International Standard 4625

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Binders for paints and varnishes — Determination of softening point — Ring-and-ball method

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Foreword

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It has been approved by the member bodies of the following countries :

Australia	India	Poland
Austria	Iran	Romania
Brazil	Ireland	South Africa, Rep. of
Bulgaria	Israel	Sweden
Canada	Italy	Switzerland
Czechoslovakia	Kenya	Turkey
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No member body expressed disapproval of the document.

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Binders for paints and varnishes — Determination of softening point — Ring-and-ball method

1 Scope and field of application

This International Standard specifies a method for the determination of the softening point of resins (including rosin) and similar materials by means of the ring-and-ball apparatus.

2 Definition

For the purpose of this International Standard the following definition applies.

softening point : The temperature at which a disk of the sample held within a horizontal ring is forced downwards a distance of 25 mm under the weight of a steel ball as the test piece is heated at a prescribed rate in a water or glycerol or paraffin oil bath.

3 Principle

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 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.

4 Apparatus

4.1 Shouldered rings, of brass, conforming to the dimensions shown in figure 1b). For use in the powder method of sample preparation, the ring may be made of steel to minimize the possibility of its deformation during the compacting operation.

4.2 Steel balls, diameter 9,5 mm, and mass 3,50 ± 0,05 g.

4.3 Ball-centring guide, constructed of brass and having the general shape and dimensions illustrated in figure 1d); its use is optional.

4.4 Container : a glass vessel, capable of being heated, not less than 85 mm in diameter and not less than 125 mm in depth from the bottom of the flare (an 800 ml low-form beaker of heat-resistant glass meets this requirement).

4.5 Thermometers (see the specifications in the annex)

4.5.1 Low-softening-point thermometer.

4.5.2 High-softening-point thermometer.

4.6 Support for ring and thermometer

Any convenient method for supporting the ring and thermometer may be used, provided that it meets the following requirements :

4.6.1 The rings (4.1) shall be supported in a substantially horizontal position.

4.6.2 The bottom of the ring shall be 25 mm above the horizontal plate below it; the bottom surface of the horizontal plate shall be 13 to 19 mm above the bottom of the container (4.4), and the depth of liquid in the container shall be not less than 100 mm.

4.6.3 The thermometer (4.5.1 or 4.5.2) shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 13 mm but not touching the ring. For referee work, no more than two rings shall be used.

4.7 Mechanical stirrer, two-bladed, motor-driven, attached to the bottom of a true vertical shaft to ensure uniform heat distribution [see figure 1e) for dimensions]. The direction of shaft rotation shall move the liquid upward. The rotational frequency shall be within the range 500 to 700 r/min.

4.8 Apparatus for test piece preparation

4.8.1 For the powder method (5.1)

4.8.1.1 Sieves, with 63 µm and 315 µm mesh openings.

4.8.1.2 Porcelain mortar.

4.8.1.3 Mortar and **pestle,** of steel, with sleeve, knock-out button and ring support, conforming to the dimensions shown in figure 2.

4.8.1.4 Hammer, of a soft material (for example Babbit's metal or lead-impregnated rubber), of mass about 1 kg.

Alternatively :

4.8.1.5 Hydraulic press, capable of maintaining sustained pressures up to 55 kPa.

4.8.2 For the moulding method (5.2)

4.8.2.1 Hot-plate, 3-heat, 200 mm, 1 000 W, with variable transformer.

4.8.2.2 Aluminium or steel plates, 100 mm × 150 mm × 1,6 mm.

4.8.2.3 Spatula, 200 mm, with stiff blade.

4.8.2.4 Tongs.

4.8.3 For the pour method (5.3)

4.8.3.1 Container, in which the sample can be melted.

4.8.3.2 Knife or **spatula.**

4.8.3.3 Copper or aluminium plate, on which filled rings can be placed.

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

5 Sampling and preparation of test piece

5.1 Powder method

5.1.1 Field of application

This method is applicable to resins and other materials that cannot be melted and poured without altering the softening point.

5.1.2 Selection of sample

Select a sample representative of the material under test. 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 using, avoiding inclusion of finely divided material or dust.

5.1.3 Procedure

Break up the lumps until there are no particles larger than 3 mm. Mix the material thoroughly, and quarter down until a suitable quantity (approximately 50 to 75 g) is obtained for powdering. Pulverize the quartered sample in the porcelain mortar (4.8.1.2) or by other suitable means, and fractionate by screening through the 63 µm and 315 µm sieves (4.8.1.1). Immediately use the material passing the 315 µm sieve and retained on the 63 µm, for preparation of the test piece.

Assemble one of the shouldered rings (4.1) with the ring support, the mortar, and the knock-out button (4.8.1.3), together with the sleeve, as shown in figure 2. Take care to ensure that the ring is properly centred and seated in the cut-out section of the sleeve. Pour the powdered material into the sleeve until it is about 13 mm above the top of the ring (approximately 3 g is required).

Place the pestle in the sleeve and compact the powder by rapping the pestle sharply 50 to 60 times with the hammer (4.8.1.4) or by applying a pressure of 48 to 51 kPa in the press (4.8.1.5) and holding this pressure from 3 to 5 min. Remove the ring from the mortar and sleeve. If the material sticks, the bottom of the mortar and the pestle may be covered with aluminium foil. An excess of material shall remain above the top surface of the ring. Carefully scrape this off, until the top of the test piece is level with the ring. If the top and bottom surfaces of the test piece in the ring are not smooth and level with the ring, discard the test piece and repeat the compacting operation, using a clean ring and fresh powder.

5.2 Moulding method

5.2.1 Field of application

This method is applicable to resins (except rosin) and other materials that are heat-sensitive and cannot be melted and poured without altering the softening point.

5.2.2 Selection of sample

Select a sample representative of the material under test. 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 using, avoiding inclusion of finely divided material or dust, and then crush to small lumps in a mortar.

5.2.3 Procedure

Turn the hot-plate (4.8.2.1) to low heat and allow to come to temperature. Place one of the shouldered rings (4.1), bottom down, on one end of an aluminium or steel plate (4.8.2.2) and then place this assembly on the hot-plate. Place 10 to 15 g of the crushed resin on the metal plate so as to form a layer about 6 mm thick and about 8 mm in diameter. The resin in contact with the metal plate will quickly begin to soften. By means of the spatula (4.8.2.3), scrape and knead the entire mass until it is soft and plastic, using a second spatula if necessary (see note).

Finally, gather the entire blob of resin on the spatula, remove the metal plate from the hot-plate with the tongs (4.8.2.4) and quickly press the resin into the shouldered ring, applying pressure to the top of the spatula. Allow the assembly to cool somewhat; then tap the plate and spatula to free the ring. Trim off the excess resin on the periphery of the ring. In order to remove excess resin from the top, grasp the ring with the tongs and draw the top surface quickly and firmly over the surface of a heated metal plate (4.8.2.2).

NOTE — In general no more than 5 min should be required for this operation. Excessive exposure to elevated temperature will promote changes in the sample, resulting in high melting points.

5.3 Pour method

5.3.1 Field of application

This method is only applicable to rosin.

5.3.2 Selection of sample

Select a sample representative of the material under test. 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 using, avoiding inclusion of finely divided material or dust.

5.3.3 Procedure

Take a quantity of the sample at least twice that necessary to fill the desired number of rings (4.1) but in no case less than 40 g, and melt it immediately in a clean container (4.8.3.1) using an oven, hot-plate, sand bath or oil bath (4.8.3.4) to prevent local overheating. Take care to avoid incorporating air bubbles in the material, which shall not be heated above the temperature necessary to pour it readily. 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 temperature at which the material is to be poured. The rings, while being filled, should rest on the copper or aluminium plate (4.8.3.3). Pour the sample into the rings so as to leave an excess on cooling. After cooling for a minimum of 30 min, cut off the excess material cleanly with the slightly heated knife or spatula (4.8.3.2). If the test is repeated, use a clean container and a fresh quantity of the sample.

6 Procedure

NOTE — If the material is partially soluble in water (6.1) or glycerol (6.2), a high boiling paraffin oil may be used. In this case, the temperature shall be raised 1 °C/min.

6.1 Materials having softening points of 80 °C or below

6.1.1 Assembly of apparatus

Fill the container (4.4), to a depth of not less than 100 mm and not more than 108 mm, with freshly boiled distilled water at 5 °C. For resins (including rosin), use water which has been cooled to not less than 45 °C below the anticipated softening point, but in no case lower than 5 °C. (See also the note to 6.)

Locate the axis of the shaft of the stirrer (4.7) near the back wall of the container, with the blades clearing the wall and with the bottom of the blades 19 mm above the top of the rings (4.1). Unless the ball-centring guide (4.3) is to be used, make a slight indentation in the centre of the test piece by pressing one of the balls (4.2) or a rounded rod, slightly heated for hard materials, into the material at this point. Suspend the rings containing the test pieces in the water so that their lower surfaces are 25 mm above the upper surface of the lower horizontal plate [see figure 1a)], which is at least 13 mm and not more than 19 mm above the bottom of the container.

Place the balls (4.2) in the water but not on the test pieces. Suspend a low-softening-point thermometer (4.5.1) so that the bottom of its bulb is level with the bottom of the rings, and within 13 mm of, but not touching, the rings. Maintain the initial temperature of the water for 15 min. With the forceps (4.8.2.4) place the balls in the centre of the upper surface of the material in the rings.

Start stirring, with the stirrer (4.7), and continue the stirring at 500 to 700 r/min until completion of the determination.

6.1.2 Heating

Apply heat in such a manner that the temperature of the water is raised 5 °C/min. Avoid the effect of draughts by using shields if necessary. (See also the note to 6.)

The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any period of 1 min after the first 3 min shall be 0,5 °C. Reject all tests in which the rate of rise exceeds these limits.

6.1.3 Determination of softening point

Record as the softening point the temperature of the thermometer at the instant the test piece touches the lower horizontal plate [see figure 1a)]. Make no correction for the emergent stem of the thermometer.

6.2 Materials having softening points above 80 °C

Use the same procedure as described in 6.1, but fill the bath with glycerol and use a high-softening-point thermometer (4.5.2). Use glycerol that has been cooled to not less than 45 °C below the anticipated softening point, but in no case lower than 32 °C. (See also the note to 6.)

7 Precautions

7.1 The stirrer motor shall be so mounted that any vibrations created by its rotation are not conveyed directly to the test piece support.

7.2 The use of freshly boiled distilled water is essential, as otherwise air bubbles may form on the test piece and affect the result. Rigid adherence to the prescribed rate of heating is absolutely essential for reproducibility of results.

8 Expression of results

Calculate the mean of two determinations and round it to the nearest 0,2 °C.

9 Precision

9.1 Repeatability (r)

The value below which the absolute difference between two single test results, on identical material, obtained by one operator in one laboratory using the same equipment within a short interval of time using the standardized test method, may be expected to lie with a 95 % probability, is 1 °C.

9.2 Reproducibility (R)

The value below which the absolute difference between two single test results, on identical material, obtained by operators in different laboratories, using the standardized test method, may be expected to lie with a 95 % probability, is 2 °C.

10 Test report

The test report shall include at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard;

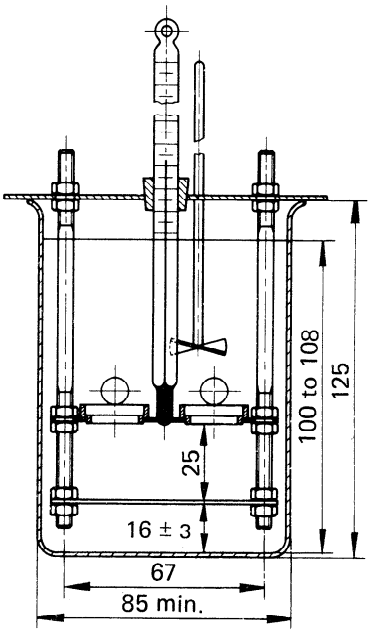
c) for materials softening around 80 °C, whether water or glycerol was used, since a glycerol bath yields slightly higher results than a water bath, or whether a high boiling paraffin oil was used;

d) any deviation, by agreement or otherwise, from the test procedure specified;

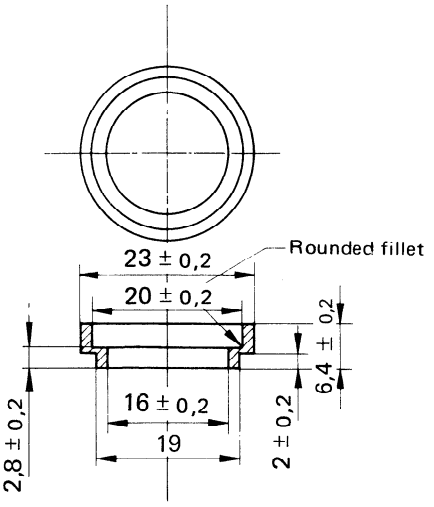
e) the result of the test;

f) the date of the test.

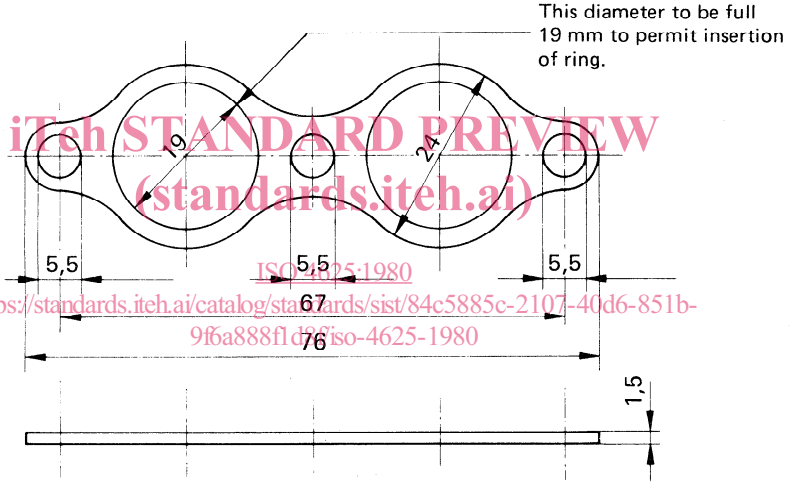
Dimensions in millimetres



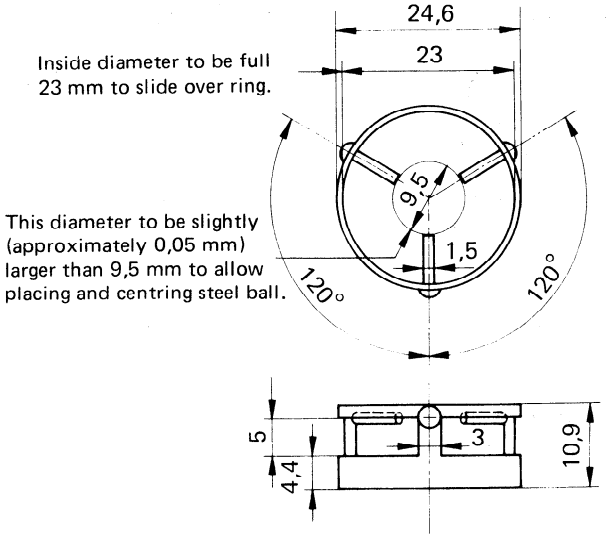
a) Assembly



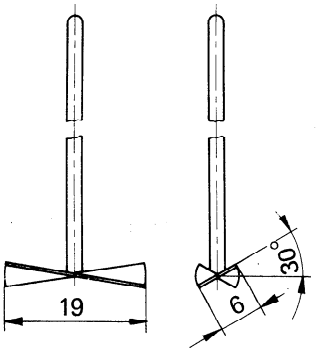
b) Shouldered ring



c) Ring holder



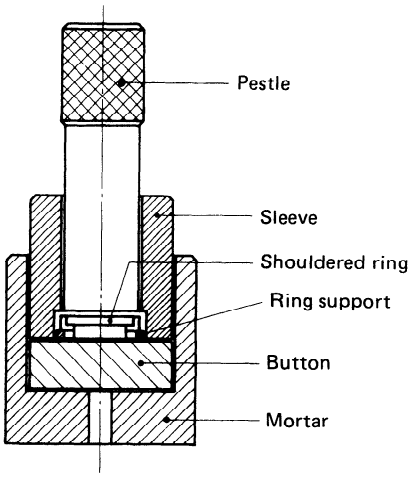
d) Ball centring guide



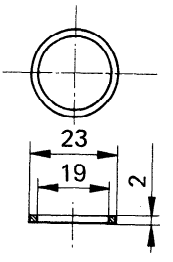
e) Detail of stirrer

Figure 1 — Assembly and components for two-ring apparatus

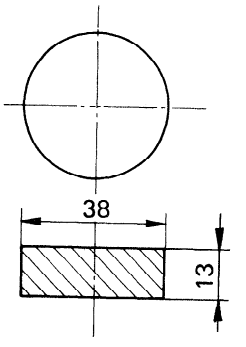
Dimensions in millimetres



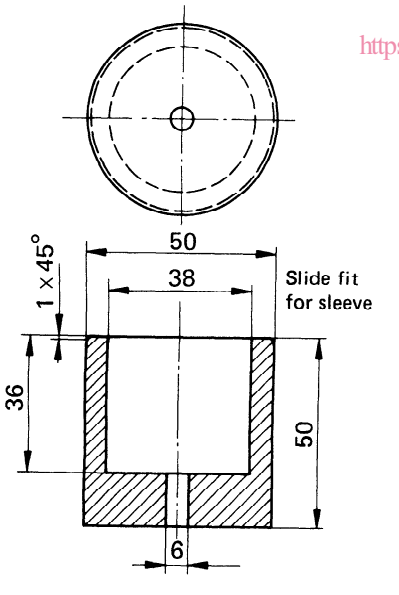
a) Assembled mortar and pestle



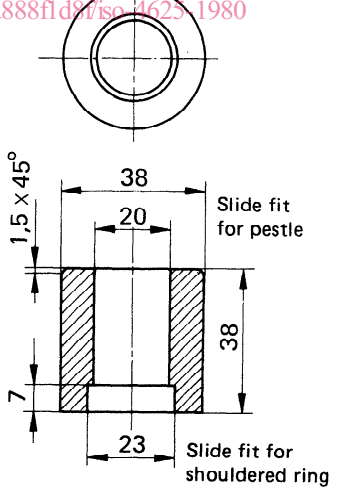
b) Ring support



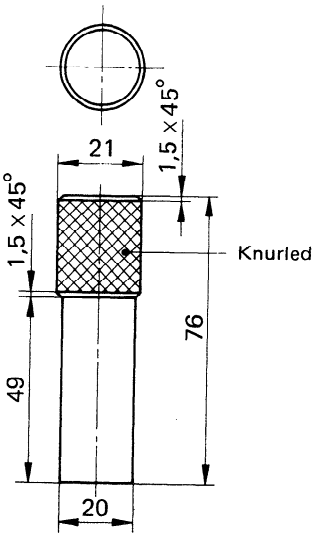
c) Button



d) Mortar



e) Sleeve



f) Pestle

Figure 2 — Assembly and components of mortar and pestle

Annex

Specifications for high- and low-softening-point thermometers

Characteristic	Low	High
Range, °C	− 2 to 80	30 to 200
Immersion	total	total
Subdivision, °C	0,2	0,5
Long lines at each	1 °C	1 °C
Numbers at each	2 °C	5 °C
Permissible scale error, max. °C	0,2	0,3
Expansion chamber permitting heating to, °C	130	250
Total length, mm	397 ± 5	397 ± 5
Outer diameter of stem, mm	6 to 7	6 to 7
Length of bulb, mm	9 to 14	9 to 14
Outer diameter of bulb, mm	4,5 to 5,1	4,5 to 5,1
Distance of bottom of bulb, mm, to line at	75 0 °C	75 30 °C
Distance of bottom of bulb, mm to line at	333 to 354 80 °C	333 to 354 200 °C