



Designation: D 6493 – 99

Standard Test Methods for Softening Point of Hydrocarbon Resins by Automated Ring and Ball Apparatus¹

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1. Scope

1.1 These test methods are intended for determining the softening point of hydrocarbon resins and similar materials by means of an automated ring-and-ball apparatus.

1.1.1 The ring and ball softening point of a hydrocarbon resin may be run with lower precision using the manual ring and ball softening point in Test Method E 28.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard method 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:*

E 1 Specification for ASTM Thermometers²

E 28 Test Methods for Softening Point of Resins Derived from Naval Stores by Ring-and-Ball Apparatus³

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁴

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

3. Significance and Use

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 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 these test methods, 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 25.4 mm (1 in.) under the weight of a steel ball as the sample is heated at 5°C/min in a water, glycerin, silicone oil, ethylene glycol/water or glycerin/water bath.

4. Sample Preparation

4.1 *Preparation of Sample by the Pour Method:*

4.1.1 This procedure is suitable for materials that can be heated and poured without adverse effects on the softening point.

4.1.2 Select a sample representative of the material to be tested. The sample should consist of flakes, pastilles, or freshly broken lumps. Avoid inclusion of finely divided material or dust.

4.1.3 Select a quantity at least twice that necessary to fill the desired number of rings, and melt it in a clean container, using an oven, hot plate, sand bath or oil bath to prevent local overheating. Take care to avoid incorporating air bubbles in the sample. Melt the sample completely, but do not heat it above a temperature necessary to pour the material readily. The time from the beginning of heating to the pouring of the sample should not exceed 15 min.

4.1.4 For materials that tend to crack or shrink in the ring on cooling, immediately before filling the ring, preheat the ring to approximately the temperature at which the material is to be poured. The ring, while being filled, should rest **bottom down** on a suitable metal surface. Pour the sample into the ring so as to leave an excess on cooling. After cooling a minimum of 30 min, trim off the excess resin on the periphery of the ring. To remove excess resin from the top, cut the excess material off cleanly with a slightly heated knife or spatula, or grasp the ring in a pair of tongs and draw the top surface quickly and firmly over the surface of a heated metal plate. In case the test is repeated, use a clean container and fresh sample.

4.2 *Preparation of Sample by the Powder Method:*

4.2.1 See Appendix X1.1, Alternate Sample Preparation Procedures.

4.3 *Preparation of Samples Having a Low Softening Point (up to 35°C (95°F)):*

4.3.1 Place a ring on a piece of aluminum foil. Pour the material to be tested into the ring, then place the foil and the filled ring on dry ice or in a freezer to cool. The material in the ring must be free of bubbles.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications, and is the direct responsibility of Subcommittee D01.38 on Hydrocarbon Resins.

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² *Annual Book of ASTM Standards*, Vol 14.03.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

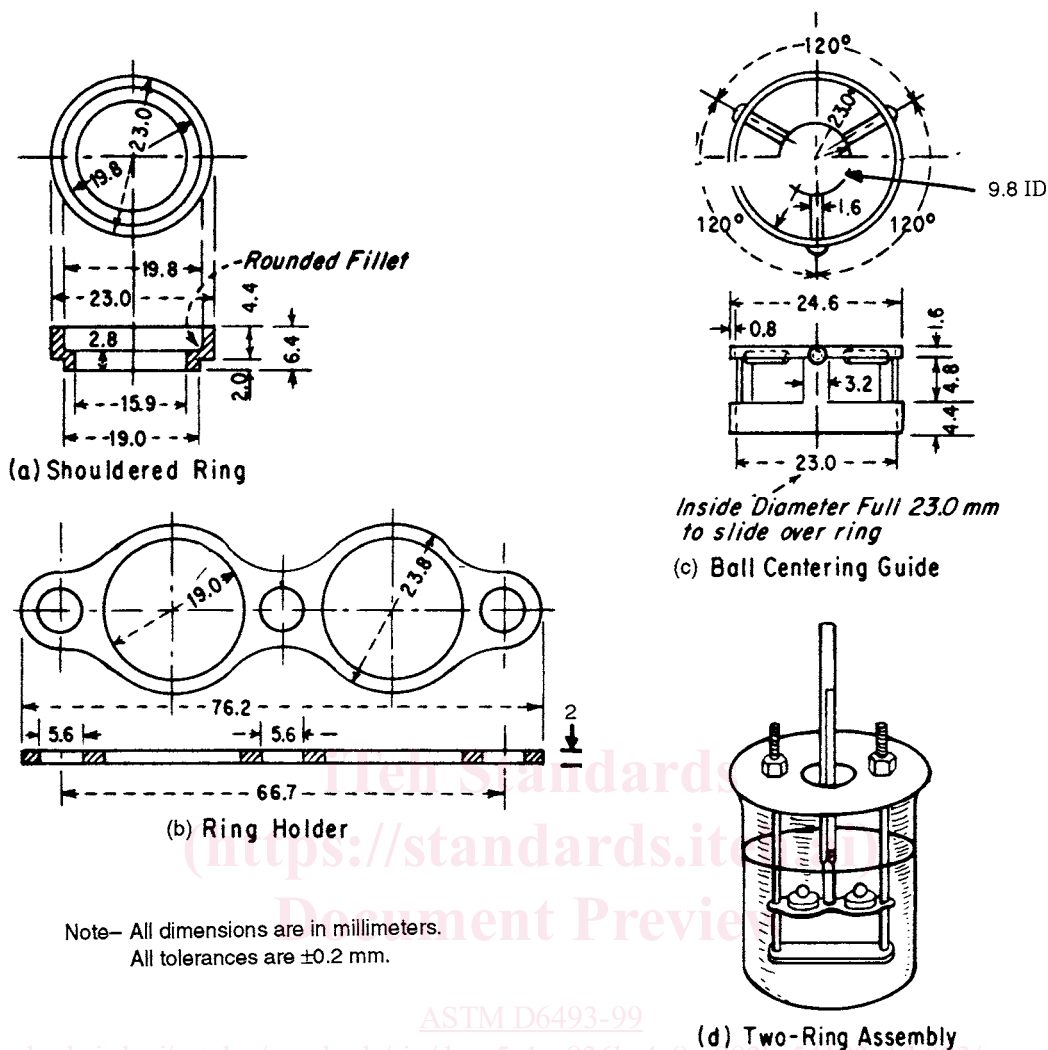


FIG. 1 Shouldered Ring, Ring Holder, Ball-Centering Guide, and Assembly of Apparatus Showing Two Rings

4.3.2 After cooling, cut and scrape off any excess material using a slightly heated spatula, then slide the ring gently from the foil. Place the ring in the supporting apparatus, and perform the softening point analysis according to Section 11.

5. Apparatus

5.1 *Automated Ring and Ball Softening Point Instrument with Control Unit*, test units, and test inserts.

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

5.3 *Ball*—A steel ball, 9.53 ± 0.1 mm ($\frac{3}{8}$ in.) in diameter, weighing between 3.45 and 3.55 g.

5.4 *Beaker*, 600 mL. Ensure that the dimensions will properly fit the heating unit.

5.5 *Stir Bar*—The dimensions must be such that the bar spins freely under the test stand.

6. Reagents and Materials

6.1 Bath Liquids:

6.1.1 *Distilled or Deionized Water, Freshly Boiled*—For softening points between 35°C (95°F) and 80°C (176°F).

6.1.1.1 Use distilled or deionized water that has been cooled to at least 27°C (81°F) below the anticipated softening point, but in no case lower than 5°C (41°F).

6.1.2 *USP Glycerin*—For softening points between 80°C (176°F) and 150°C (302°F). Repeated use of glycerin will increase the moisture content over time and may affect results. Replace with fresh glycerin if any change in appearance is noted.

NOTE 1—Glycerin should not be used for softening points greater than 150°C (302°F) due to the 160°C (320°F) flash point of glycerin.

6.1.3 *Silicone Oil (Polydimethylsiloxane)*—For softening points above 80°C (176°F). The oil must have a temperature range of 200°C+ (392°F+), remain clear within the temperature range, have no apparent effect or reactivity with the test specimen, have a high water repellency, and maintain a uniform viscosity and stirring rate within the temperature range.⁵

⁵ Supporting data are available from ASTM Headquarters. Request RR:D01-1113.