
International Standard



5940

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Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of softening point by the ring-and-ball method

Produits carbonés utilisés pour la production de l'aluminium — Brai pour électrodes — Détermination du point de ramollissement par la méthode bille et anneau

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Descriptors : pitch (materials), electrodes, tests, softening tests, softening point, test equipment.

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5940 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in November 1978.

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

Australia	India	South Africa, Rep. of
Belgium	Israel	Sweden
Bulgaria	Italy	Switzerland
Czechoslovakia	Korea, Rep. of	Thailand
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France	New Zealand	United Kingdom
Germany, F. R.	Poland	USA
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Canada
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This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of softening point by the ring-and-ball method

1 Scope and field of application

This International Standard specifies 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 Reference

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

3 Principle

Preparation of 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.

Determination of 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).

4 Reagents

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

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

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

5 Apparatus

Ordinary laboratory apparatus and

5.1 Ring-and-ball apparatus (see figure 1 for assembled apparatus), comprising :

5.1.1 Rings, brass, tapered (see figure 2), having the following dimensions :

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

5.1.2 Steel balls, of diameter 9,53 mm, each weighing $3,50 \pm 0,05$ g.

5.1.3 Ball centring guides.

A suitable form of guide is shown in figure 3.

5.1.4 Bath; a heat-resistant glass squat form beaker of capacity 800 ml.

5.1.5 Ring support, having the shape and dimensions shown in figures 1 and 4.

The support consists essentially of two parallel brass plates, one above the other, and $25 \pm 0,5$ mm apart. The upper plate is designed to support the two rings (5.1.1) 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 thermometer (5.1.6).

5.1.6 Thermometer, solid-stem, mercury-in-glass type, having :

- a) a range from 0 to 120 °C for pitches which have softening points between 30 and 120 °C;
- b) a range from 50 to 210 °C for pitches which have softening points greater than 120 °C.

Details of suitable thermometers are given in annex A, but other thermometers of similar characteristics may also be used.

5.1.7 Tripod and open mesh wire gauze, supporting the bath (5.1.4).

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

5.1.9 Moulding plate, having the dimensions shown in figure 5.

5.2 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.

6 Procedure

6.1 Preparation of test piece

Using the method specified in annex B, melt a test portion of approximately 50 g of the laboratory sample (see ISO 6257, sub-clause 3.10) and heat the rings (5.1.1).

Place the heated rings in the sets of guide pins on the moulding plate (5.1.9), 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.

6.2 Preparation of the apparatus

Avoiding any aeration, place in the bath (5.1.4) a sufficient amount (600 ml is usually adequate) of either the glycerol (4.1) or the water (4.2) 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 (5.1.5) and fit this assembly into the bath. Insert the appropriate thermometer (5.1.6) 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 (5.1.3) above each ring and place two of the balls (5.1.2) in the bath, but not on the pitch contained in the rings. Place the beaker on the tripod (5.1.7) 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.

6.3 Determination

Place the Bunsen burner (5.1.8) 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 thermometer as indicated in figures 1a) and 1b).

Heat so that the temperature is raised by 5 °C each minute. Except for the first 3 min, it is essential to maintain this heating rate to within $\pm 0,5$ °C over each 1 min period. It is essential that this rate should not be merely the average over the period of the test. Reject any results in which the heating rate does not conform to these requirements.

Record the temperature at which the pitch surrounding each ball first touches the lower plate of the support. If, for the two balls, 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.

7 Expression of results

7.1 Method of calculation

The softening point is given by the mean of the two temperatures recorded as described in 6.3. Express the result to the nearest 0,2 °C.

7.2 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

8 Test report

The test report shall include the following particulars :

- an identification of the sample;
- the reference of the method used, stating whether glycerol or water was used as the heating medium;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operations not included in this International Standard, or in the International Standard to which reference is made, or regarded as optional.

Annex A

Details of thermometers

Range	0 to 120 °C	50 to 210 °C
Graduation	0,5 °C	0,5 °C
Immersion	100 mm	100 mm
Longer lines at each ¹⁾	1 and 5 °C	1 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

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

Annex B

Melting of pitch samples

B.0 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.1 Apparatus

B.1.1 Dish, 55 mm diameter, 35 mm deep.

Dishes sold for use with penetrometers are suitable.

B.1.2 Oven, capable of being maintained at 50 °C above the expected softening point of the sample.

B.2 Procedure

Weigh approximately 50 g of the laboratory sample, powdered if its softening point permits, into the dish (B.1.1) and place it in the oven (B.1.2) set at a temperature of 50 °C above the expected softening point of the pitch. Also place the rings (5.1.1) 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 6.1.

NOTE — 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, suggesting the presence of water, discard it and prepare another melt using a further 50 g of the laboratory sample, powdered if its softening point permits, which has been dried by allowing it to stand in an evacuated desiccator in the presence of a suitable desiccant.

Dimensions in millimetres

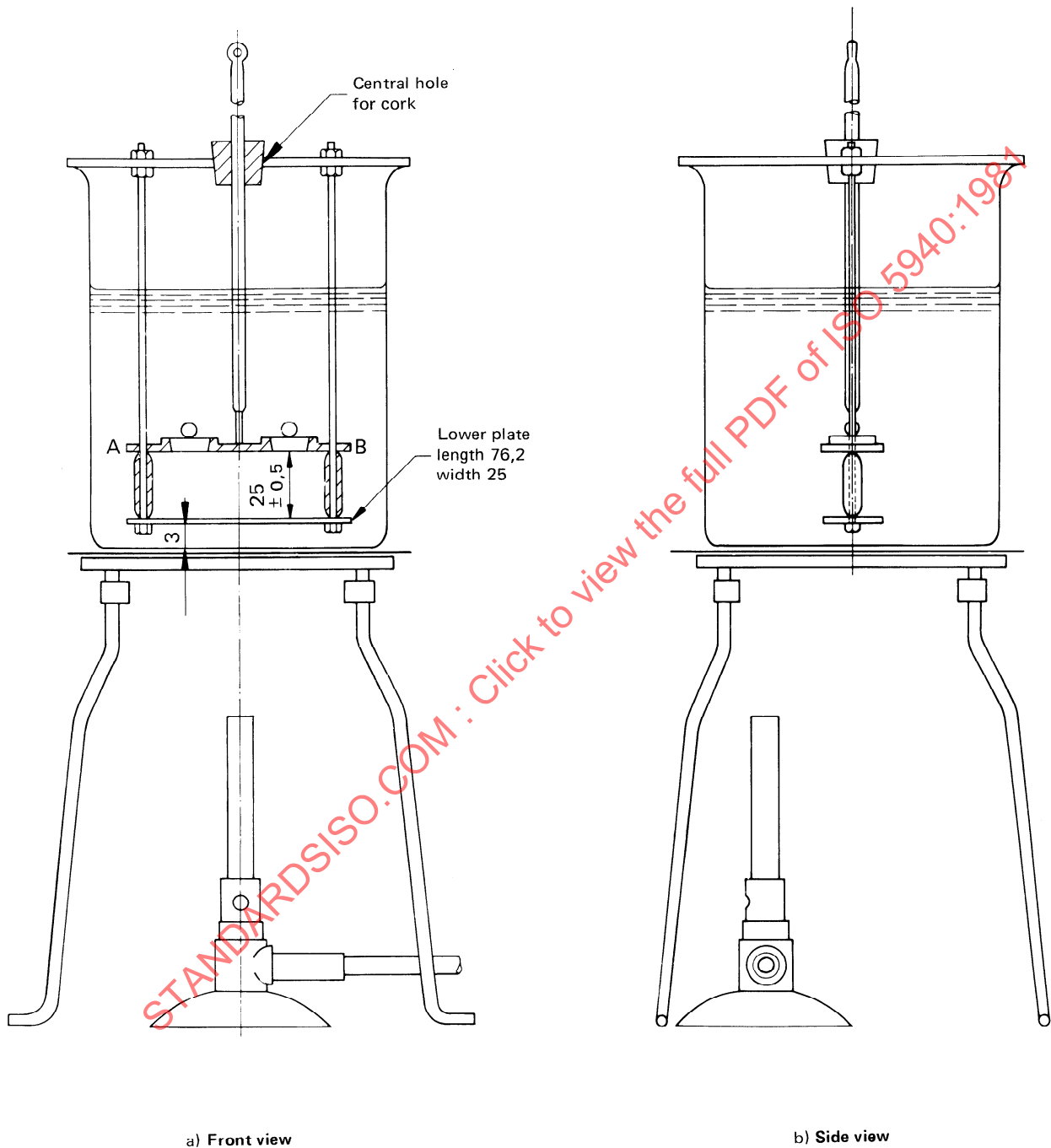


Figure 1 — Assembled apparatus

NOTE — Ball centring guides have been omitted for clarity.