

Standard Test Method for SOFTENING POINT BY RING-AND-BALL APPARATUS¹

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This test method has been approved for use by the agencies of the Department of Defense to replace Method 4495 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

¹ NOTE—Section 2 was added editorially and subsequent sections renumbered in June 1985.

1. Scope

1.1 This test method covers the determination of the softening point of resins (including rosin) and similar materials by means of the ring-and-ball apparatus.

NOTE 1—For testing asphalts, tars, and pitches, see Test Method D 2398.

2. Applicable Documents

2.1 ASTM Standards:

D 2398 Test Method for Softening Point of Bitumen in Ethylene Glycol (Ring and Ball)²

E 1 Specification for ASTM Thermometers³

3. Softening Point

3.1 In general, with materials of these types, softening does not take place at a definite temperature. As the temperature rises, these materials gradually and imperceptibly change from brittle or exceedingly thick and slow-flowing materials to softer and less viscous liquids. For this reason, the determination of the softening point must be made by a fixed, arbitrary, and closely defined method if the results obtained are to be comparable.

3.2 In this test method, the softening point is defined as the temperature at which a disk of the sample held within a horizontal ring is forced downward a distance of 2.54 cm (1 in.) under the weight of a steel ball as the sample is heated at a prescribed rate in a water or glycerin bath.

4. Apparatus

4.1 *Ring*—A brass-shouldered ring conform-

ing to the dimensions shown in Fig. 1 (a). If desired, the ring may be attached by brazing or other convenient manner to a brass wire of about 13 B & S gage 1.5 to 2.0 mm (0.06 to 0.08 in.) in diameter as shown in Fig. 2 (a). For use in the powder method of sample preparation, the ring may be made of steel to minimize the possibility of its deformation during the compacting operation.

4.2 *Ball*—A steel ball, 9.53 mm ($\frac{3}{8}$ in.) in diameter, weighing between 3.45 and 3.55 g.

4.3 *Ball-Centering Guide*—A guide for centering the ball, constructed of brass and having the general shape and dimensions illustrated in Fig. 1 (c), the use of which is optional.

4.4 *Container*—A glass vessel, capable of being heated, not less than 8.5 cm (3.34 in.) in diameter and not less than 12.7 cm (5 in.) in depth from the bottom of the flare. (An 800-mL low-form Griffin beaker of heat-resistant glass meets this requirement.)

4.5 *Support for Ring and Thermometer*—Any convenient method for supporting the ring and thermometer may be used, provided it meets the following requirements:

4.5.1 Ring shall be supported in a substantially horizontal position.

4.5.2 When using the apparatus shown in Fig.

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² Annual Book of ASTM Standards, Vol 04.04.

³ Annual Book of ASTM Standards, Vol 14.01.

(1.0 in.) above the horizontal plate below it; bottom surface of the horizontal plate shall be 12.7 to 19.05 mm (0.5 to 0.75 in.) above the bottom of the beaker; and depth of liquid in the beaker shall be not less than 10.16 cm (4.0 in.).

4.5.3 When using the apparatus shown in Fig. 2 (e): the bottom of the ring shall be 2.54 cm (1.0 in.) above the bottom of the beaker, with the bottom end of the rod resting on the bottom of the beaker; and depth of liquid in the beaker shall be not less than 10.2 cm (4.0 in.) (see Fig. 2 (a), (b), and (c)).

4.5.4 In both assemblies: Thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 1.27 cm (0.5 in.) but not touching the ring. For referee work, no more than two rings shall be used.

4.6 Thermometers:

4.6.1 An ASTM Low-Softening-Point Thermometer, having a range from -2 to 80°C , (30 to 180°F) and conforming to the requirements for thermometer 15C or 15F as prescribed in Specification E 1.

4.6.2 An ASTM High-Softening-Point Thermometer, having a range from 30 to 200°C , (85 to 392°F), and conforming to the requirements for Thermometer 16C or 16F as prescribed in Specification E 1.

4.7 *Mechanical Stirrer*—A two-bladed motor-driven stirrer attached to the bottom of a true vertical shaft may be used to ensure uniform heat distribution (see Fig. 2 (d) for dimensions). The direction of shaft rotation shall move the liquid upward. The speed shall be within the range from 500 to 700 R/min.

4.8 For the powder method of sample preparation the following additional apparatus is required:

4.8.1 *Mortar and Pestle*—A steel mortar and pestle with sleeve, knock-out-button, and ring support conforming to the dimensions shown in Fig. 3.

4.8.2 *Hammer*—A babbitt metal or a lead-impregnated rubber hammer weighing 0.9 kg (2 lb).

4.9 *Hydraulic Press*—A hydraulic press capable of maintaining sustained pressures up to 55 kPa (8000 psi), to be used alternatively with the hammer.

5. Preparation of Sample by Pour Method

5.1 *Rosins*—Select a sample representative of

of freshly broken lumps free of oxidized surfaces. For samples received as small lumps, scrape off the surface layer of the lumps immediately before using, avoiding inclusion of finely divided material or dust. Select a quantity at least twice that necessary to fill the desired number of rings, but in no case less than 40 g, and melt it immediately in a clean container, using an oven, hot plate, sand, or oil bath to prevent local overheating. Take care to avoid incorporating air bubbles in the sample, which must not be heated above the temperature necessary to pour the material readily without inclusion of air bubbles. The time from the beginning of heating to the pouring of the sample shall not exceed 15 min. Immediately before filling the rings, preheat them to approximately the temperature at which the material is to be poured. The rings, while being filled, should rest on an amalgamated brass plate. Pour the sample into the rings so as to leave an excess on cooling. After cooling a minimum of 30 min, cut the excess material off cleanly with a slightly heated knife or spatula. In case the test is repeated, use a clean container and fresh sample.

6. Preparation of Sample by Powder Method

6.1 *Resins (Except Rosin), and Other Materials that Cannot Be Melted and Poured Without Altering the Softening Point*—Select the sample as described in Section 5 and break up the lumps until there are no particles larger than 3.175 mm ($\frac{1}{8}$ in.). Mix the material thoroughly, and quarter down until a suitable quantity (approximately 50 to 75 g) is obtained for powdering. Pulverize the quartered sample in a porcelain mortar or by other suitable means, and fractionate by screening through Nos. 50 and 200 sieves. Immediately use the material passing the No. 50 sieve and retained on the No. 200 sieve, for preparation of the ring. Assemble the ring with the ring support, the mortar, and the knock-out-button, together with the sleeve, as shown in Fig. 3. Take care to ensure that the ring is properly centered and seated in the cut-out section of the sleeve. Pour the powdered material into the sleeve until it is about 1.27 cm (0.5 in.) above the top of the ring (approximately 3 g required). Place the pestle in the sleeve and compact the powder by rapping the pestle sharply 50 to 60 times with the leaded hammer, or by applying a pressure of 48 to 51 kPa (7000 to 7500 psi) in a suitable press, and holding this pressure for 3 to 5 min. Remove the

ring from the mortar and sleeve. An excess of material shall remain above the top surface of the ring. Carefully scrape this off, until the top of the sample is level with the ring. If the top and bottom surfaces of the sample in the ring are not smooth and level with the ring, discard, and repeat the compacting operation, using a clean ring and fresh powder.

7. Preparation of Sample by Molding Method

7.1 Resins (Except Rosin) and Other Materials that Are Heat-Sensitive and Cannot Be Melted and Poured Without Altering the Softening Point:

7.1.1 Selection of Sample—Select a sample representative of the material under test. The sample shall consist of freshly broken lumps free of oxidized surfaces. For samples received as small lumps, scrape off the surface layer of the lumps immediately before using, avoiding inclusion of finely divided material or dust, and then grind superficially in a mortar.

7.1.2 Apparatus:

7.1.2.1 Hot Plate, 3-heat, 20.32-cm (8-in.), 1000-W, with variable transformer, 7.5 A.

7.1.2.2 Aluminum or Steel Plates, 10.16 cm (4 in.) by 15.24 cm (6 in.) by 1.6 mm ($\frac{1}{16}$ in.).

7.1.2.3 Spatula, 20.32-cm (8-in.), stiff blade.

7.1.2.4 Tongs.

7.1.3 Procedure—Turn the hot plate to low heat and allow to come to temperature. Depending on the expected softening point of the resins, use the following approximate transformer settings:

Expected Softening Point	Transformer Setting
85 to 90°C	80
125 to 130°C	110

7.1.3.1 Place the shouldered ring, bottom down, on one end of the aluminum or steel plate, and then place this assembly on the hot plate. Place 10 to 15 g of the crushed resin on the metal plate so as to form a layer about 6.3 mm ($\frac{1}{4}$ in.) thick and 7.62 cm (3 in.) in diameter. The resin adjacent to the metal plate will quickly begin to soften. By means of the spatula, scrape and knead the entire mass until it is soft and plastic, using a second spatula if necessary (Note 2). Finally, gather the entire blob of resin on the spatula, remove the metal plate from the hot plate with a pair of tongs, and quickly press the resin into the the shouldered ring, applying pressure to the top

of the spatula. Allow the assembly to cool somewhat; then tap the plate and spatula to free the ring. Trim off the excess resin on the periphery of the ring. In order to remove excess resin from the top, grasp the ring in a pair of tongs and draw the top surface quickly and firmly over the surface of a heated metal plate.

NOTE 2—Not over 5 min should be required for this operation. Excessive exposure to elevated temperature will promote changes in the sample that result in high melting points.

8. Procedure for Materials Having Softening Points 80°C (176°F) or Below

8.1 Assembly of Apparatus—Fill the glass vessel, to a depth of not less than 10.16 cm (4.0 in.) and not more than 10.78 cm (4.25 in.) with freshly boiled distilled water at 5°C (41°F). For resins (including rosin), use water which has been cooled to not less than 45°C (81°F) below the anticipated softening point, but in no case lower than 5°C (41°F). Locate the axis of the stirrer shaft near the back wall of the beaker, with the blades clearing the wall and with the bottom of the blades 1.90 cm (0.75 in.) above the top of the ring. Unless the ball-centering guide is to be used, make a slight indentation in the center of the sample by pressing the ball or a rounded rod, slightly heated for hard materials, into the material at this point. Suspend the ring containing the sample in the water so that the lower surface of the filled ring is 2.54 cm (1.0 in.) above the upper surface of the lower horizontal plate (Fig. 1 (d)), which is at least 1.27 cm (0.5 in.) and not more than 1.90 cm (0.75 in.) above the bottom of the glass vessel, or 1.0 in. above the bottom of the glass container (Fig. 2 (e)). Place the ball in the water but not on the test specimen. Suspend an ASTM low-softening-point thermometer so that the bottom of its bulb is level with the bottom of the ring, and within 0.5 in. but not touching the ring. Maintain the initial temperature of the water for 15 min. With suitable forceps, place the ball in the center of the upper surface of the material in the ring. Start stirring, and continue the stirring at 500 to 700 r/min until completion of determination.

8.2 Heating—Apply heat in such a manner that the temperature of the water is raised 5°C (10°F)/min. Avoid the effect of drafts, using shields if necessary.

8.3 Permissible Variation in Rise of Temperature—The rate of rise of temperature shall be