
Standard Method of Test for

Spot Test of Asphaltic Materials

AASHTO Designation: T 102-09 (2013)

AASHTO

1. SCOPE

- 1.1. This method of test is applicable only to asphaltic products derived from petroleum and should not be applied to natural asphalts containing nonbituminous matter insoluble in xylene.
- 1.2. Materials, which by use of standard solvent are classed as positive, may be further tested to determine their degree of positiveness by means of their “xylene equivalent.” The xylene equivalent shall be the lowest percentage by volume of xylene in a solvent composed of xylene and standard naphtha or xylene and normal heptane, as specified, that produces a “negative spot” for the material in question. These shall be known as the “naphtha-xylene equivalent” and “heptane-xylene equivalent,” respectively. The percentage of xylene in the solvents shall be stated in even 5.0 percent increments. When neither xylene equivalent is specified, the standard naphtha only shall be used as the solvent.
- 1.3. The values stated in SI units are to be regarded as the 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. *AASHTO Standards:*
 - M 81, Cutback Asphalt (Rapid-Curing Type)
 - M 82, Cutback Asphalt (Medium-Curing Type)
 - M 226, Viscosity-Graded Asphalt Cement
 - M 231, Weighing Devices Used in the Testing of Materials
 - R 66, Sampling Asphalt Materials
 - T 78, Distillation of Cutback Asphalt Products
- 2.2. *ASTM Standards:*
 - D86, Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure
 - D611, Standard Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents
 - D1015, Standard Test Method for Freezing Points of High-Purity Hydrocarbons
 - E1, Standard Specification for ASTM Liquid-in-Glass Thermometers

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3. SIGNIFICANCE

- 3.1. The spot test is used to determine if an asphaltic material has been overheated during processing. A “positive” result indicates that an asphaltic material has been overheated. A “negative” result indicates that the materials have not been overheated.
- 3.2. The spot test has been used as an indicator of the compatibility of the components of an asphalt binder, with a negative spot indicating good compatibility and a positive spot indicating poor compatibility.
- 3.3. The spot test is an optional test for asphaltic materials as specified in M 81, M 82, and M 226.

4. APPARATUS

- 4.1. *Flask*—50-mL capacity, either Florence boiling flask or wide-mouth flat-bottom Soxhlet extraction flask approximately 45 mm (1³/₄ in.) in diameter by 60 mm (2³/₈ in.) high.
- 4.2. *Stopper*—cork or rubber stopper for flask provided with a 200-mm (8-in.) length of 6.4-mm (1/4-in.) glass tubing.
- 4.3. *Filter Paper*—Whatman No. 50. (The 70-mm size is sufficient.)
- 4.4. *Glass Plate*—A smooth, clear glass plate shall first be cleaned with trichloroethylene, then washed with soap and water, wiped dry, cleaned with a suitable glass-cleaning preparation, and wiped dry and free from dust and lint. After this cleaning treatment, a drop of the asphalt mixture when applied to the glass should flow out evenly to form a smoothly bounded elliptical stain. If the stain is jagged and uneven in outline, the glass should again be cleaned with a suitable glass-cleaning liquid until a drop of the asphalt mixture flows out as described.
- 4.5. Pipette or burette with 0.1-mL graduations.
- 4.6. *Thermometer*—ASTM Precision Thermometer 64C (64F) conforming to requirements of ASTM E1.
- 4.7. *Balance*—balance having sufficient capacity and conforming to M 231, Class G 1.
- 4.8. *Water Bath*—maintained at temperature of 32.0° ± 0.5°C (89.6° ± 1.0°F).

5. MATERIALS

- 5.1. The standard naphtha shall be a straight run overhead distillate free from cracked products of any kind and shall conform to the requirements as indicated in Table 1.

Table 1—Naphtha

Gravity A.P.I.	49–50
Distillation:	
Initial boiling point	Above 149°C (300°F)
50 percent over	168–179°C (335–355°F)
End point	Below 210°C (410°F)
Aniline number	59–63°C (138–145°F)

- 5.1.1. The aniline number of the solvent shall be determined according to ASTM D611.
Note 1—Naphtha conforming with these requirements may be obtained under the name of Skelly Solve “S” from Barton Solvents Inc., 201 S. Cedar, Valley Center, KS.
- 5.2. Xylene shall be chemically pure xylene showing a boiling range of 137 to 140°C (278.6 to 284°F) when distilled in accordance with ASTM D86.
- 5.3. Normal heptane shall conform to the requirements as indicated in Table 2 (Note 2).

Table 2—Heptane

ASTM Motor Octane Number	0.0 ± 0.2
Density at 20°C, g/mL	0.68375 ± 0.00015
Refractive index, ND at 20°C	1.38775 ± 0.00015
Freezing point, ^a °C	−90.72 minimum
Distillation:	
50 % recovery, °C	98.43 ± 0.05
Increase from 20 to 80 % recovery, °C	0.20 maximum

^a Determined by means of Standard Test Method for Freezing Points of High-Purity Hydrocarbons (ASTM D1015).

Note 2—Normal heptane meeting this specification may be obtained from the following sources:

- Enjay Co., Inc., 15 West 51st Street, New York, NY.
- Phillips Petroleum Co., Chemical Products Department, Bartlesville, OK.
- Westvaco Chlorine Products Corporation, 405 Lexington Avenue, New York, NY.
- Standard Oil Development Co., Linden, NJ.

6. SAMPLING

- 6.1. For asphalt cements, perform the test on the original material. For cutback asphalts, perform the test upon the residue from the distillation test T 78.

7. PROCEDURE

- 7.1. Place a 2.00- ± 0.02-g sample in the flask. If it does not flow readily at room temperature, carefully heat the flask until the sample can be spread in a thin film covering the bottom of the lower portion of the flask. Then allow the flask to cool to room temperature.
- 7.2. With the pipette or burette, place 10.2 mL of the specified solvent in the flask. Quickly insert the stopper with its 200-mm (8-in.) length of tubing into the neck of the flask, and swirl the flask with a rapid circular motion for 5 s. The flask shall then be immersed to its neck in a bath of gently boiling water for 55 s.
- 7.3. Remove the flask from the bath and swirl for 5 s. Each minute thereafter alternately immerse the flask for 55 s and remove and swirl for 5 s until complete dispersion has taken place.
- 7.4. After complete dispersion as judged by tilting the flask, lower the end of the glass tube below the level of the solution. Allow the flask to cool for 30 min at room temperature.

- 7.5. Warm the asphalt-solvent mixture for 15 min in a water bath maintained at $32.0 \pm 0.5^\circ\text{C}$ ($89.6 \pm 1.0^\circ\text{F}$). Thoroughly stir the asphalt-solvent mixture. By means of a clean stirring rod, place a drop of the warm mixture on the Whatman No. 50 filter paper. After 5 min, examine the spot by holding the paper at arm's length with the plane of the paper at approximately a right angle to the line of vision with a good light source (preferably diffused daylight) at the observer's back. If the drop forms a brown or yellowish-brown circular stain, with a darker solid or annular nucleus in the center, report the test as "positive."
- 7.6. If the drop forms a uniformly brown circular stain, set the asphalt-solvent mixture aside in its tightly stoppered flask at room temperature in a subdued light to be retested 24 h after the first examination. Warm the mixture to $32.0 \pm 0.5^\circ\text{C}$ ($89.6 \pm 1.0^\circ\text{F}$) for 15 min as before. Vigorously stir until uniform. Place a drop of the asphalt-solvent mixture on filter paper. If the drop from the 24-h-old mixture still forms a uniformly brown circular stain, report the test as "negative"; but if a darker solid or annular nucleus, as described in Section 7.5, now forms in the center of the stain, report the test as "positive."

8. PROCEDURE IN DISPUTED CASES

- 8.1. In case of dispute, repeat the entire test. Make up any loss in mass of solvent during dispersion by adding additional solvent, and after dispersion is complete, keep the flask in subdued light at $25.0^\circ \pm 1.7^\circ\text{C}$ ($77^\circ \pm 3^\circ\text{F}$) until the 24-h tests are made. Warm the asphalt-solvent mixture for 15 min to $32.0 \pm 0.5^\circ\text{C}$ ($89.6 \pm 1^\circ\text{F}$), then place a drop of the mixture on a filter paper. If the appearance of the drop on the filter paper made with the 24-h-old mixture is still in dispute, run the test on the glass plate as specified below.
- 8.2. Place a drop of the 24-h-old mixture on the glass plate held at an angle of 45 degrees with the horizontal. If, as the drop flows out, a dull matted streak develops in the center of its path, report the test as "positive."
- 8.3. If a drop of the 24-h-old mixture flows out to a uniform, clear, glossy brown film, without the central streak as described in the Section 8.2, report the test as "negative."

9. PROCEDURE FOR XYLENE EQUIVALENTS

- 9.1. The method used in testing for "xylene equivalents" is the same as specified in Section 7 except that the solvent shall be composed either of xylene and the standard naphtha or of xylene and normal heptane as specified.
- 9.2. To determine the xylene equivalent, disperse two or more samples of the asphalt to be examined in the prescribed mixed solvent. Vary the percentage of xylene by successive 5 percent (volume) increments until two asphalt solutions have been found, one of which shows a positive spot while the next (in which the solvent contains 5 percent more xylene) shows a negative spot. Report the xylene equivalent by the xylene percentage in the two solvents used in these two solutions; for example, as "10–15 percent naphtha-xylene equivalent" or "20–25 percent heptane-xylene equivalent."
- 9.3. When acceptance of material is based on a specified xylene equivalent, the exact lowest percentage of xylene that produces a "negative spot" need not be determined. The sample may be tested with solvent composed of the specified percentages of xylene and standard naphtha or xylene and normal heptane as required, and any material that shows negative for this solvent shall be reported as being less than the particular xylene equivalent that has been designated; for example, "less than 20 percent naphtha-xylene equivalent" or "less than 25 percent heptane-xylene equivalent."

10. REPORT

- 10.1. *This report shall include the following:*
- 10.1.1. Identification of sample;
 - 10.1.2. Solvent used in testing
 - 10.1.3. “Positive” or “Negative” result; and
 - 10.1.4. If performed, the xylene equivalent.

11. PRECISION AND BIAS

- 11.1. *Precision*—The research required to develop precision estimates has not been conducted.
- 11.2. *Bias*—This test method has no bias since the values determined can be defined only in terms of this test method.

12. KEYWORDS

- 12.1. Asphalt, spot test, xylene equivalent.