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Standard Test Method for SOFTENING POINT OF BITUMEN IN ETHYLENE GLYCOL (RING-AND-BALL)¹

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1. Scope

1.1 This method covers the determination of the softening point of bitumen in the range from 30 to 110°C (85 to 230°F) using the ringand-ball apparatus in an ethylene glycol bath.

Note 1—For softening points above 110°C (230°F) use Method D 36, Test for Softening Point of Bitumen (Ring-and-Ball).

2. Applicable Documents

- 2.1 ASTM Standards.
- C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials²
- D36 Test for Softening Point of Bitumen (Ring-and-Ball)³
- D 92 Test for Flash and Fire Points by Cleveland Open Cup³
- D 140 Sampling Bituminous Materials⁴
- E 1 Specification for ASTM Thermometers⁵

3. Summary of Method

3.1 A steel ball of specified mass is placed upon a disk of bitumen contained within a horizontal, shouldered, metal ring of specified dimensions. The assembly is heated in an ethylene glycol bath at a uniform, prescribed rate and the softening point taken as the temperature at which the bitumen becomes soft enough to allow the ball, enveloped in the bitumen to fall a distance of 25 mm (1 in.).

4. Significance

4.1 Bitumens are viscoelastic materials without sharply defined melting points. Bitumens gradually become softer and less viscous as the temperature rises. For this reason, the determination of softening point must be made by an arbitrary and closely defined method if the results obtained are to be reproducible.

4.2 Softening point is useful in the classification of bitumen and is indicative of the tendency of the material to flow at elevated temperatures encountered in service.

5. Apparatus

5.1 Ring-A brass, shouldered ring conforming to the dimensions shown in Fig. 1 (a).

5.2 Ball—A steel ball, 9.5 mm (% in.) in diameter, and having a mass of 3.50 ± 0.05 g.

5.3 Ball-Centering Guide-A guide for centering the ball, constructed of brass and having the general shape and dimensions shown in Fig. 1 (c).

5.4 Ring Holder-The rings shall be supported on a brass ring holder, Fig. 1 (b), as shown in the assembly in Fig. 1 (d).

5.4.1 Rings, shall be supported in a horizontal position with the bottoms of the rings 25 mm (1 in.) above the upper surface of the bottom plate. The distance between the bottom plate and the bottom of the bath shall be 16 \pm 3 mm (% ± % in.).

5.4.2 The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the rings and within 13 mm (0.5 in.)

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² Annual Book of ASTM Standards, Parts 13, 14, 15, and 19. ³ Annual Book of ASTM Standards, Part 15. ⁴ Annual Book of ASTM Standards, Parts 15

Annual Book of ASTM Standards, Parts 15 and 19.

^{*} Annual Book of ASTM Standards, Parts 25 and 44.

of the rings but not touching them.

5.5 Brass Pouring Plate—A flat, smooth brass plate that has been treated to prevent the bituminous material from adhering to it.

NOTE 2—A suitable treatment is to coat the plate just before use with a thin layer of a mixture of glycerin and dextrin, talc, or china clay.

5.6 Bath—A glass vessel, capable of being heated, not less than 85 mm in diameter and not less than 120 mm in depth from the bottom of the flare.

NOTE 3-An 800-mL, low-form Griffin beaker of heat-resistant glass meets this requirement.

5.7 Thermometer—An ASTM Bituminous Material Softening Point Thermometer conforming to the requirements for Thermometer 113C (-1 to 175°C) or 113F (30 to 350°F) as prescribed in Specification E 1.

6. Reagents and Materials

6.1 Bath Liquid—Ethylene glycol with a boiling point between 195 and 197°C (383 and 387°F), or its equivalent.

NOTE 4—Caution—Ethylene glycol is toxic when taken internally or when inhaled as a vapor. Avoid prolonged or repeated contacts with skin and inhalation of vapors. Its flash point, in accordance with Method D 92, is 115°C (240°F).

7. Sampling

7.1 Sample the material in accordance with Methods D 140.

8. Test Specimens

8.1 Heat the sample with care to prevent local overheating, with frequent stirring until it has become sufficiently fluid to pour. In no case, however, shall the temperature be raised to more than 55° C (100°F) above the expected softening point for tar pitch, or to more than 110°C (200°F) above the expected softening point for asphalt. Avoid incorporating air bubbles in the sample.

8.2 Bring an asphalt sample to the pouring temperature in not more than 2 h. Bring coaltar pitch to the desired pouring conditions in minimum time, not exceeding 30 min.

Note 5—An electric hot plate having a minimum wattage-to-surface ratio of 37 kW/m^2 has been found satisfactory for this purpose.

8.3 Pour a slight excess of the heated sample into two rings, preheated to approximately the pouring temperature. While being filled, the rings shall rest on the brass pouring plate, previously treated to prevent bituminous materials from adhering to it.

8.4 Cool the specimen disks for a minimum of 30 min at room temperature. Cool specimens that are soft at room temperature for at least 30 min at a minimum of 10°C (18°F) below the expected softening point.

8.5 The cooling time shall be limited so that not more than 240 min elapse between the time of pouring specimens and the completion of the test.

8.6 After cooling, cut the excess material off cleanly with a slightly heated knife or spatula.

8.7 In case the test is repeated, use a clean container and fresh sample to prepare the test specimens.

9. Procedure

9.1 Conduct the test in a vented laboratory hood, or other well-ventilated location with sufficient air flow to ensure removal of toxic ethylene glycol vapors.

9.2 Assemble the apparatus with the rings, ASTM Thermometer 113C or 113F, and ballcentering guides in position and fill the bath with ethylene glycol to a depth of 105 ± 5 mm $(4\% \pm \% \text{ in.}).$

9.3 Maintain the bath temperature at $5 \pm 1^{\circ}C$ (41 $\pm 2^{\circ}F$) for 15 min, placing the test container in ice water if necessary. Using forceps, place a ball, previously adjusted to the bath temperature, in each ball-centering guide.

Note 6—Avoid any contamination of the ethylene glycol with water.

9.4 Apply heat in such a manner that the temperature of the liquid is raised $5^{\circ}C$ (9°F)/min. Avoid the effect of drafts, using shields if necessary.

NOTE 7—Rigid adherence to the prescribed rate of heating is absolutely essential for reproducibility of results. Either a gas burner or electric heater may be used; however, the latter must be of the low-lag, variable output type to maintain the necessary rate of heating.

9.5 The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any 1-min period after the first 3 min shall be $\pm 0.5^{\circ}$ C ($\pm 0.9^{\circ}$ F). Reject all tests in which the rate of rise does not fall within these limits.

9.6 Record for each ring and ball the tem-