Standard Test Method for Determination of Benzene-Soluble Particulate Matter in Workplace Atmospheres[1]

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 (ε) 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^3 by sampling a 1-m^3 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.[2] Some of the determinations have been used in epidemiological studies. Furthermore, the use of benzene is specified in existing federal standards.[3] 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[4]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

3. Terminology

3.1 *Definitions:*

3.1.1 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.

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

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[1] 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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[2] "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.

[3] "Appendix B—Industrial Hygiene and Medical Surveillance Guidelines," 20CFR 1910.1029 Coke Oven Emissions.

[4] 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.

than TFE-fluorocarbon 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 calibration rate of about 2 L/min for 8 h.

7.2 *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.

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

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

7.5 *Filtration Apparatus*—Allihn tubes with fine porosity fritted glass, 30 mL; Gelman Acrodisc CR disposable syringe filter (0.45 to 1.0 µm pore size) attached to a 5 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.

7.6 *Microbalance,* capable of weighing to 1 µg. (If entire sample is to be used for the benzene-soluble determination, a five-place balance is acceptable.)

7.7 *Weighing Cups,* aluminum or TFE-fluorocarbon of suitable capacity with a weight not to exceed 500 mg.

7.8 *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.

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

10. Sampling

10.1 Place the cassette assembly, in the closed face mode, in the breathing zone of the employee and for the entire shift, draw air through the filter using a calibrated sampling pump. The pump should be operated at a rate of about 2 L/min and should be checked periodically to ensure maintenance of this rate.

10.2 Upon completion of sampling, seal the cassette with the plugs provided 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.

10.3 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.

11. Calibration and Standardization

11.1 No standards are required for this test method.

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 TFE-fluorocarbon-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 (±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. Reweigh 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:

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

where:

C = concentration, mg/m^3,
A = residue weight of sample, mg,
B = residue weight of blank, mg,
V = volume of air sample, m^3, and
F = volume of aliquot, mL.

14. Precision and Bias[5]

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. Keywords

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

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[5] 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.

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