INTERNATIONAL STANDARD

ISO 4625-1

Second edition
2020-07

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
ISO 4625-1:2020(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 139, *Paints and varnishes,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 4625-1:2004), which has been technically revised. The main changes compared to the previous edition are as follows:

— the automated method has been classified to be the reference method;

— an introduction with information on all three methods described in the three parts of ISO 4625 series has been added;

— CAS-numbers have been added to the chemicals used;

— the text has been editorially revised;

— the normative references have been updated.

A list of all parts in the ISO 4625 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The ISO 4625 series specifies three widely used procedures for the measurement of the softening point of rosin-based resins. This document presents the ring-and-ball method, which has been the accepted standard for many years. ISO 4625-2 presents the Mettler cup-and-ball method. A new method, called the Mettler method without the ball, is to be presented in a future document.

This document is still the only standard test method accepted in regulatory documents such as Title 21 of the Code of Federal Regulations (CFR) – Food and Drugs.

Other parts of the ISO 4625 series concern Mettler cup-and-ball methods. Although the recommended testing conditions differ, the only difference between the equipment required in such methods is that Mettler method without the ball does not use a ball. Surveys have shown that the Mettler cup-and-ball method specified in ISO 4625-2 is the most widely used in the USA, while the Mettler method without the ball is the most widely used in Europe. These methods are less time consuming than the ring-and-ball manual method and the equipment is less expensive than the ring-and-ball automated method.

As a consequence of the thermoplastic nature of the test resins, the softening points obtained using the recommended test conditions for all three methods are not generally the same. Consequently, the test method and the testing conditions used should be noted in the final report.

Binders for paints and varnishes — Determination of softening point —

Part 1:
Ring-and-ball method

1 Scope

This document specifies the test methods for determining the softening point of resins (including rosin) and similar materials by means of ring-and-ball apparatus.

Both manual and automated methods are specified, the automated method being the reference method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 4618, *Paints and varnishes — Terms and definitions*

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

IEC 60751, *Industrial platinum resistance thermometer and platinum temperature sensors*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
softening point
temperature at which a disc of sample held within a horizontal ring is forced downward a distance of 25 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 shall be made by a fixed, closely defined method if the results obtained are to be comparable.

For the purpose of these test methods, the definition of softening point given in Clause 3 applies.

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 with a low softening point (up to 35 °C)

5.3.1 Apparatus

5.3.1.1 Aluminium foil.

5.3.1.2 Dry ice [CO_2 (solid)] 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 (8.1.6) 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, (CAS-No 56-81-5) for softening points between 80 °C and 150 °C, USP[1)] grade or equivalent.

Repeated use of glycerol increases 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 mm^2/s 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 high water repellence, 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, (CAS-No 107-21-1) 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.

7 Automated ring-and-ball method (reference method)

7.1 Apparatus

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

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

7.1.3 Beaker, 600 ml. Ensure that the dimensions are such that the beaker will properly fit into the heating unit.

7.1.4 Stir bar, of dimensions such that the bar spins freely under the test insert.

1) U.S. Pharmacopeia.