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ISO 4625-2

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Binders for paints and varnishes — Determination of softening point —

Part 2: Cup-and-ball method

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

Partie 2: Méthode de la coupe 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 4625-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*, in collaboration with ASTM D01.34, *Naval Stores*. It has been harmonized with ASTM D 6090-99, *Standard Test Method for Softening Point of Resins (Mettler Cup and Ball Method)*.

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 2: Cup-and-ball method

1 Scope

This part of ISO 4625 specifies a method for determining the softening point of resins (including rosin) by means of a cup-and-ball apparatus and can, under user-defined conditions, give results comparable to those obtained using the ring-and-ball method (ISO 4625-1).

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 4625-1, *Binders for paints and varnishes — Determination of softening point — Part 1: Ring-and-ball method*

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

3 Terms and definitions

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

3.1
softening point
temperature at which a test sample in a cylindrical cup with a 6,35 mm hole in the bottom, with a stainless-steel ball 8,7 mm in diameter centred on top of the test sample in the cup, flows downward a distance of 19 mm to interrupt a light beam as the test sample is heated at a constant rate in air

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.

A test sample, with the ball on it, is placed in a cup and heated at a specified rate. The softening test sample is forced downwards by the weight of the ball. The temperature at which the test sample has sunk by 19 mm is called the softening point.

5 Apparatus

5.1 Softening point apparatus[1], consisting of a control unit with a digital temperature indicator, matched measuring cell, cartridge assembly (cup and upper portion) and accessories. The control unit automatically regulates the heating rate of the measuring cell. The softening point is indicated on the readout, and the heating programme is stopped, when the test sample flow triggers a photocell detector. Further details are given in 5.1.1 to 5.1.4.

5.1.1 Central processor, capable of regulating the heating rate to provide a continuous, linear increase in temperature from 25 °C to 375 °C.

5.1.2 Measuring cell, capable of heating the cup assembly (5.4) at a constant rate with an accuracy of ± 0,2 °C/min. It shall include a sensing system capable of detecting the softening point with a precision of 0,1 °C.

5.1.3 Cup assembly, made of chromium-plated brass, consisting of the cup itself and an upper portion, conforming to the dimensions shown in Figure 1.

5.1.4 Ball, made of stainless steel, 8,7 mm in diameter and weighing (2,77 ± 0,22) g.

Dimensions in millimetres

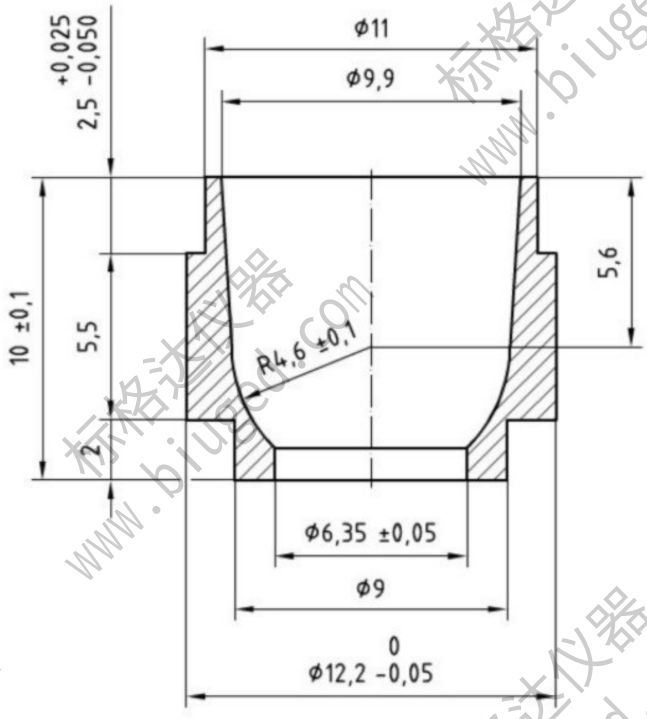

Figure 1 — Cup

1) The apparatus is available commercially. Details may be obtained from the Secretariat of ISO/TC 35/SC 10.

6 Calibration of apparatus using a primary standard

6.1 General

This step, required only occasionally, is designed to establish that the temperature indicated by the instrument is in agreement with a known standard. A special cup with a bottom orifice of 2,8 mm is used instead of the one specified for testing the resin.

NOTE The stainless-steel ball is not used during calibration.

6.2 Reagent

Use either analytical reagent or primary standard grade benzoic acid for the calibration. As this material is hygroscopic, store it in a tightly sealed container and replace it with fresh material from a newly opened supply if the softening point of the material is out of the expected range.

6.3 Procedure

6.3.1 Filling the cup

Place the cup on a clean, flat surface and fill it with benzoic acid crystals. Hand press the material into the cup with a 9,2 mm rod of dowel stock or equivalent. Refill and repeat the compression step until the cup is filled with benzoic acid. Remove any crystals from the exterior of the cup.

6.3.2 Heating

Preheat the measuring cell to 121 °C, and maintain it at that temperature. Place the cartridge assembly containing the benzoic acid in position in the measuring cell, taking care that the slits for the light beam are properly positioned. With the instrument set in the dropping-point mode, start the required method, which includes a waiting period of 30 s to allow temperature equilibration between the measuring cell and benzoic acid followed by an automatic temperature ramp of 0,2 °C/min. The temperature will rise steadily at the correct rate until the drop point is reached, then remain steady on the readout.

6.3.3 Cleaning

Immediately remove the cartridge assembly. Check to determine that benzoic acid has passed through the light beam and no pre-triggering has occurred. If a malfunction is suspected, repeat the entire procedure. Inspect the apparatus carefully to ensure that no residue remains. Wash the cartridge parts with a suitable solvent to remove last traces of residue.

6.3.4 Interpretation

If the result is not $(123,5 \pm 0,5)$ °C, repeat the determination. If the second value remains outside the acceptable range, repeat the determination using a fresh cup-full of benzoic acid. If the results remain outside the acceptable range, the instrument requires recalibration or repair. Consult the manufacturer's instruction manual.

7 Instrument check of apparatus using a secondary standard

The primary calibration standard, benzoic acid, might not have a dropping point similar to the softening point of the resin being tested. In such cases, it is desirable to calibrate the instrument using a secondary standard having a dropping point similar to that of the resin being tested. Materials suitable for use as secondary standards are listed, but are not limited to, those in Table 1. Demonstration of conformance to the dropping point of benzoic acid shall be completed before a secondary standard is used.

Table 1 — Materials suitable as secondary standards for cup-and-ball softening point

Approximate dropping point °C	Material
60	Stearyl alcohol
80	Diphenyl carbonate
100	Phenoxyacetic acid 4-*tert*-Butylphenol Methyl urea
110	*m*-Toluic acid Resorcinol
120	Benzoic acid
130	Methyl 4-hydroxybenzoate Urea Benzamide 1,10-Decanedicarboxylic acid
140	Dimethyl terephthalate *o*-Toluamide

Follow the same procedure as in 6.3.2 and 6.3.3 but start about 3 °C below the actual dropping point of the secondary standard.

8 Filling the cups with resin

8.1 Preparation

Fold the tab on an oil-free disposable aluminium weighing dish to a horizontal position and pinch a pour spout into the dish directly opposite the tab. Weigh 5 g to 10 g of resin into the dish.

Place the dish and resin on a preheated hot-plate, and cover with a 600 ml or larger beaker. Heat the resin until it is almost melted.

Remove the beaker from the hot-plate, grasp the tab with forceps or needle-nose pliers, and stir the resin with a probe-type thermometer until it is completely melted and homogeneous. Avoid entraining air in the resin melt. The temperature of the resin shall be no more than 60 °C above the expected softening point. Remove the thermometer and wipe clean. If the resin smokes or appears to boil, repeat the steps described above, setting the maximum temperature 10 °C less than the previous trial.

8.2 Filling

Arrange two cups, small outlet down, on a piece of aluminium foil placed on a smooth, flat surface.

Grasping the dish tab with forceps or pliers, pour the molten resin into both of the cups. Fill the cups to the brim.

Allow the resin to cool for at least 10 min. Cleanly trim off the excess resin on each cup with a slightly heated knife or spatula, or grasp the cup in a pair of tongs and draw the top surface quickly and firmly over the surface of a heated metal plate. Do not make a determination on resin which contains air bubbles.

Assemble the cartridge by placing the cup on the collector, then the upper portion on the cup. Carefully place the ball on the top of the cup.

Make sure that there is no resin on the outside of the cup, the inside of the jacket, or the collecting sleeve. These components shall remain clean during the determination. Wipe the assembly clean before inserting it into the measuring cell.

9 Procedure

The procedure described is for measuring the cup-and-ball softening point of resins. The heating rate is usually determined by the user. Heating rates of 1,0 °C/min to 2,5 °C/min are typical for resins. If a correlation is desired between the cup-and-ball softening point and the ring-and-ball softening point as described in ISO 4625-1, a specific heating rate for that correlation shall be determined for each type of resin.

NOTE To facilitate a comparison of cup-and-ball softening point results between laboratories, a set of reference conditions is provided below:

Cup: 6,35 mm diameter

Heating rate: 1,6 °C/min

Starting temperature: 20 °C below the expected softening point of the resin

Preset the starting temperature of the central processor at 20 °C to 25 °C below the expected softening point of the resin and maintain this temperature for a few minutes. Assemble the cartridge with the stainless-steel ball carefully centred on top of the resin. Place the cartridge assembly in the measuring cell, taking care that the slits for the light beam are properly positioned. With the instrument in the softening-point mode, start the determination, which includes a waiting period of 30 s to allow temperature equilibration between the measuring cell and resin followed by an automatic temperature ramp at the specified rate in degrees Celsius per minute. Heating will be maintained at this rate until the drop point is reached, after which a steady temperature will be displayed on the readout. Record this temperature as the softening point of the resin.

Remove the cartridge assembly upon completion of the test. Check to ensure that the resin has passed through the light beam and no pretriggering has occurred. If a malfunction is suspected, reject the result and repeat the determination. Inspect the apparatus closely to ensure that no particles of residue remain.

Clean the cup and ball by soaking in a suitable solvent.

Care shall be taken in cleaning the cup in order not to scratch or alter the size of the cup orifice.

Clean the interior of the cell immediately after removing the cup assembly. Use soft cotton swabs moistened in an appropriate solvent.

10 Expression of results

Calculate the mean of two determinations and round it to the nearest 1,0 °C.

11 Precision and bias

11.1 General

A limited cup-and-ball softening point interlaboratory precision study was conducted using the same three resins as were used in the study reported in ISO 4625-1.

The study was run in the U.S. in 1998 by 10 laboratories. The test conditions were those listed in the Note in Clause 9.

11.2 Precision data

The precision data given below is for the comparison of two test results:

— repeatability limit, 95 % (within laboratory): 0,5 °C to 1,3 °C;

— reproducibility limit, 95 % (between laboratories): 1,4 °C to 2,1 °C.

The above terms (repeatability limit and reproducibility limit) are used as specified in ISO 5725-1. The respective standard deviations in the test results, related to the above numbers by the factor 2,8, are:

— repeatability standard deviation: 0,2 °C to 0,5 °C;

— reproducibility standard deviation: 0,5 °C to 0,8 °C.

11.3 Bias

Since there is no accepted reference material or method suitable for determining the bias of this method for determining the cup-and-ball softening point, no statement on bias is possible.

12 Test report

The test report shall include at least the following information

a) all details necessary for complete identification of the product tested, including the type of product;

b) a reference to this part of ISO 4625 (ISO 4625-2);

c) the result of the test, as indicated by Clause 10;

d) any deviation from the test procedure specified;

e) the date of the test.

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