



Designation: D6494 – 22

Standard Test Method for Determination of Asphalt Fume Particulate Matter in Workplace Atmospheres as Benzene Soluble Fraction¹

This standard is issued under the fixed designation D6494; 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 covers the determination of asphalt fume particulate matter (as benzene soluble fraction) and total particulate matter weight in workplace atmospheres using a polytetrafluoroethylene (PTFE) filter methodology.

1.2 This procedure has been adapted from NIOSH Method 5023 (withdrawn prior to 4th edition (1994) and replaced in 1998 with NIOSH Method 5042) and OSHA Method 58. This adaptation was made to reduce the level of background contamination providing better reproducibility.

1.3 This procedure is compatible with high flow rate personal sampling equipment—0.5 to 2.0 L/min. It can be used for personal or area monitoring.

1.4 The sampling method develops a time-weighted average (TWA) sample and can be used to determine short-term exposure limit (STEL).

1.5 The applicable concentration range for the TWA sample is from 0.2 to 2.0 mg/m³.

NOTE 1—A study has suggested candidate solvents for benzene replacement.² A less toxic solvent for this analysis would be more appropriate, although the substitution with a solvent other than benzene needs further validations with field data.

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

1.7 *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.* For more specific precautionary statements, see Section 9.

1.8 *This international standard was developed in accordance with internationally recognized principles on standard-*

ization 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:*³

[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](#)

[D6062 Guide for Personal Samplers of Health-Related Aerosol Fractions](#)

[D8358 Guide for Assessment and Inclusion of Wall Deposits in the Analysis of Single-Stage Samplers for Airborne Particulate Matter](#)

2.2 *Occupational Safety and Health Administration (OSHA) Methods:*⁴

[OSHA Method 58 Coal Tar Pitch Volatiles](#)

2.3 *National Institute for Occupational Safety and Health (NIOSH) Manual of Analytical Methods:*⁵

[NIOSH Method 5023 Coal Tar Pitch Volatiles](#)

[NIOSH Method 5042 Benzene—Soluble Fraction and Total Particulate \(Asphalt Fume\)](#)

2.4 *Other Documents:*

[ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories](#)⁶

3. Terminology

3.1 *Definitions*—For definitions of terms relating to this test method, refer to Terminology [D1356](#).

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

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² Sutter, B., Ravera, C., Hussard, C., Langlois, E., “Alternatives for Benzene in the Extraction of Bitumen Fume from Exposure Sample Media,” *Ann. Occup. Hyg.*, Vol 60, No. 1, 2015, pp. 101–112.

³ 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.

⁴ Available from Occupational Safety and Health Administration (OSHA), 200 Constitution Ave., NW, Washington, DC 20210, <http://www.osha.gov>.

⁵ Available from National Institute for Occupational Safety and Health (NIOSH), Cincinnati, OH. <http://www.cdc.gov>

⁶ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3.1.1 *asphalt fume particulate matter*—particulate matter generated during the processing of hot asphalt.

4. Summary of Test Method

4.1 A known volume of sample air is passed through a PTFE filter mounted in an appropriate air sampler. Asphalt fumes are removed from the air stream by deposition on the filter.

4.2 The asphalt fume is extracted with a known volume of benzene. The benzene extract is then evaporated to dryness in a vacuum oven. The benzene soluble fraction (BSF) is then determined gravimetrically.

5. Significance and Use

5.1 Asphalt is a material used in the construction of roads and as a roofing material and sealant.

5.2 This test method provides a means of evaluating exposure to asphalt fume in the working environment at the presently recommended exposure guidelines (for example, Threshold Limit Values and Biological Exposure Indices, ACGIH).⁷

5.3 This procedure has been adapted from NIOSH Method 5023 (withdrawn prior to 4th edition (1994) and replaced in 1998 with NIOSH Method 5042) and OSHA Method 58 to reduce the level of background contamination providing better reproducibility.

6. Interferences

6.1 All materials (not just asphalt fume) collected that are soluble in benzene will be included in the benzene soluble fraction weight.

6.2 Changes in temperature or humidity during pre- and post-collection weighing may affect accuracy.

7. Apparatus

7.1 Sampling Apparatus:

7.1.1 *Filter Sampling Cassette*—A sampling cassette is a two-piece opaque filter cassette containing a 37 mm pure PTFE filter with 2 µm pore size seated upon a 37 mm PTFE spacer ring. PTFE filters should be preweighed (see 11.2.2) and the cassette labeled prior to use. A sampler designed to collect the inhalable fraction of airborne particles (see Guide D6062) can be used when the exposure limits apply (for example, ACGIH TLV, see 5.2), to the inhalable fraction.

7.1.1.1 To accurately determine low levels of asphalt fumes, a low background PTFE filter is required. Filter blank values should be checked prior to use. A minimum of three filters from each lot of filters should be analyzed for benzene soluble contamination (see 11.2.3) prior to use.

NOTE 2—Only filters with average background contamination below 0.06 mg should be used.

⁷ ACGIH Threshold Limit Values and Biological Exposure Indices, available from American Conference of Governmental Industrial Hygienists, Inc. (ACGIH), 1330 Kemper Meadow Dr., Cincinnati, OH 45240, www.acgih.org, updated annually.

7.1.1.2 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 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.

7.1.2 *Personal Sampling Pumps*, portable, battery-operated, capable of maintaining a flow rate between 0.5 L/min and 2 L/min through the 2 µm PTFE filter for a period of 8 h.

7.1.3 *Flow Measuring Device*, used in accordance with Practice D5337.

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

7.2 Analytical Equipment:

7.2.1 *Balance*, electronic capable of measuring 0.01 mg.

7.2.2 *Polonium Antistatic Strip*.

7.2.3 *Desiccator*.

7.2.4 *Tube Heater Block*.

7.2.5 *Concentrator Tubes*, 10 mL.

7.2.6 *Gas Sparge Manifold*, for nitrogen blow down of extracts.

7.2.7 *Vacuum Oven*.

7.2.8 *Filtration Unit*, containing disposable 0.5 µm PTFE filter and syringe fitting.

7.2.9 *Aluminum Weighing Cups*, disposable or reusable weighing cups capable of holding at least 1 mL of benzene without leakage.

NOTE 3—PTFE may be used if appropriate aluminum cups cannot be obtained.

7.2.9.1 If reusable weighing cups are used, appropriate cleaning procedures must be employed to ensure no cross contamination from sample to sample.

7.2.10 *Pasteur Pipes*, disposable, glass, or equivalent.

7.2.11 *Miscellaneous Borosilicate Glassware (Pipets, Syringes, and so forth)*—All pipets and syringes shall be calibrated Class A volumetric glassware.

8. Reagents

8.1 *Benzene*—HPLC grade or equivalent with evaporation residue of <0.0005 %.

8.1.1 To determine benzene evaporation residue, perform the following:

8.1.1.1 Add 6.5 mL of benzene to a concentrator tube, place the tube in the heater block with a gentle flow of nitrogen.

8.1.1.2 Evaporate the benzene to approximately 0.5 mL, then transfer the concentrate with 3 small rinses of approximately 0.2, 0.2, and 0.1 mL of benzene using a precleaned Pasteur pipet to a preweighed weighing boat. Let stand in a laboratory fume hood for 1 h.

8.1.1.3 Transfer the weighing boat to a vacuum oven. The vacuum oven should be vented into a fume hood to prevent release of benzene vapors into the work area.

8.1.1.4 Allow the sample to further evaporate in the vacuum oven at ambient temperature and 20 mm Hg to 25 mm Hg vacuum for 24 h (or at least overnight). Slowly release vacuum.

Remove weighing boats and reweigh. If the evaporation residue is 0.03 mg or greater, do not use the benzene.

8.2 *Dichloromethane*—HPLC grade.

9. Safety Precautions

9.1 Benzene is a known carcinogen and flammable. Dichloromethane is a known animal carcinogen. Dichloromethane can cause nausea and central nervous system effects. Dermal contact can cause irritation and burns. Handling of benzene and dichloromethane solutions should be done in an appropriate fume hood.

9.2 Avoid inhalation of, or skin contact with benzene, asphalt extracts, and dichloromethane. Impervious gloves should be worn to minimize the potential for skin contact. See safety data sheets (SDS) for additional hazard and handling information.

10. Calibration

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

NOTE 4—Use of a standard 100 mg weight is recommended.

11. Procedure

11.1 *Air Sampling*—Refer to Practice **D1357** for general information on sampling.

11.1.1 Adjust the personal sampling pumps to the recommended flow rate with an assembled cassette between the pump and the flow-measuring device in accordance to Practice **D5337**. After the sampling, perform a post sampling flow rate verification. Pre- and post-sampling flow rates are expected to be within $\pm 5\%$ of a nominal value, in which case the nominal value can be used, or within $\pm 5\%$ of each other, when the average value can be used. If the difference between the two measurements is greater than $\pm 5\%$ 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.

11.1.2 Remove end plugs from sampling cassette. Place an identifying label on the cassette.

11.1.3 Attach the sampling device to a sample pump that has been adjusted at the recommended flow rate using flexible tubing. Place the back of the sampling device closest to the pump.

11.1.4 For a breathing zone sample, fasten the sampling pump to the worker’s clothing and attach the inlet of the sampling device as close as possible to the worker’s breathing zone. (**Warning**—Ensure that the presence of the sampling equipment is not a safety hazard to the worker.)

11.1.5 Turn on the pump that has previously been adjusted in **11.1.1**. Record the start time, end time, sampling location, sampling rate, pump number, and other pertinent sample information.

NOTE 5—Typically samples are collected for 8 h at 2 L/min.

11.1.6 Check the pump periodically to ensure that it is still running and within acceptable limits.

11.1.7 At the end of the sampling period, determine and record the flow rate, or pump register reading, if appropriate, turn the pump off, and note and record the time, temperature, and barometric pressure.

NOTE 6—(Minimum recommended sample volume is 15 L.)

11.1.8 Disconnect the sampling device from the sampling pump. Plug the inlet and outlet of the filter cassette using supplied plastic plugs.

11.1.9 Blank filter cassettes should be taken into the field and opened along with each sample set. These field blanks should be immediately resealed and shipped with the sample set. No air should be drawn through the field blanks.

11.1.10 Ship samples to the laboratory as soon as possible for analysis.

11.1.11 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.

11.2 Analysis:

11.2.1 Prerinse all reusable and disposable glassware with dichloromethane before use to minimize any potential contamination or contribution to BSF weight.

11.2.2 Total Mass Determination:

11.2.2.1 Remove the end plugs from the filter cassettes and desiccate for 12 h to 24 h.

11.2.2.2 Remove the filter from the cassette with forceps (taking care not to touch the filtration area). Place the filter on a polonium antistatic strip for several seconds, then weigh on the electronic balance.

NOTE 7—An equilibration time of approximately 2 min should be adequate to obtain a stable weight reading for filters and the weighing cups used in the benzene soluble procedure.

11.2.2.3 Record the gross mass in milligrams to the nearest hundredth of a milligram (that is, 0.01 mg).

11.2.3 Benzene Soluble Fraction Analysis:

11.2.3.1 With forceps, place the filter in an appropriately labeled 7 mL vial with a PTFE lined cap. Add 5 mL of benzene, cap the vial, and sonicate for at least 1 h.

NOTE 8—Benzene must be HPLC grade with <0.0005 % residue on evaporation.

11.2.3.2 Calibrate the electronic balance per manufacturer’s recommendations.

11.2.3.3 Preweigh aluminum weighing cups on an electronic balance or equivalent. Record the preweight.

11.2.3.4 Precondition the filtration unit immediately before use by filtering with nitrogen pressure ~ 1 mL of benzene through the unit attached to a 5 mL or 10 mL syringe with needle fitting. The benzene wash should be blown out of the filter with nitrogen but it is not necessary to blow the filter dry. **DISCARD THE WASH.** It is not part of the sample extract.

11.2.3.5 Using a clean Pasteur pipet, transfer the benzene extract as indicated in **11.2.3.1** to a prerinsed 5 mL or 10 mL glass syringe with the disposable filter attached. Use nitrogen (at pressure of approximately 48 kPa to 69 kPa or 7 psi to 10