



# Standard Test Method for Determination of Asphalt Fume Particulate Matter in Workplace Atmospheres as Benzene Soluble Fraction<sup>1</sup>

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 ( $\epsilon$ ) 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1.2 This procedure has been adapted from NIOSH Method 5023 (withdrawn prior to 4th edition (1994) and replaced in 1998 with NIOSH Method 5042) and OSHA Method 58 to reduce the level of background contamination providing better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 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/m<sup>3</sup>.

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 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:<sup>2</sup>

<sup>1</sup> 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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<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.

### D1356 Terminology Relating to Sampling and Analysis of Atmospheres

#### 2.2 Occupational Safety and Health Administration (OSHA) Methods:<sup>3</sup>

##### OSHA Method 58 Coal Tar Pitch Volatiles

#### 2.3 National Institute for Occupational Safety and Health (NIOSH) Manual of Analytical Methods:<sup>4</sup>

##### NIOSH Method 5023 Coal Tar Pitch Volatiles

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

## 3. Terminology

3.1 *Definitions*—For definitions of terms relating to this test method, refer to Terminology D1356.

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. 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/m<sup>3</sup> 8-h TWA, (1998 Threshold Limit Values and Biological Exposure Indices, ACGIH<sup>5</sup>).

<sup>3</sup> Available from Occupational Safety and Health Administration (OSHA), 200 Constitution Ave., NW, Washington, DC 20210, <http://www.osha.gov>.

<sup>4</sup> Available from National Institute for Occupational Safety and Health (NIOSH), Cincinnati, OH. <http://www.cdc.gov>

<sup>5</sup> Available from American Conference of Governmental Industrial Hygienists, Inc. (ACGIH), 1330 Kemper Meadow Dr., Cincinnati, OH 45240, <http://www.acgih.org>.

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.

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 mg should be used.

7.1.2 *Personal Sampling Pumps*, portable, battery-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.

11.1.8 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.

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

## 11.2 Analysis:

11.2.1 Prerinse all reusable and disposable glassware with methylene chloride 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 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 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 or 10 mL glass syringe with the disposable filter attached. Use nitrogen (at

pressure of approximately 48 to 69 kPa or 7 to 10 psi) to force extract through filter into a 10 mL concentrator tube. All glassware should be cleaned with dichloromethane prior to use.

11.2.3.6 Rinse the 7 mL vial immediately after the sample transfer of the extract with two aliquots of approximately 0.5 mL of benzene and filter into the 10 mL concentrator tube.

11.2.3.7 Using a clean Pasteur pipe, rinse the syringe with approximately 0.5 mL of benzene making sure to include the entire inner wall area of the syringe. Filter into the 10 mL tube.

NOTE 9—Change the disposable filter for each new sample.

11.2.3.8 Place the concentrator tube in a heater block at 85°C and reduce volume to approximately 0.5 mL using gentle stream of nitrogen. Exercise extreme care during this evaporation step to avoid blowing the sample to dryness. If the sample evaporates to dryness, the sample should be voided.

11.2.3.9 Transfer the extract, with three small rinses of approximately 0.2, 0.2, and 0.1 mL of benzene using a precleaned Pasteur pipet from the concentrator tube to a preweighed aluminum or PTFE cup. The final 1-mL extract should be allowed to air dry for 1 h in a clean fume hood before transferring it to a vacuum oven.

11.2.3.10 Place the sample cups (in a numbered vial rack) in the vacuum oven at ambient temperature and 20 to 25 mm Hg vacuum for 24 h (or at least overnight). Sample cups must be protected from particulate contamination during this period.

NOTE 10—Vacuum oven should be vented into a fume hood to prevent release of benzene vapors into the work area.

11.2.3.11 Weigh the cups using the procedure outlined in 11.2.3.2 and 11.2.3.3 and record the final BSF mass.

11.2.3.12 Analyze a blank filter with each sample set in the same manner as a sample starting with 11.2.3.1 to determine the contribution of the filter and benzene residue.

## 12. Calculation

12.1 *Blank Corrected Total Mass*—Subtract the sample filter premass from the sample filter postmass. Also subtract any blank mass (blank filter postmass minus blank filter premass).

$$\text{Total Mass} = (A_{\text{sample}} - B_{\text{sample}}) - (A_{\text{blank}} - B_{\text{blank}}) \quad (1)$$

where:

$A_{\text{sample}}$  = mass of sample filter after sample collection, mg,  
 $B_{\text{sample}}$  = initial mass of sample filter, mg,  
 $A_{\text{blank}}$  = postmass of field blank, mg, and  
 $B_{\text{blank}}$  = initial mass of field blank, mg.

12.2 *Sample Benzene Soluble Fraction Mass*—Subtract the mass of the aluminum cup from the mass of the aluminum cup after evaporation of the extracted sample benzene residue (11.2.3.11):

$$BSF_{\text{sample}} = C_{\text{sample}} - D_{\text{sample}} \quad (2)$$

where:

$C_{\text{sample}}$  = mass of sample cup after extract evaporation, mg, and  
 $D_{\text{sample}}$  = initial mass of sample cup in mg.

12.3 *Blank Benzene Soluble Mass*—Subtract the mass of the aluminum cup from the mass of the aluminum cup after evaporation of the extracted blank benzene residue:

$$BSF_{\text{blank}} = C_{\text{blank}} - D_{\text{blank}} \quad (3)$$

where:

$C_{\text{blank}}$  = mass of blank cup in mg after extract evaporation, and

$D_{\text{blank}}$  = initial mass of blank cup, mg.

**12.4 Blank Corrected Benzene Soluble Fraction Mass**—Subtract the blank mass (12.2) from the sample mass (12.1).

$$BSF_{\text{corrected}} = BSF_{\text{sample}} - BSF_{\text{blank}} \quad (4)$$

**12.5 Airborne Benzene Soluble Fraction (in mg/m<sup>3</sup>)**—Divide the mg BSF by the air volume drawn through the sample in metres<sup>3</sup> (or equivalently multiply the mg BSF by 1000 then divide by the air volume in litres). The results are in mg/m<sup>3</sup>.

$$BSF_{\text{mg/m}^3} = \frac{BSF_{\text{corrected}}}{E_{\text{sample}}} \times \frac{1000 \text{ L}}{\text{m}^3} \quad (5)$$

where:

$E_{\text{sample}}$  = sample air volume, L.

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

**13.1 Repeatability**—Two representative asphalt samples were extracted with benzene. These benzene extracts were used to generate three asphalt extract levels for each asphalt type (0.244, 1.220, 2.440 mg Asphalt A/sample; 0.216, 1.080, and 2.160 mg Asphalt B/sample). Each level was analyzed in triplicate by six participating laboratories. The single operator relative standard deviation (RSD) varied from 3.35 to 12.6 % with an average of 6.55 %.

**13.2 Bias**—The average test method bias ranged from +2.02 to –16.27 % with an average bias of –3.24 % for the six laboratories that participated in the round robin study.

### 14. Keywords

14.1 air monitoring; asphalt fume; benzene soluble fraction; sampling and analysis; workplace atmospheres

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<sup>6</sup> Report to the Asphalt Institute, Summary Report for the Benzene Solubles Round Robin, 97TP56. Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D22-1029. Contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org).

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