
**Carbonaceous materials for the
production of aluminium — Pitch for
electrodes —**

**Part 1:
Determination of softening point by
the ring-and-ball method**

*Produits carbonés utilisés pour la production de l'aluminium — Brai
pour électrodes —*

*Partie 1: Détermination du point de ramollissement par la méthode
bille et anneau*



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Published in Switzerland

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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.

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).

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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 226, *Materials for the production of primary aluminium*.

This first edition of ISO 5940-1 cancels and replaces ISO 5940:1981, which has been technically revised.

The main changes compared to the previous edition are as follows:

- 5.1.6 has changed to [6.1.2](#) which has been modified so that mercury thermometers are no longer required.
- [Annex A](#) has been modified.

A list of all parts in the ISO 5940 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.

Carbonaceous materials for the production of aluminium — Pitch for electrodes —

Part 1:

Determination of softening point by the ring-and-ball method

1 Scope

This document specifies the ring-and-ball method, a method of using the ring-and-ball apparatus for the determination of the softening point (softening temperature) of pitch used in the production of aluminium.

The method is applicable to pitches having softening points equal to or greater than 30 °C.

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 6257, *Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

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/>

4 Principle

Prepare a disc-shaped test piece by moulding a test portion in a ring, fitting the prepared ring into a heated bath and placing a steel ball on the upper surface of the test piece.

Determine the temperature at which the test piece, on heating at a prescribed rate, softens sufficiently to sag to a specified depth under the mass of the ball.

This temperature corresponds to the softening point (softening temperature).

5 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled water, or water of equivalent purity, freshly boiled and cooled.

5.1 Glycerol, for use with pitches having softening points greater than 85 °C.

5.2 Water, for use with pitches having softening points in the range 30 °C to 85 °C.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Ring-and-ball apparatus (see [Figure B.1](#) for assembled apparatus), comprising:

6.1.1 Rings, brass, tapered (see [Figure B.2](#)), having the dimensions:

- depth: 6,4 mm ± 0,1 mm;
- internal diameter at bottom: 15,9 mm ± 0,1 mm;
- internal diameter at top: 17,5 mm ± 0,1 mm;
- external diameter above shoulder: 20,6 mm ± 0,1 mm;
- external diameter below shoulder: 18,5 mm ± 0,5 mm.

6.1.1.1 Steel balls, of diameter 9,53 mm, each weighing 3,50 g ± 0,05 g.

6.1.1.2 Ball centering guides. A suitable form of guide is shown in [Figure B.3](#).

6.1.1.3 Bath beaker, a heat-resistant glass squat form beaker of capacity 800 ml.

6.1.1.4 Ring support, having the shape and dimensions shown in [Figures B.1](#) and [B.4](#).

The support consists essentially of two parallel brass plates, one above the other, and 25 mm ± 0,5 mm apart. The upper plate is designed to support the two rings ([6.1.1](#)) and its thickness is equal to the distance between the lower surface of one ring and its shoulder.

The two plates are supported by a circular or rectangular holder which rests on the top of the bath and which has a central hole intended for the thermometric device ([6.1.2](#)).

6.1.2 Thermometric device, a thermometric device as described in [Table A.1](#). The thermometric device shall have equal or higher accuracy than the thermometers described in [Table A.2](#).

- a) For pitches with softening point from 30 °C to 120 °C, a device with a range of at least 0 °C to 120 °C. And shall be capable of indicating the temperature to 0,5 °C and stable to within 0,5 °C for the complete length of the test.
- b) For pitches with softening point from 30 °C to 180 °C, a device with a range of at least 0 °C to 210 °C, and also capable of indicating the temperature to 1,0 °C and stable to within 1,0 °C for the complete length of the test.

Thermometers may be used if mercury in glass thermometers are legislation compliant in the jurisdiction where the testing facility is located.

6.1.3 Tripod and open mesh wire gauze, supporting the bath.

6.1.4 Bunsen burner, fitted with a governor if the gas supply is liable to fluctuate.

6.1.5 Moulding plate, having the dimensions shown in [Figure B.5](#).

6.1.6 Draught screen, of suitable design to protect the assembled apparatus from draughts.

NOTE It is also possible to use an automatic ring-and-ball apparatus in place of the manual apparatus, provided that it has the same dimensions and that the same results are obtained.

7 Procedure

7.1 Preparation of test portion

Using the method specified in [Annex B](#), melt a test portion of approximately 50 g of the laboratory sample that has been sampled and prepared as specified in ISO 6257 and heat the rings ([6.1.1](#)). Place the heated rings in the sets of guide pins on the moulding plate ([6.1.5](#)), which shall have been treated to prevent adhesion of the pitch, and immediately pour the pitch into the rings in a steady stream.

The quantity of pitch used for each ring shall be such that, after 20 min, a slight excess remains above the level of the ring. At the end of the cooling period, remove the excess with a knife, but without applying heat. It is essential that the two rings be filled from a single melt and that they be tested together.

7.2 Preparation of the apparatus

Avoiding any aeration, place in the bath beaker ([6.1.1.3](#)) a sufficient amount (600 ml is usually adequate) of either the glycerol ([5.1](#)) or the water ([5.2](#)) so that the level is at least 50 mm above the top of the rings. The liquid shall be previously cooled, if necessary, to at least 45 °C below the expected softening point of the pitch, with the exception that, for soft pitches, the temperature should not be below 5 °C.

Place the prepared rings on their support ([6.1.1.4](#)) and fit this assembly into the bath. Insert the appropriate thermometric device ([6.1.2](#)) coaxially in the beaker, so that the bottom of its bulb is at the level of the lower surface of the rings and is equidistant from each.

Place the ball centring guides ([6.1.1.2](#)) above each ring and place two of the balls ([6.1.1.1](#)) in the bath, but not on the pitch contained in the rings. Place the beaker on the tripod ([6.1.3](#)) so that the ring supporting holder is horizontal.

Allow the assembled apparatus to stand for 15 min without allowing the temperature of the bath to rise, and then, using tongs, place the balls in their guides on the surface of the discs of pitch.

7.3 Determination

Place the Bunsen burner ([6.1.4](#)) midway between the axis and the edge of the beaker and on a diameter at right-angles to that which includes the rings and the thermometric device as indicated in [Figures B.1 a\)](#) and [b\)](#). Heat so that the temperature is raised by 5 °C each minute. Except for the first 3 min, this heating rate shall be maintained to within $\pm 0,5$ °C over each 1 min period. This rate shall not merely be the average over the period of the test and any result from a measurement in which the heating rate does not conform to these requirements shall be rejected. Record the temperature at which the pitch surrounding each ball first touches the lower plate of the support. For the two balls, if this temperature differs by more than 1 °C, reject the results and repeat the test. If the test is repeated for any reason, carry out the whole procedure using a new test portion.

NOTE At about 85 °C, slightly higher results are obtained when using glycerol than when using water.

8 Expression of results

8.1 Method of calculation

The softening point is given as the mean of the two temperatures recorded as described in [7.3](#). Express the result to the nearest 0,2 °C.

8.2 Precision

For pitches having melting points less than 85 °C:

- repeatability = 0,5 °C
- reproducibility = 2,5 °C

For pitches having melting points greater than 85 °C:

- repeatability = 1,5 °C
- reproducibility = 3,0 °C

9 Test report

The test report shall include the following particulars:

- a) an identification of the sample;
- b) the International Standard used, i.e. ISO 5940-1:2019, stating whether glycerol or water was used as the heating medium;
- c) the results and the method of expression used, including a reference to the clause which explains how the results were calculated;
- d) the type of thermometric device used and confirmation of capability to obtain the results reported here;
- e) any deviations from the procedure;
- f) any unusual features observed during the determination;
- g) the date of the test.

Annex A (normative)

Thermionic devices for temperature sensing and output

Table A.1 — Temperature sensing devices

Thermionic device	Example for illustration only	Range of application	Accuracy
Platinum resistance thermometer (PRTs). Class A	Pt 100	−250 °C to 850 °C	0,03 °C typical 0,3
Thermistor	NTC type thermistor	−100 °C to 300 °C	0,1 °C typical
Thermocouple	Type K	−200 °C to 1 300 °C	1 °C typical
Mercury in glass thermometer	See below		

Table A.2 — Details of mercury in glass thermometers

Range	0 °C to 120 °C	50 °C to 210 °C
Graduation	0,5 °C	0,5 °C
Immersion	100 mm	100 mm
Longer lines at each ^a	1 °C and 5 °C	1 °C and 5 °C
Fully figured at each	10 °C	10 °C
Maximum overall length	430 mm	430 mm
Minimum length of main scale	240 mm	240 mm
Maximum error	±0,6 °C	±0,8 °C

^a The lines corresponding to each 5 °C shall be slightly longer than those corresponding to each 1 °C.

Annex B (normative)

Melting of pitch samples

B.1 Introduction

Prior to determining the softening point of pitch, it is necessary to melt the sample. The precision of the determination, and particularly its reproducibility, is considerably influenced by the homogeneity of the molten sample and its freedom from air bubbles. The procedure specified in this annex is designed to minimize variations in test results by standardizing the temperature and heating time.

B.2 Apparatus

B.2.1 Dish, 55 mm in diameter and 35 mm in depth.

Dishes sold for use with penetrometers are suitable.

B.2.2 Oven, capable of being maintained at 50 °C above the expected softening point of the sample.

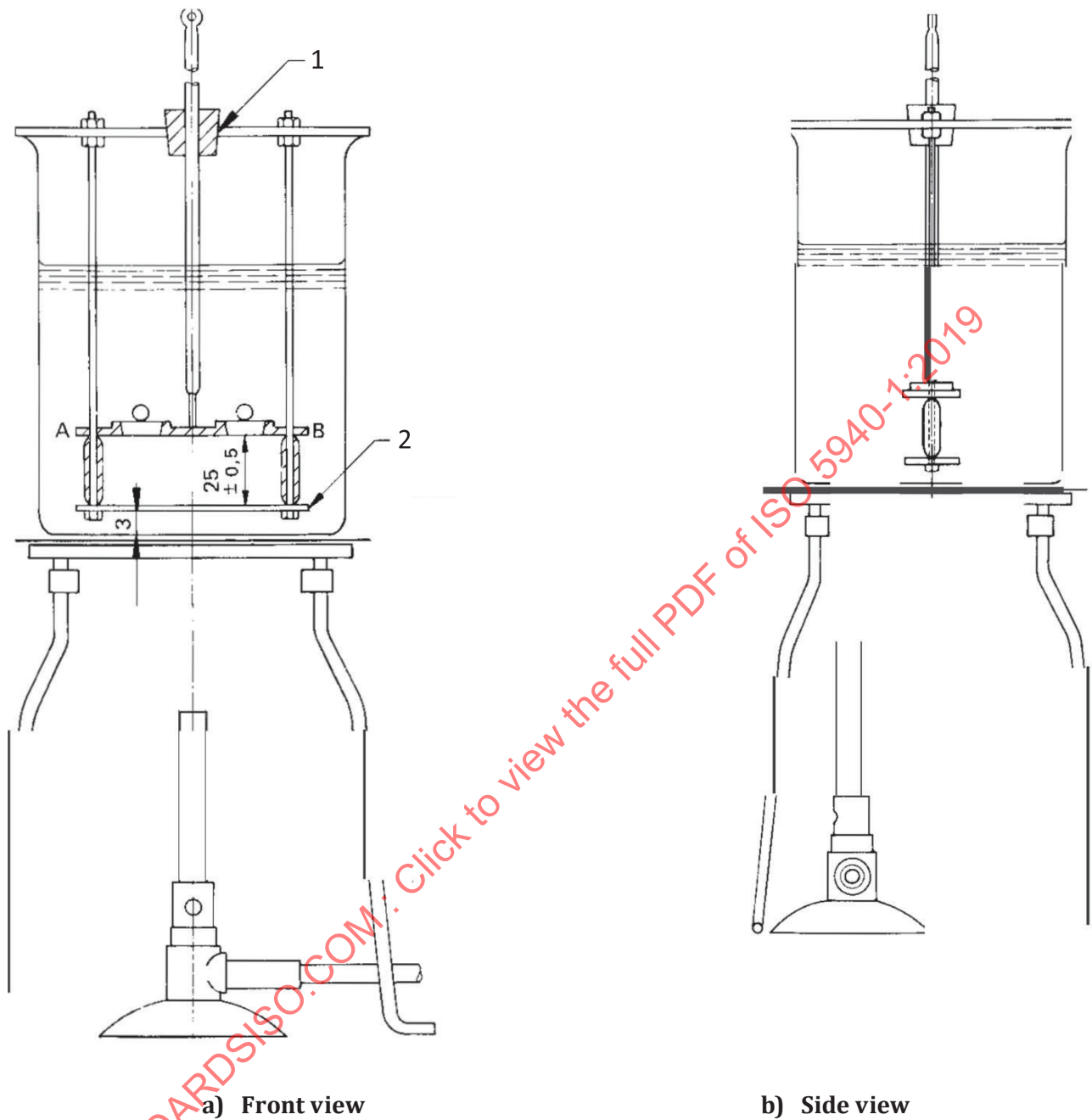
B.3 Procedure

Weigh approximately 50 g of the laboratory sample, into the dish. The laboratory sample shall be powdered if its softening point permits. Hand place it in the oven set at a temperature of 50 °C above the expected softening point of the pitch. Also place the rings ([6.1.1](#)) in the oven.

Maintain the pitch and the rings at this temperature for 2 h and then use the heated rings and the molten pitch to prepare the test piece as described in [7.1](#).

After heating, the surface of the molten pitch should be smooth, shiny and free of skin.

If the surface of the molten pitch is covered with froth this shows the presence of water in the test portion. The test portion shall then be discarded. A new sample shall be prepared according to this annex procedure [B.3](#). Before being placed in the oven the laboratory sample shall be dried. Place the laboratory sample in an evacuated desiccator in the presence of a suitable desiccant.



Key

- 1 central hole for cork
- 2 lower plate, length 76,2, width 25

Figure B.1 — Assembled apparatus

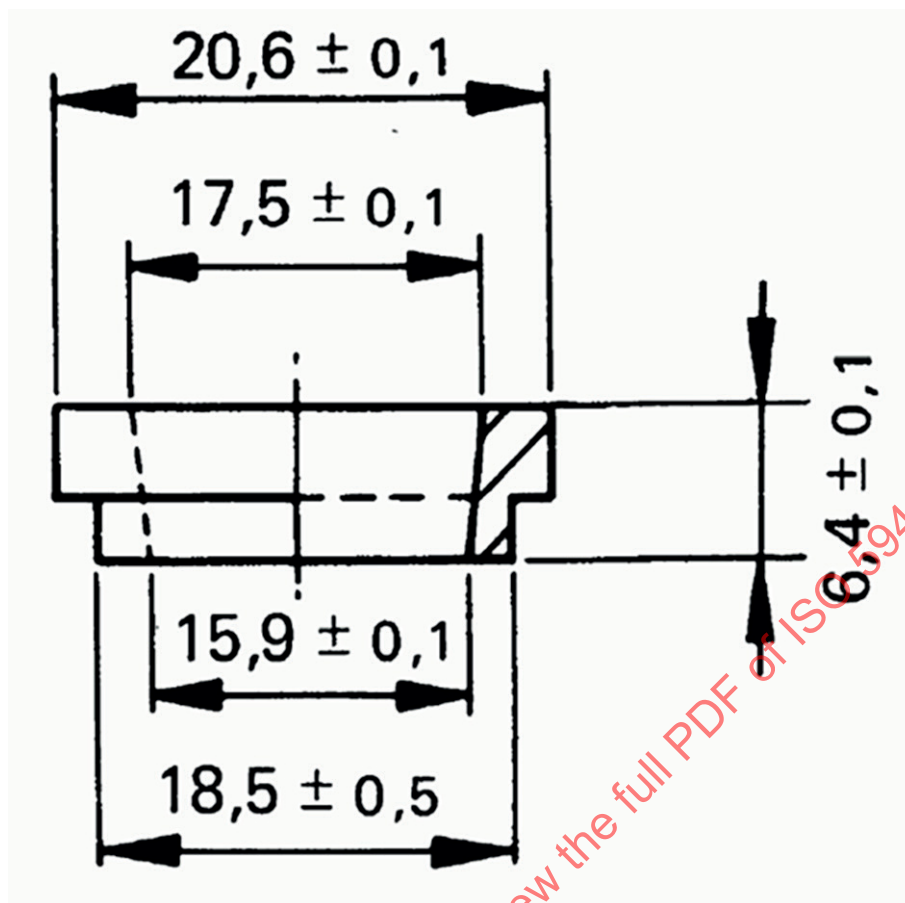


Figure B.2 — Tapered ring