



Standard Test Method for Softening Point of Certain Alkali-Soluble Resins¹

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1. Scope

1.1 This test method covers the determination of the softening point of certain alkali-soluble resins having uniform plastic flow characteristics as the melting point is approached.

1.2 The resin manufacturer should specify whether or not this test method may be used for his product(s).

1.3 This test method is not suitable for styrene-maleic anhydride resins.

NOTE 1—For testing rosin and other resins, see Test Method E 28.² For testing asphalts, tars, and pitches, see Test Method D 2398.

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 ASTM Standards:

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

E 1 Specification for ASTM Thermometers³

E 28 Test Method for Softening Point by Ring and Ball Apparatus⁴

3. Terminology

3.1 Definition:

3.1.1 *softening point*—the temperature at which a disk of the sample held within a horizontal ring is forced downward a distance of 1 in. (25.4 mm) under the weight of a steel ball as the sample is heated at a prescribed rate in a glycerin bath.

4. Significance and Use

4.1 In general, with materials of this type, softening does not take place at a definite temperature. As the temperature rises, these materials gradually and imperceptibly change from brittle solids to soft, viscous liquids. For this reason, the

determination of the softening point must be made by a fixed, arbitrary, and closely defined methods if the results are to be comparable.

5. Apparatus

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

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

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

5.4 *Container*—A glass vessel, capable of being heated, not less than 85 mm (3.34 in.) in diameter and not less than 127 mm (5 in.) in depth from the bottom of the flare. (An 800-mL, low-form Griffin beaker of heat-resistant glass meets this requirement.)

5.5 *Support for Ring and Thermometer*, as shown in Fig. 1(d). Note the following requirements:

5.5.1 The ring shall be supported in a horizontal position.

5.5.2 The bottom of the ring shall be 25.4 mm (1 in.) above the horizontal plate below it.

5.5.3 The bottom surface of the horizontal plate shall be at least 12.5 mm (0.5 in.) and not more than 19 mm (0.75 in.) above the bottom of the beaker.

5.5.4 The depth of liquid in the beaker shall be not less than 102 mm (4 in.).

5.5.5 The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 12.7 mm (0.5 in.) but not touching the ring.

5.6 *Thermometer*—An ASTM High Softening Point Thermometer, having a range from 30 to 200°C, and conforming to the requirements for Thermometer 16C as prescribed in Specification E 1.

5.7 *Mechanical Stirrer*—A variable-speed, motor-driven stirrer attached to the bottom of a true-vertical shaft must be used to ensure uniform heat distribution. The stirrer shall be positioned and its speed shall be regulated so that the fluid in the bath is completely but gently agitated, with no vortexing, turbulence, or air entrainment.

6. Precautions

6.1 Note the instructions concerning stirrer speed (5.7) and follow closely. Take care that the stirrer motor does not impart

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² *Annual Book of ASTM Standards*, Vol 04.04.

³ *Annual Book of ASTM Standards*, Vol 14.03.

⁴ *Annual Book of ASTM Standards*, Vol 06.03.

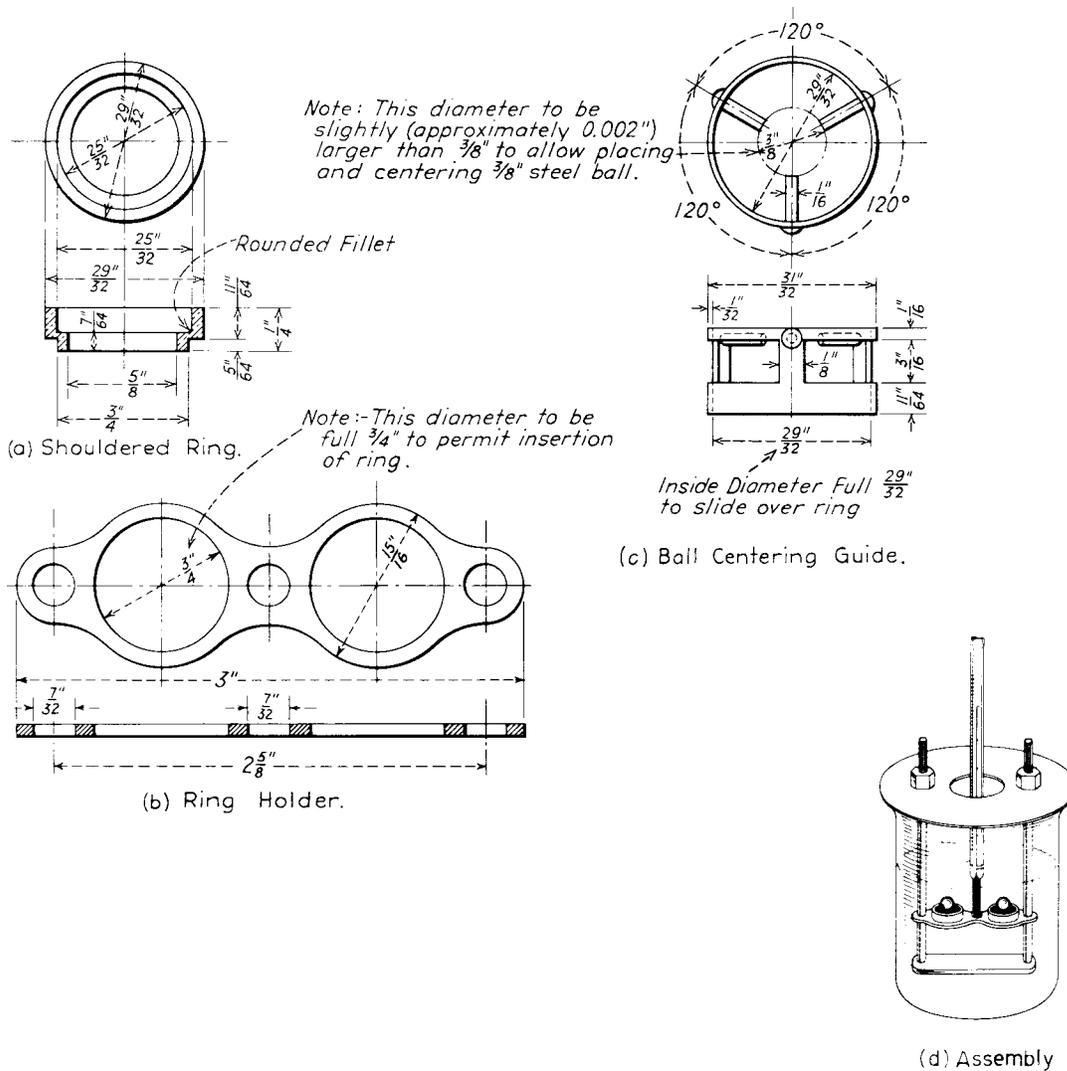


FIG. 1 Shouldered Ring, Ring Holder, Ball-Centering Guide, and Assembly of Apparatus Showing Two Rings

vibrations to the bath through the support system.

6.2 Previously boil the glycerin used in the bath in a fume hood.

6.3 Rigid adherence to the prescribed rate of heating (Section 8) is absolutely essential to the accuracy and reproducibility of this test method. Reject all tests in which the rate of increase exceeds the limits.

6.4 When the ball drops through the ring, it should be completely surrounded by softened resin and should drop straight down to the lower horizontal plate.

7. Preparation of Sample

7.1 Select a sample representative of the resin under test. Select a quantity at least twice that necessary to fill the ring and melt it in a clean container on a hot plate. Take care to avoid overheating the sample or incorporating air bubbles into it; in no case should the sample 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 should not exceed 15 min. Immediately before filling the rings, preheat them to approximately the temperature

at which the resin is to be poured. The rings, while being filled, should rest on a smooth metal plate. Pour the molten resin into the rings so as to leave an excess above the top surface on cooling. Allow the filled ring to cool only enough to permit handling the ring. Working quickly, smooth off the excess resin with a heated metal spatula so that the top surface of the ring is level and even. Trim off any resin that has adhered to the outer circumference of the ring. Examine the sample; if it shows any air bubbles or cracks, discard it and prepare another sample.

8. Preparation of Apparatus

8.1 Fill the glass vessel to a depth of 102 to 108 mm (4 to $4\frac{1}{4}$ in.) with boiled glycerin. The starting temperature of the glycerin shall be not less than 45°C below the anticipated softening point of the resin. Place the ring in the ring holder, cover it with a ball-centering guide, and gently place a ball in the center of the guide. Suspend the assembly in the bath and position the thermometer (in accordance with 5.5.5) and start the stirrer.

9. Procedure

9.1 Apply heat to the bath in such a manner that the temperature of the bath increases 1°C/11 to 12 s. This rate of heating is critical, it must be uniform, and should be checked at least once every 5°C and the heat source adjusted as necessary. Avoid the effects of drafts, shielding the apparatus if necessary. Record as the softening point the temperature of the thermometer at the instant the resin touches the lower horizontal plate. Make no correction for the emergent stem of the thermometer.

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10. Precision and Bias

10.1 *Precision*—Duplicate results by the same operator shall not be considered suspect unless they differ by more than $\pm 5^{\circ}\text{C}$.

10.2 *Bias*—This test has no bias because the values produced are defined only in terms of this test method.

11. Keywords

11.1 alkali soluble resins; melting point; polish; resins; ring and ball flow point; softening point