

Designation: D 6494 - 99

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 D 6494; 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- 1.2 This procedure has been adapted from NIOSH Method 5023 (withdrawn prior to 4th edition (1994) and replaced in 1998 with NIOSH Method 5042) and OSHA Method 58 to reduce the level of background contamination providing better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 personnel 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.
- 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. For more specific precautionary statements, see Section 9.

2. Referenced Documents

- 2.1 ASTM Standards:
- D 1356 Terminology Relating to Sampling and Analysis of Atmospheres²
- 2.2 Occupational Safety and Health Administration (OSHA) Methods³

OSHA 58 Coal Tar Pitch Volatiles

2.3 National Institute of Occupational Safety and Health (NIOSH) Methods⁴

NIOSH 5023 Coal Tar Pitch Volatiles

¹ This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Committee D22.04 on Workplace Atmospheres.

NIOSH 5042 Benzene —Soluble Fraction and Total Particulate (Asphalt Fume)

3. Terminology

- 3.1 Definitions:
- 3.1.1 asphalt fume particulate matter—particulate matter generated during the processing of hot asphalt.
- 3.1.2 For definitions of terms relating to this test method, refer to Terminology D 1356.

4. Summary of Test Method

- 4.1 A known volume of sample air is passed through a PTFE filter. 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.
- 5.2.1 The threshold limit value (TLV) for asphalt (petroleum) fumes is 5 mg/m³ 8-h TWA, (1998 Threshold Limit Values and Biological Exposure Indices, ACGIH⁵).
- 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.

² Annual Book of ASTM Standards, Vol 11.03.

³ Available from Occupational Safety and Health Administration, Salt Lake Technical Ctr., 1781 S. 300 W., Salt Lake City, UT 84115.

⁴ Available from National Institute of Occupational Safety and Health, Cincinnati, OH.

⁵ Available from American Conference of Government Industrial Hygienists, 1014 Broadway, Cincinnati, OH 45202.



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 μ m 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.
- 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 1—Only filters with average background contamination below $0.06\ \mathrm{mg}$ should be used.

- 7.1.2 *Personal Sampling Pumps*,, portablebattery operated, equipped with a flow-monitoring device (rotameter, critical orifice) or a constant-flow device and capable of drawing 2 L/min of air through the 2-µm PTFE filter for a period of 8 h.
 - 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 2—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 %.

Note 3—Benzene evaporation residue should be checked by evaporating 6.5 mL of benzene (see 8.1.1). Benzene with evaporation residue of greater than 0.03 mg for the 6.5 mL is unacceptable.

8.1.1 Add the benzene to a concentrator tube, place the tube in the heater block with a gentle flow of nitrogen. 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. Transfer the weighing boat to a vacuum oven. Allow the sample to further evaporate in the vacuum oven at ambient temperature and 20 to 25 mm Hg vacuum for 24 h (or at least

overnight). Slowly release vacuum. Remove weighing boats and reweigh. The vacuum oven should be vented into a fume hood to prevent release of benzene vapors into the work area.

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 methylene chloride solutions should be done in an appropriate fume hood.
- 9.2 Avoid inhalation of, or skin contact with benzene, asphalt extracts and methylene chloride. Impervious gloves should be worn to minimize the potential for skin contact. See MSDS for additional hazard and handling information.

10. Calibration

10.1 Air Sampling Pumps—All air sampling pumps must be calibrated before use. For an accurate calibration, attach filters to be used for collecting the samples to the pump prior to the calibration. Calibration may be effected by drawing air through a calibrated rotameter or through a soap bubble flowmeter. Pumps should be calibrated at a flow rate of 0.5 to 2.0 L/min.

10.2 *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:
- 11.1.1 Remove end plugs from opaque sampling cassette. Place an identifying label on the cassette.
- 11.1.2 Attach the sampling device to a sample pump that has been calibrated at 0.5 to 2.0 L/min using rubber or plastic tubing. Place the back of the sampling device closest to the pump.
- 11.1.3 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.)
- 11.1.4 Turn on the pump that has previously been adjusted and calibrated in 10.1. 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.
- 11.1.5 Check the pump periodically to ensure that it is still running and within acceptable limits.
- 11.1.6 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.)

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