Designation: D3642 – 13 (Reapproved 2021)

Standard Test Method for Softening Point of Certain Alkali-Soluble Resins[1]

This standard is issued under the fixed designation D3642; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 E28. For testing asphalts, tars, and pitches, see Test Method D2398.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2398 Test Method for Softening Point of Bitumen in Ethylene Glycol (Ring-and-Ball) (Withdrawn 1984)[3]

E28 Test Methods for Softening Point of Resins Derived from Pine Chemicals and Hydrocarbons, by Ring-and-Ball Apparatus

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions:*

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 (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.

[1] This test method is under the jurisdiction of ASTM Committee D21 on Polishes and is the direct responsibility of Subcommittee D21.02 on Raw Materials.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

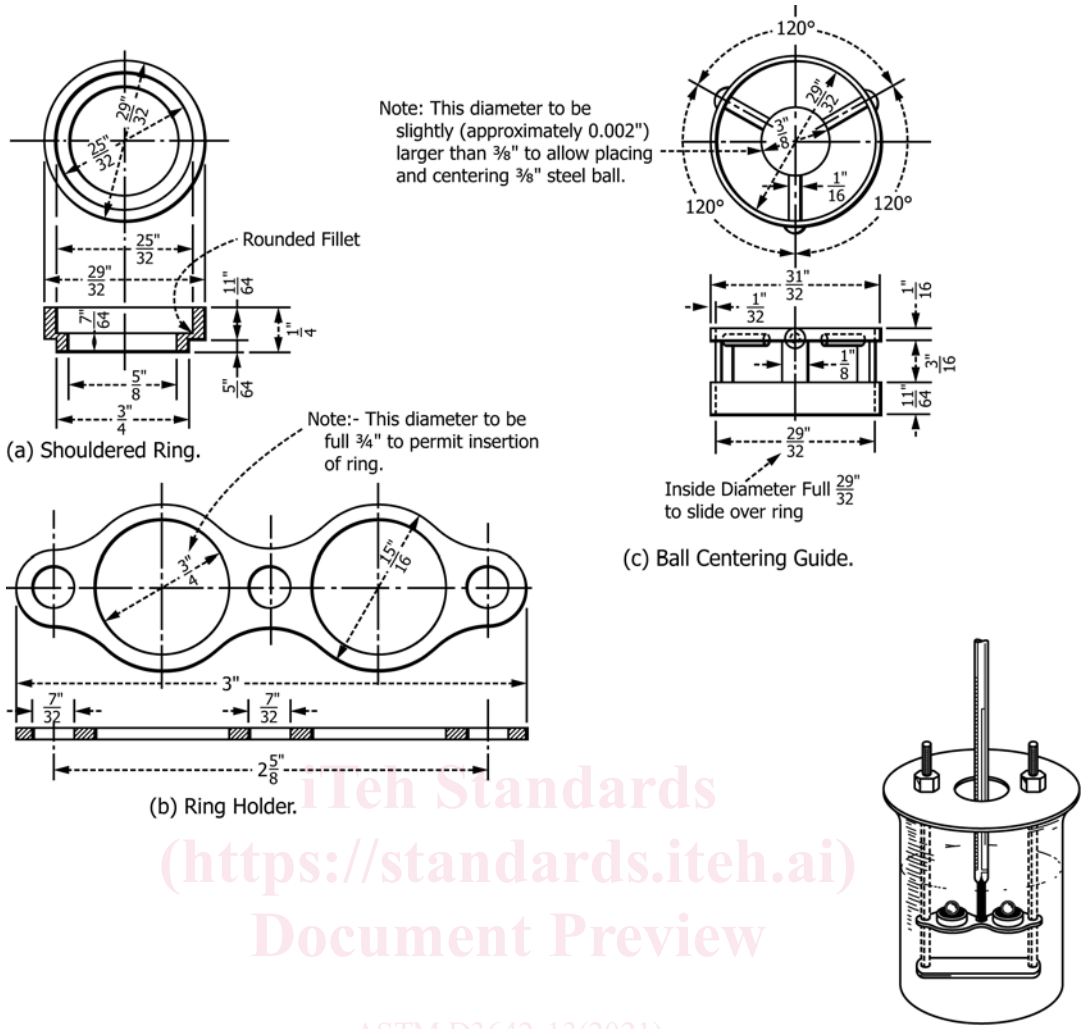

FIG. 1 Shouldered Ring, Ring Holder, Ball-Centering Guide, and Assembly of Apparatus Showing Two Rings

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 E2251.

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 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