INTERNATIONAL STANDARD

ISO 4625-1

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Binders for paints and varnishes — Determination of softening point —

Part 1: Ring-and-ball method

Liants pour peintures et vernis — Détermination du point de ramollissement —

Partie 1: Méthode de l'anneau et de la bille

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4625-1 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

It cancels and replaces ISO 4625:1980, which has been technically and editorially revised to harmonize it with ASTM E 28-99, *Standard Test Methods for Softening Point of Resins Derived from Naval Stores by Ring-and-Ball Apparatus.* The main changes are the introduction of an automated procedure and the splitting of the softening point values into four ranges: less than 35 °C, 35 °C to less than 80 °C, 80 °C to 150 °C and greater than 150 °C. The moulding method for the preparation of test pieces has been deleted.

ISO 4625 consists of the following parts, under the general title *Binders for paints and varnishes — Determination of softening point*:

— *Part 1: Ring-and-ball method*

— *Part 2: Cup-and-ball method*

Binders for paints and varnishes — Determination of softening point —

Part 1:
Ring-and-ball method

1 Scope

This part of ISO 4625 specifies methods of determining the softening point of resins (including rosin) and similar materials by means of the ring-and-ball apparatus.

Both manual and automatic methods are specified.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

IEC 60751, *Industrial platinum resistance thermometer sensors*

ASTM E 691, *Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
softening point
temperature at which a disc of sample held within a horizontal ring is forced downward a distance of 25,4 mm under the weight of a steel ball as the disc is heated at a prescribed rate in a water, glycerol, silicone oil, ethylene glycol/water or glycerol/water bath

4 Principle

In general, with materials of the types mentioned in Clause 1, 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, closely defined method if the results obtained are to be comparable.

In these test methods, the softening point is defined as the temperature at which a disc of the sample held within a horizontal ring is forced downward a distance of 25,4 mm under the weight of a steel ball as the sample is heated at 5 °C/min in a water, glycerol, silicone oil, ethylene glycol/water or glycerol/water bath.

5 Sampling and preparation of test pieces

5.1 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

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 use, avoiding inclusion of finely divided material or dust.

5.2 Preparation of test pieces by the pour method

5.2.1 Field of application

This preparation procedure is suitable for resins (including rosin) and other substances that can be heated and poured without adverse effects on the softening point.

5.2.2 Apparatus

5.2.2.1 **Container**, in which the sample can be melted.

5.2.2.2 **Knife** or **spatula**.

5.2.2.3 **Oven, hot-plate, sand bath** or **oil bath**.

5.2.3 Procedure

Take a quantity of the sample (see 5.1) which is at least twice that necessary to fill the desired number of rings (7.1.1), but in no case less than 40 g, and melt it immediately in a clean container (5.2.2.1) using an oven, hot-plate, sand bath or oil bath (5.2.2.3) to prevent local overheating. Take care to avoid incorporating air bubbles in the sample. Melt the sample completely, but do not heat it above the minimum temperature necessary to pour it easily. The time from the beginning of heating to the pouring of the sample shall not exceed 15 min.

For materials that are heat-sensitive, continuously inert the atmosphere in the container holding the sample with nitrogen (N_2) during the melting procedure.

For materials that tend to crack or shrink in the rings on cooling, preheat the rings immediately before filling them to approximately the temperature at which the material is to be poured. The rings, while being filled, shall rest on a suitable metal surface. Pour a sufficient quantity of the sample into the ring so as to leave an excess on cooling. Allow to cool for at least 30 min and trim off the surplus material at the periphery of the ring. To remove excess material from the top, cut the excess material off cleanly with a slightly heated knife or spatula (5.2.2.2) or grasp the ring in a pair of tongs and draw the top surface of the test piece quickly and firmly over the surface of a heated metal plate. If the determination is repeated, use a clean container and a fresh quantity of the sample.

5.3 Preparation of test pieces from samples having a low softening point (up to 35 °C)

5.3.1 Apparatus

5.3.1.1 **Aluminium foil**.

5.3.1.2 **Dry ice** or **freezer**.

5.3.1.3 **Knife** or **spatula**.

5.3.2 Procedure

Take a suitable quantity of the sample (see 5.1). Place one of the rings (7.1.1) on a piece of aluminium foil (5.3.1.1). Pour the material to be tested into the ring. Then place the foil and filled ring on dry ice or in a freezer (5.3.1.2) to cool. The material in the ring shall be free of bubbles.

After cooling, cut or scrape off any excess material using a slightly heated knife or spatula (5.3.1.3), then slide the ring gently from the foil. Place the ring in the ring holder (7.1.8) and immediately perform the softening point determination as described in 7.3.

6 Materials (heating-bath liquids)

6.1 **Distilled or deionized water**, freshly boiled, for softening points between 35 °C and 80 °C

Use freshly boiled water that has been cooled to at least 27 °C below the anticipated softening point, but not lower than + 5 °C. The use of freshly boiled water is essential, as otherwise air bubbles may form on the test piece and affect the result.

6.2 **Glycerol**, for softening points between 80 °C and 150 °C, USP[1] grade or equivalent.

Repeated use of glycerol will increase the moisture content over time and may affect results. Use fresh glycerol if any change in appearance is noted.

Do not use glycerol for softening points greater than 150 °C due to the 160 °C flash point of glycerol.

6.3 **Silicone oil (polymethylsiloxane)**, 50 cSt viscosity, for softening points above 80 °C.

The silicone oil shall be stable up to a temperature of at least 200 °C, remain clear within this temperature range, have no apparent reactivity with the test piece, have a high water repellency, and maintain a uniform viscosity and stirring rate within the temperature range.

Replace with fresh silicone oil if any change in appearance is noted. Do not use silicone oil that contains any gels as gels are an indicator that degradation has occurred.

6.4 **Ethylene glycol**, for softening points below 35 °C.

Prepare a fresh 1 + 1 (by volume) mixture of distilled water and ethylene glycol prior to the determination. For softening points between 0 °C and 35 °C, a 1 + 1 (by volume) mixture of glycerol and water may be used as an alternative.

1) U.S. Pharmacopeia.

7 Manual ring-and-ball method

7.1 Apparatus

7.1.1 Shouldered rings, of brass or steel, conforming to the dimensions shown in Figure 1a).

7.1.2 Steel balls, diameter (9,53 ± 0,1) mm and mass (3,50 ± 0,05) g.

7.1.3 Ball-centering guide (optional), constructed of brass and having the shape and dimensions illustrated in Figure 1c).

7.1.4 Heat-resistant glass beaker, not less than 85 mm in diameter and not less than 125 mm in depth from the bottom to the flare (an 800 ml low-form beaker of heat-resistant glass meets this requirement).

7.1.5 Thermometers:

7.1.5.1 Thermometer for low softening points, having a range from – 2 °C to + 80 °C.

7.1.5.2 Thermometer for medium softening points, having a range from 30 °C to 200 °C.

7.1.5.3 Thermometer for high softening points, having a range from – 2 °C to + 300 °C.

Or, as an alternative to any of the above three thermometers:

7.1.5.4 Resistance thermometer, e.g. Pt100 in accordance with IEC 60751.

7.1.6 Holder for ring and thermometer.

Any convenient apparatus may be used to hold the ring and thermometer in place, provided that it meets the following requirements:

7.1.6.1 The rings (7.1.1) shall be held in a horizontal position.

7.1.6.2 When using the apparatus as shown in Figure 1d), the bottom of each ring shall be 25 mm above the horizontal plate below it, the bottom surface of the horizontal plate shall be 13 mm to 19 mm above the bottom of the beaker (7.1.4), and the depth of liquid in the beaker shall be not less than 100 mm.

7.1.6.3 The thermometer shall be suspended so that the bottom of the bulb is level with the bottoms of the rings and within 13 mm of each ring, but not touching either ring. For referee work, no more than two rings shall be used.

7.1.7 Stirrer.

The liquid in the heating bath (beaker) shall be stirred at a speed sufficient to ensure uniform heat distribution without causing sideways displacement of the resin as it softens in the ring. A stirring rate of 500 r/min to 700 r/min is typical. A mechanical motor-driven stirrer, mounted so that any vibrations created by its rotation are not conveyed directly to the ring holder, or a magnetic stirrer placed under the bath, can be used.

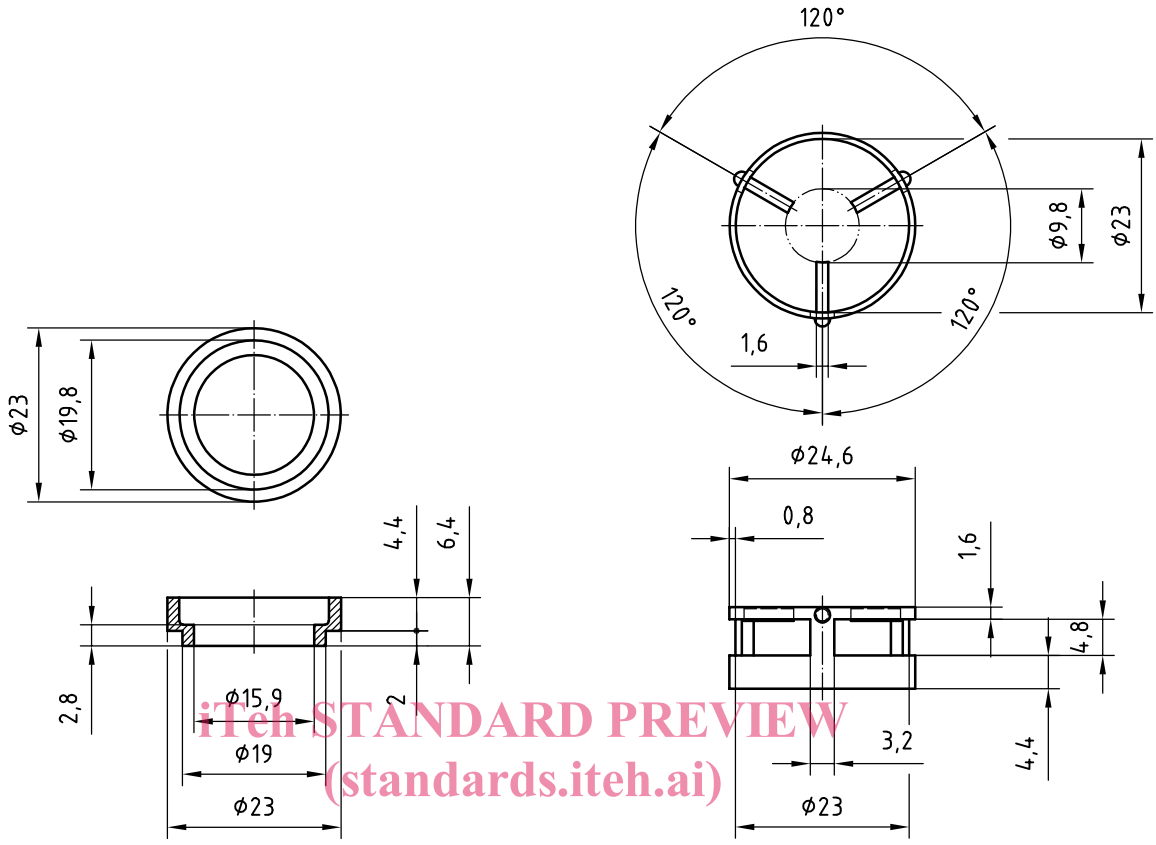
7.1.8 Device for heating the heating-bath liquid, capable of maintaining the required heating rate.

An apparatus may be used which increases the temperature and registers the softening point automatically.

7.2 Calibration

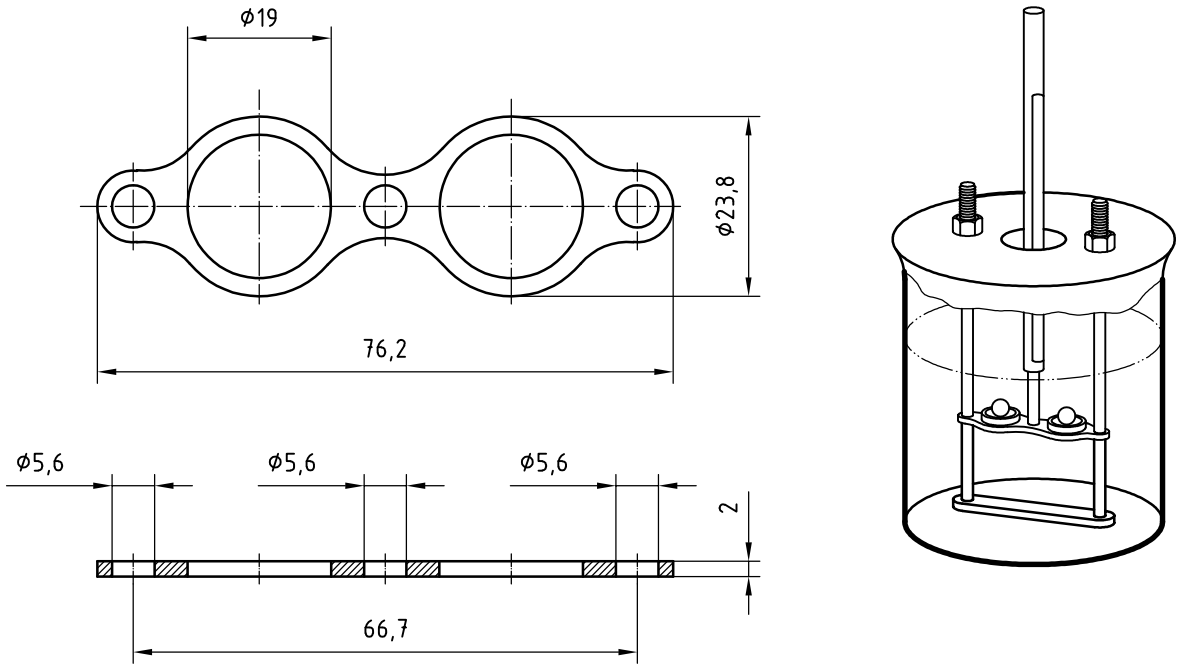
A calibration check of any temperature controllers used in the manual ring-and-ball softening point apparatus shall be performed on a regular basis since accurate temperature control is required.

Dimensions in millimetres
All tolerances ± 0,2 mm



a) Shouldered ring

b) Ball-centering guide



c) Ring holder

d) Assembled apparatus

Figure 1 — Apparatus used for manual determination of softening point