight for the aliquot.

12.8 When entire sample is to be used for the benzene-soluble determination, decant the solution into the filtering vessel. Pressure filter the solution into a preweighed ( $\pm 0.01$  mg) 50 mL beaker. Rinse filtration apparatus with three 1 mL washes of benzene, directly into the beaker.

12.9 Evaporate the solvent in a preheated vacuum oven at 40 °C and 26.66 kPa and continue drying for a total of 3 h.

Reweight the beaker after cooling. The difference between the final weight and the tare weight of the beaker is the residue weight of the sample.

## 13. Calculation

13.1 Calculate the air concentration of soluble particulate organic matter as follows:

$$C = \frac{(A - B) 5}{V F} \quad (1)$$

where:

$C$  = concentration, mg/m<sup>3</sup>,

$A$  = residue weight of sample, mg,

$B$  = residue weight of blank, mg,

$V$  = volume of air sample, m<sup>3</sup>, and

$F$  = volume of aliquot, mL.

## 14. Precision and Bias<sup>6</sup>

14.1 *Precision, Repeatability (Both Single Analyst and Overall)*—A high-volume air sample was collected on a filter at a coke oven battery and extracted with benzene. The extract was used to generate four levels of benzene soluble material: 0.09, 0.18, 0.36, and 0.72 mg/sample. Each concentration was analyzed in triplicate by nine participating laboratories. The single operator relative standard deviation (RSD) varied from 25 % to 5 % with a pooled relative standard deviation of 13 %. The overall relative standard deviation for the nine laboratories varied from 47 % to 10 % with a pooled RSD of 32 %.

14.2 *Bias*—The bias of this method has not been determined because the reference values were established by the method itself.

## 15. Report

15.1 Data to report shall include, at a minimum, the following:

- 15.1.1 All sample receipt and chain-of-custody information.
- 15.1.2 Sample analysis results.
- 15.1.3 Applicable quality assurance/quality control data.
- 15.1.4 Identity of laboratory and analyst(s).
- 15.1.5 Record any relevant observations on received samples or sample changes during recovery.

## 16. Keywords

16.1 air monitoring; benzene-soluble particulate matter; coal tar pitch volatiles; sampling and analysis; workplace atmospheres

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained on loan by requesting Research Report RR:D22-1016. Contact ASTM Customer Service at service@astm.org.