

GLYCEROL ESTERS OF ROSINS

Ring and ball softening point method

The ring-and-ball 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 1 in. (25.4 mm) under the weight of a steel ball as the sample is heated at a prescribed rate in a water or glycerol bath.

Apparatus

The apparatus illustrated in Figures 1 and 2 consists of the components described in the following paragraphs.

Ring

A brass-shouldered ring conforming to the dimensions shown in Figure 1a should be used. If desired, the ring may be attached by brazing or other convenient manner to a brass wire of about 13 B & S gauge (0.06 to 0.08 in., or 1.52 to 2.03 mm, in diameter) as shown in Figure 2a.

Ball

A steel ball, 3/8 in. (9.53 mm) in diameter, weighing between 3.45 and 3.55 g, should be used.

Ball-Centering Guide

A guide for centering the ball, constructed of brass and having the general shape and dimensions, as illustrated in Figure 1c may be used if desired.

Container

Use a heat-resistant glass vessel, such as an 800-ml low-form Griffin beaker, not less than 3.34 in. (8.5 cm) in diameter and not less than 5 in. (12.7 cm) in depth from the bottom of the flare.

Support for Ring and thermometer

Any convenient device for supporting the ring and thermometer may be used, provided that it meets the following requirements: (1) the ring shall be supported in a substantially horizontal position; (2) when using the apparatus shown in Figure 1d, the bottom of the ring shall be 1.0 in. (25.4 mm) above the horizontal plate below it, the bottom surface of the horizontal plate shall be at least 0.5 in. (13 mm) and not more than 0.75 in. (18 mm) above the bottom of the container, and the depth of the liquid in the container shall be not less than 4.0 in. (10.2 cm); (3) when using the apparatus shown in Figure 1e, the bottom of the ring shall be 1.0 in. (25.4 mm) above the bottom of the container, with the bottom end of the rod resting on the bottom of the container, and the depth of the liquid in the container shall be not less than 4.0 in. (10.2 cm), as shown in Figure 1 a, b and c; and (4) in both assemblies, the thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 0.5 in. (13 mm) but not touching the ring.

Thermometers (mercury-in-glass)

Depending upon the expected softening point of the sample, use either an ASTM 15C low-softening-point thermometer (-2° to 80°) or an ASTM 16C high-softening-point thermometer

(30° to 200°).

Stirrer

Use a suitable mechanical stirrer rotating between 500 and 700 rpm. To ensure uniform heat distribution in the heating medium, the direction of the shaft rotation should move the liquid upward. (See Figure 2d for recommended dimensions.)

Sample Preparation

Select a representative sample of the material under test consisting of freshly broken lumps free of oxidized surfaces. Scrape off the surface layer of samples received as lumps immediately before use, avoiding inclusion of finely divided material or dust. The amount of sample taken should be at least twice that necessary to fill the desired number of rings, but in no case less than 40 g. Immediately melt the sample in a clean container, using an oven, hot plate, or sand or oil bath to prevent local overheating. Avoid incorporating air bubbles in the melting 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 same temperature at which the sample is to be poured. While being filled, the rings should rest on an amalgamated brass plate. Pour the sample into the rings so as to leave an excess on cooling. Cool for at least 30 min, and then cut off the excess material cleanly with a slightly heated knife or spatula. Use a clean container and a fresh sample if the test is repeated.

Procedure

Materials having softening points above 80°: Fill the glass vessel with glycerol to a depth of not less than 4.0 in. (10.2 cm) and not more than 4.25 in. (10.8 cm). The starting temperature of the bath shall be 32°. For resins (including rosin), the glycerol should be cooled to not less than 45° below the anticipated softening point, but in no case lower than 35°. Position the axis of the stirrer shaft near the back wall of the container, with the blades clearing the wall and with the bottom of the blades 0.75 in. (18 mm) above the top of the ring. Unless the ball-centering guide is 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 sample at this point. Suspend the ring containing the sample in the glycerol bath so that the lower surface of the filled ring is 1.0 in. (25.4 mm) above the surface of the lower horizontal plate (see Figure 1d), which is at least 0.5 in. (13 mm) and not more than 0.75 in. (18 mm) above the bottom of the glass vessel, or 1.0 in. (25.4 mm) above the bottom of the container (see Figure 2e). Place the ball in the glycerol but not on the test specimen. Suspend an ASTM high-softening-point thermometer (16C) in the glycerol so that the bottom of its bulb is level with the bottom of the ring and within 0.5 in. (13 mm) but not touching the ring. Maintain the initial temperature of the glycerol for 15 min, and then, using suitable forceps, place the ball in the center of the upper surface of the sample in the ring. Begin stirring, and continue stirring at 500 to 700 rpm until completion of the determination. Apply heat at such a rate that the temperature of the glycerol is raised 5° per min, avoiding the effects of drafts by using shields if necessary.

[Note: The rate of rise of the temperature shall be uniform and shall not be averaged over the test period. Reject all tests in which the rate of rise exceeds $\pm 0.5^\circ$ for any minute period after the first three.]

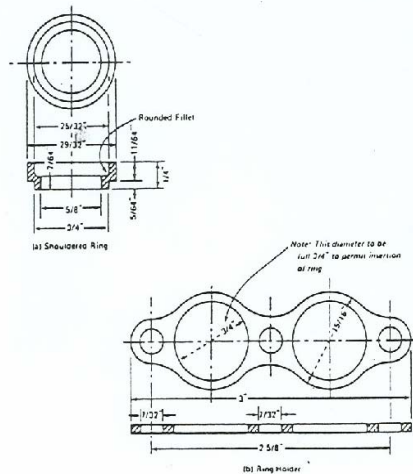
Record as the softening point the temperature of the thermometer at the instant the sample

touches the lower horizontal plate (see Figure 1d) or the bottom of the container (see Figure 2e). Make no correction for the emergent stem of the thermometer.

Materials having softening points of 80° or below: Follow the above procedure, except use an ASTM low-softening-point thermometer (15C) and use freshly boiled water cooled to 5° as the heating medium. For resins (including rosins), use water cooled to not less than 45° below the anticipated softening point, but in no case lower than 5°.

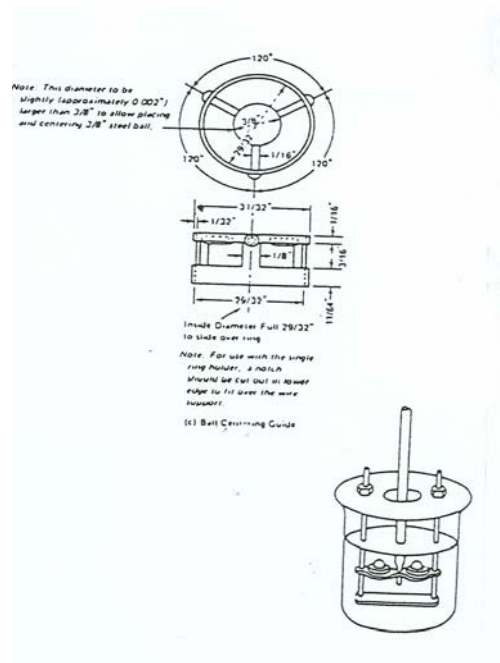
Apparatus - Ring and Ball Softening Point

(a) Shouldered Ring



(b) Ring Holder

(c) Bell Centering Guide



(d) Assembly Apparatus with Two Rings

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

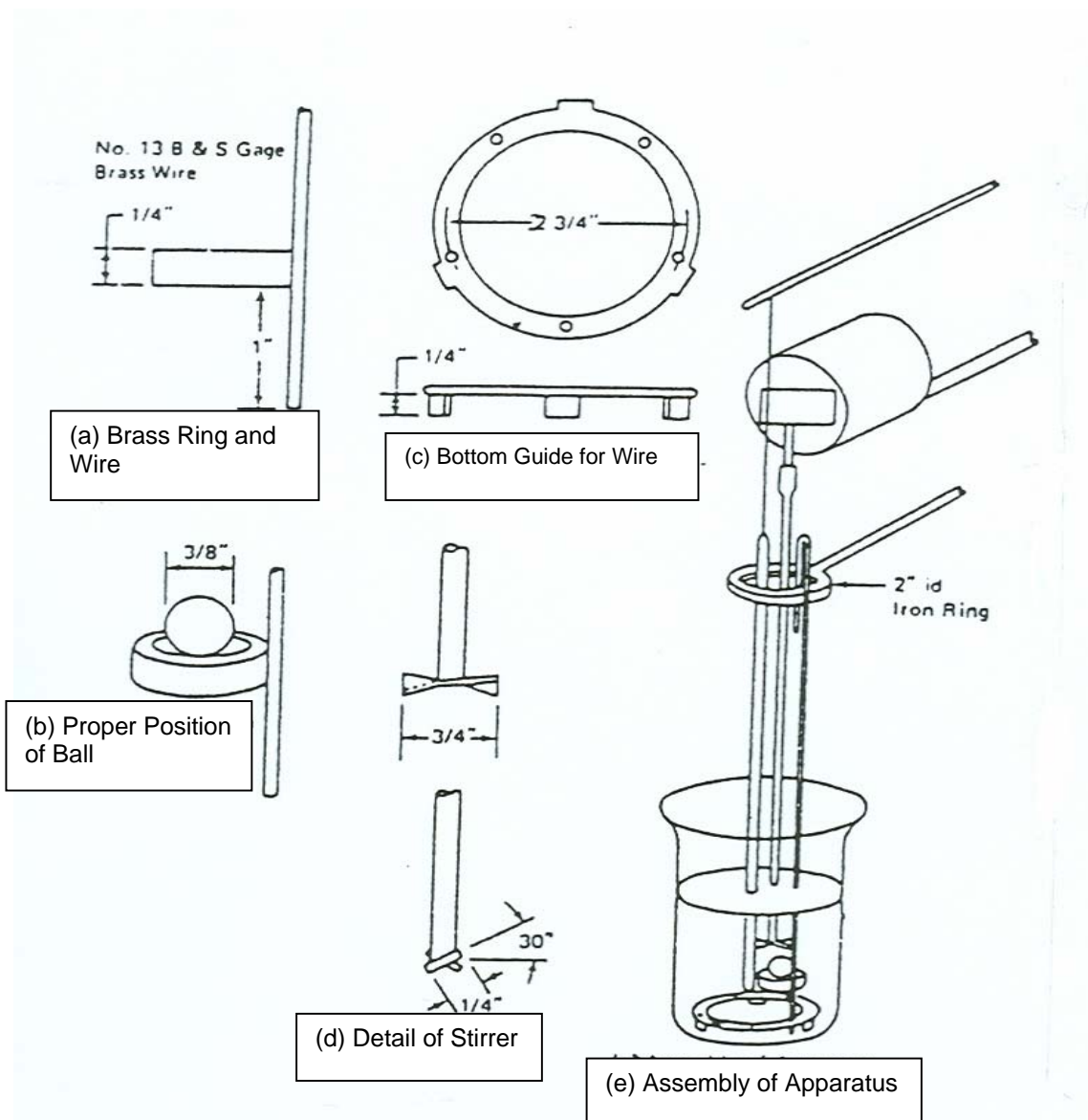


Figure 2. Assembly of Apparatus Showing Stirrer and Single Shouldered Ring.

Figures 1 and 2 are referenced from the Food Chemicals Codex, 6th Edition, 2008, p. 1161 (figures 40 and 41).

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