

*Standard Method of Test  
for*

## Spot Test of Asphaltic Materials

AASHTO DESIGNATION: T 102-83

### 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** The values stated in SI units are to be regarded as the standard.

**1.3** 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, which 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.

**NOTE 1**—Description of use of naphtha-xylene equivalent will be found in ASTM Proceedings, volume 36, part II, page 503.

### 2. MATERIALS

**2.1** 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 the table entitled "NAPHTHA".

	<u>NAPHTHA</u>
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° to 63°C (138° to 145°F)

**2.1.1** The aniline number of the solvent shall be determined as described in the ASTM Standard Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents, D 611-64.

**NOTE 2**—Naphtha conforming with these requirements may be obtained under the name of Skelly Solve "S" from the Skelly Oil Company, Kansas City, Missouri.

**2.2** Xylene used when xylene equivalents are specified shall be chemically pure xylene showing a boiling range of 137° to 140°C (278.6° to 284°F) when distilled in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosene, and Similar Petroleum Products (AASHTO T 115).

**2.3** Normal heptane shall conform to the requirements as indicated in the table entitled "Heptane" (Note 3).

	<u>Heptane</u>
ASTM Motor Octane Number	0.0 ± 0.2
Density at 20°C, g. per mL	0.68375 ± 0.00015
Refractive index, $n_D$ at 20°C	1.38775 ± 0.00015
Freezing point, <sup>a</sup> °C	-90.72 minimum
Distillation: <sup>b</sup>	
50-percent recovery, °C	98.43 ± 0.05
Increase from 20 to 80 percent recovery, °C	0.20 maximum

<sup>a</sup> Determined by means of Method of Test for Measurement of Freezing Points of High-Purity Compounds for Evaluation of Purity (ASTM D 1015).

<sup>b</sup> For equipment and methods used, see Research Paper RP 2079, Journal of Research,

National Bureau of Standards, Vol. 44, pp. 309 and 310.

**NOTE 3**—Normal heptane meeting this specification may be obtained from the following sources:

Enjay Company, Inc., 15 West 51st Street, New York, N.Y.

Phillips Petroleum Company, Chemical Products Department, Bartlesville, Oklahoma.

West Vaco Chlorine Products Company, 405 Lexington Avenue, New York 17, N.Y. Standard Oil Development Company, Linden, New Jersey.

### 3. APPARATUS

**3.1** The following apparatus is required:

**3.1.1** Flask, 50 mL capacity, either Florence pattern or wide mouth flat bottom Soxhlet pattern approximately 45 mm (1<sup>3</sup>/<sub>4</sub> in.) in diameter by 60 mm (2<sup>3</sup>/<sub>8</sub> in.) high.

**3.1.2** Cork stopper for flask provided with a 200 mm (8 in.) length of 6.4 mm (1/4 in.) glass tubing.

**3.1.3** Filter paper Whatman No. 50. (The 70 mm size is sufficient.)

**3.1.4** Glass plate—A smooth clear glass plate shall first be cleaned with benzene or carbon tetrachloride, 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 properly as just described.

**3.1.5** Pipette or Buret with 0.1 mL graduations.

**3.1.6** Thermometer—An ASTM Precision Thermometer 64C (64F) conforming to requirements of ASTM E 1.

**3.1.7** Balance—A balance conform-

ing to the requirements of AASHTO M 231, Class C.

#### 4. SAMPLES

4.1 For solid or semisolid asphalts the tests shall be conducted on the original material. For liquid asphalts of the slow-curing type having less than 15 percent by volume distilling below 360°C (680° F) by the Standard Method of Test for Distillation of Cutback Asphaltic Products (AASHTO T 78) the test may be made on the original material except in cases of dispute, when it shall be performed on the residue from the distillation test specified above. For other liquid asphalts the test in all cases shall be performed only upon the residue from the distillation test specified above.

#### 5. PROCEDURE

5.1 A  $2.00 \pm 0.02$  g sample shall be placed in the flask. If it does not flow readily at room temperature the flask shall be carefully heated until the sample can be spread in a thin film covering the bottom of the lower portion of the flask which shall then be allowed to cool to room temperature.

5.1.1 With the pipette or buret, place in the flask 10.2 mL of the specified solvent. Insert quickly into the neck of the flask the cork stopper with its 200 mm (8 in.) length of tubing and swirl the flask with a rapid circular motion for 5 seconds. The flask shall then be immersed to its neck in a bath of gently boiling water for 55 seconds (unless the sample is a thin liquid in which case heating is unnecessary).

5.1.2 The flask shall be removed from the bath and swirled for 5 seconds, and each minute thereafter the flask shall be alternately immersed for 55 seconds and removed and swirled for 5 seconds until complete dispersion has taken place.

5.2 After complete dispersion as judged by tilting the flask, the end of the glass tube shall be lowered below the level of the solution and the flask allowed to cool for 30 minutes at room temperature.

5.2.1 The asphalt solvent mixture shall then be warmed for 15 minutes in

a water bath maintained at temperature of  $32.0^\circ \pm 0.5^\circ\text{C}$  ( $89.6^\circ \pm 1.0^\circ\text{F}$ ). The asphalt solvent mixture shall be thoroughly stirred and by means of a clean stirring rod a drop of the warm mixture shall be placed on the Whatman No. 50 filter paper. After 5 minutes the spot shall be examined by holding the paper at arm's length with the plane of the paper approximately at right angles 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, the test shall be reported as positive.

5.3 If, however, the drop forms a uniformly brown circular stain, judgment shall be reserved and the asphalt solvent mixture shall be set aside in its tightly stoppered flask at room temperature in a subdued light, to be retested 24 hours after the first examination. The mixture warmed to  $32.0^\circ \pm 0.5^\circ\text{C}$  ( $89.6^\circ \pm 1.0^\circ\text{F}$ ) for 15 minutes as before shall then be vigorously stirred until uniform and a drop from it shall again be placed on the filter paper. If the drop from the 24 hours old mixture still forms a uniformly brown circular stain, the test shall be reported as "negative" but if a darker solid or annular nucleus, as described in the preceding paragraph now forms in the center of the stain, the test shall be reported as "positive."

#### 6. PROCEDURE IN DISPUTED CASES

6.1 In case of dispute the entire test shall be repeated. Any loss in mass of solvent during dispersion shall be made up by additional solvent and the flask after dispersion is completed shall be maintained in subdued light at a temperature of  $25.0^\circ \pm 1.7^\circ\text{C}$  ( $77^\circ \pm 3^\circ\text{F}$ ) until the 24 hours tests are made. A drop of the asphalt solvent mixture warmed for 15 minutes to  $32.0^\circ \pm 0.5^\circ\text{C}$  ( $89.6^\circ \pm 1^\circ\text{F}$ ) shall be placed on both the filter paper and on the glass plate. If the appearance of the drop on filter paper made with either freshly prepared or the 24-hour-old mixture is still in dispute then the test made on the glass plate in the manner specified below shall be final.

6.2 The drop of the 24-hour-old mixture shall be placed on the glass plate held at an angle of 45 degrees with the horizontal. If, as the drop flows out there develops in the center of its path a dull matted streak, the outline of which is well-defined against the smooth, clear, glossy brown outer zone when the glass is examined by reflected light against a dark background, the test shall be reported as positive.

6.3 If a drop of the 24-hour-old mixture flows out to a uniform, clear, glossy brown film, without the central streak as described in the foregoing paragraph, the test shall be reported as negative.

#### 7. XYLENE EQUIVALENTS

7.1 The method used in testing for "xylene equivalents" shall be the same as specified in the foregoing except that the solvent shall be composed either of xylene and the standard naphtha or of xylene and normal heptane as specified.

7.2 To determine the xylene equivalent two or more samples of the asphalt to be examined shall be dispersed in the prescribed mixed solvent, the percentage of xylene being made to vary 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. The xylene equivalent would then be reported 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" as the case may be.

7.3 When acceptance of material is based on a specified xylene equivalent, the exact lowest percentage of xylene which 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 which shows negative for this solvent shall be reported as being less than the particular xylene equivalent which has been designated, as for example, "less than 20 percent naphtha-xylene equivalent" or "less than 25 percent heptane-xylene equivalent."