INTERNATIONAL STANDARD

ISO 5940-2

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

Part 2: Determination of the softening point (Mettler softening point method)

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

Partie 2: Détermination du point de ramollissement (Point de ramollissement par la méthode Mettler)

Reference number
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Foreword

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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 5940-2 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

ISO 5940 consists of the following parts, under the general title *Carbonaceous materials used in the production of aluminium — Pitch for electrodes*:

— *Part 1: Determination of the softening point by the ring-and-ball method* (will replace ISO 5940:1981)

— *Part 2: Determination of the softening point (Mettler softening point method)*

Introduction

This part of ISO 5940 is based on DIN 51920 *Testing of carbonaceous materials — Determination of the Mettler softening point — Binding and impregnating materials*, prepared by NMP 281 "Prüfverfahren für Kohlenstoff und Graphit" and published by DIN, Deutsches Institut für Normung e.V., Berlin.

Coal-tar pitch is used as a binding and impregnating agent in the production of carbon anodes for primary aluminium metal production. When heated, pitch transforms gradually from a solid to a liquid. The analytical determination of a softening point described in this method is useful for characterizing this thermal behaviour of the coal-tar pitch when used as binder. The softening point is expressed as a temperature, in degrees Celsius (°C).

Annex A gives an approximate comparison with softening points determined by other methods.

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

Part 2: Determination of the softening point (Mettler softening point method)

1 Scope

This part of ISO 5940 specifies a method to determine the softening point of pitches by the Mettler method, within the range of 50 °C to 180 °C.

It can be used for other organic materials that are used as binder and impregnating agents, where they have a Mettler softening point within the range of 50 °C to 150 °C.

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

DIN 1333, *Presentation of numerical data*

DIN 51848-1, *Testing of mineral oils — Precision — General introduction, definitions and application to specifications*

3 Principle

A test portion of pitch is placed within a cylindrical sample cup with an outlet diameter of 6,35 mm. The temperature is increased under constant conditions in air, until softened material issues from the outlet and interrupts a light barrier 19 mm below.

4 Apparatus and reagents

4.1 Test apparatus[1] (see Figure 1) consisting of the parts given in 4.1.1 to 4.1.7.

4.1.1 Electrically heated furnace.

4.1.2 Sample cup (see Figure 2), made from chrome-plated yellow brass or chrome nickel-steel.

4.1.3 Resistance thermometer, capable of measuring temperatures from 50 °C to 180 °C.

4.1.4 Cup holder.

4.1.5 Light barrier, capable of sensing the material as it issues from the sample cup.

4.1.6 Control unit, with a temperature indicator for the thermometer (4.1.3), and an indicator to show the operational status of the machine.

4.1.7 Collector, for the softened material**.**

4.2 Bowl, made of porcelain, Teflon or metal with a volume of about 50 ml.

4.3 Plate, with a smooth surface, e.g. made of metal or glass.

4.4 Knife or spatula.

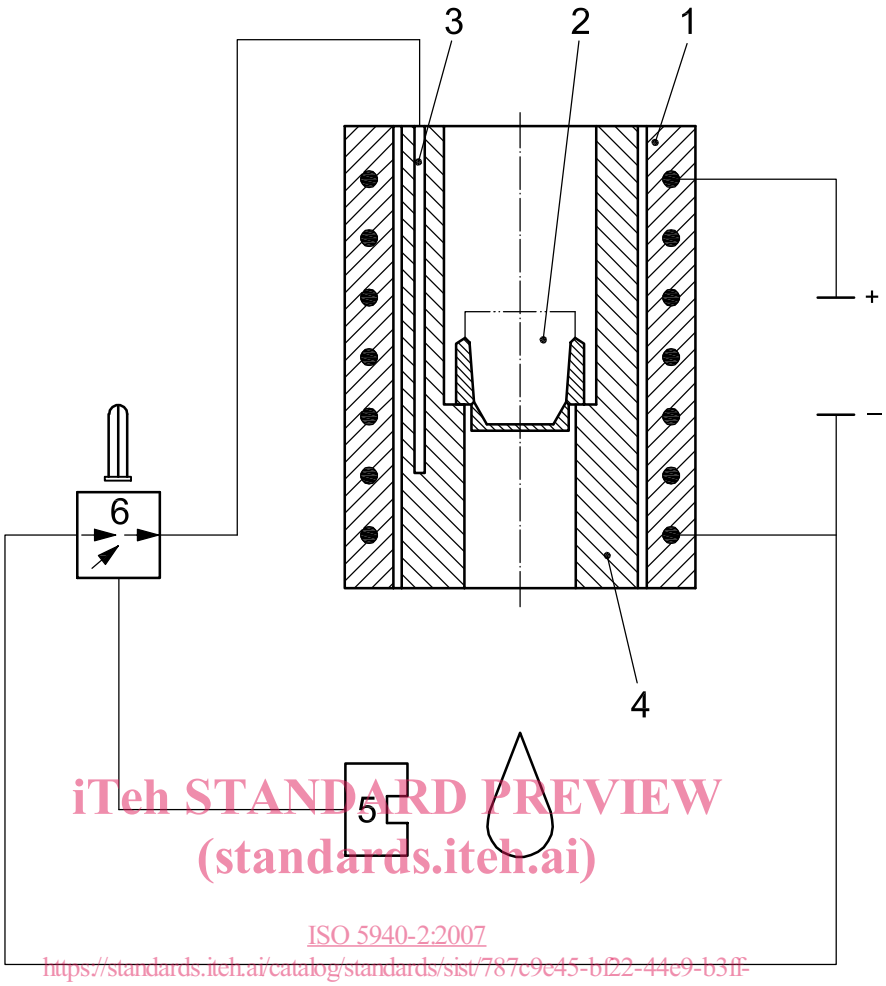
4.5 Release agent for cup, e.g. a mixture of glycerine and dextrin, ratio 1:1.

4.6 Electro-hydraulic press, as described in ASTM D3104, or another suitable press for filling the sample cup with grindable binder.

4.7 Punch, suitable for pressing grindable pitch.

4.8 Balance, capable of weighing grindable pitch to an accuracy of 0,1 g.

1) Available from Mettler Toledo Inc. http://www.mt.com. Further information on sources of supply can be obtained from: DIN-Bezugsquellen für normgerechte Erzeugnisse of DIN Deutsches Institut für Normung e. V., Burggrafenstraße 6, D-1000 Berlin 30. This information is given for the convenience of users of this part of ISO 5940 and does not constitute an endorsement by ISO of this apparatus.



Key

1 electrically heated furnace
2 sample cup
3 resistance thermometer
4 cup holder
5 light barrier
6 control unit with temperature indicator

Figure 1 — Schematic picture of the testing apparatus

Dimensions in millimetres

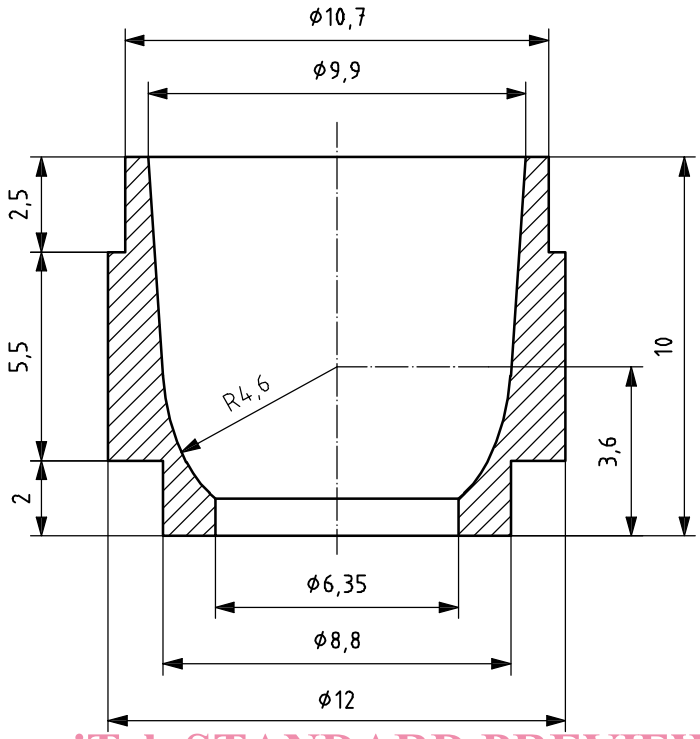


Figure 2 — Sample cup

5 Sampling

Take a sample of the material in accordance with ISO 6257.

6 Sample preparation

6.1 Types of material

Pitch can be grindable or non-grindable. Prepare non-grindable pitch by melting (see 6.2) and grindable pitch by melting or pressing (see 6.3).

6.2 Preparation of pitch by melting

Melt a test sample of about 20 g of the air-dry sample into the bowl (4.2), taking care to ensure that no overheating occurs at the wall of the vessel, and that no vapours pass off: Do not heat any part of the test portion of the sample to a temperature higher than 50 °C above the expected softening point. Remove air bubbles from the melt by carefully stirring, and remove any foam developing on the surface of the melt. Foam is an indication of water in the pitch. Keep the sample at the same temperature until foaming ends. Prepare the surface of the plate (4.3) by rubbing the release agent (4.5) onto the surface of the plate. Place the sample cup (4.1.2) onto the prepared surface of the plate. Take the bowl and molten test sample (6.1) and fill the sample cup by pouring the molten material from the bowl into the cup, such that it protrudes above the top of the sample cup by 1 mm to 2 mm. After cooling, level the sample surface with the warmed knife or spatula to become flush with the upper rim of the sample cup. Take care to avoid hollow spaces in the sample: do not simply cut away the protruding part of the sample. Remove the cup from the plate.

6.3 Preparation of pitch by pressing

Grind the sample so that the particle fraction ranges from 1 mm to 2 mm. Insert the sample cup in the cartridge assembly and fill the cup with ground material, use the punch to press the material in the cup slightly by hand, then insert the cartridge assembly in the hydraulic mould. Apply a force of 15 kN to the sample in the cup for at least 15 s. Remove the assembly from the mould; and loosen the sample cup by tapping the head of the punch.

Be aware that the temperatures at the measured softening points are lower by up to 1 °C, when using this procedure.

7 Procedure

7.1 Placing the sample in the apparatus

Assemble the filled sample cup with the sample cup holder and the collector sleeve to make a cartridge.

7.2 Operating the testing apparatus and expression of results

Choose a temperature 20 °C to 25 °C below the expected softening point as a starting temperature. Adjust the equipment to a heating rate of 2 °C per min. As soon as it is indicated that the apparatus is ready for operation, place the cartridge with the sample into the furnace and rotate until the collector sleeve is engaged. About 30 s later start the apparatus. At the end of the determination, the Mettler softening point is read from the storage of results on the apparatus, in degrees Celsius, to the nearest 0,1 °C.

Rounding to the last significant decimal place shall be done in accordance with DIN 1333.

7.3 Calibrating the temperature

7.3.1 Using a thermometer or thermocouple

The temperature indication at the operating unit can be verified as follows: The ball of a suitable thermometer or the tip of a thermocouple is put into the sample cup and cast with pitch. Then the lower part of the sample holder, with the sample cup and thermometer or thermocouple, is put into the furnace and heated to a definite temperature. 5 min after the selected temperature is reached, the reading of the suitable thermometer or thermocouple shall be not more than 0,5 °C below the reading of the resistance thermometer (4.1.3) on the control unit. After a longer duration, the difference of the readings should approach zero.

If the furnace is heated with a constant temperature rate of 2 °C/min (as in the procedure for determining the softening point), the suitable thermometer or thermocouple can be expected to indicate a temperature 1 °C to 1,5 °C less than the control unit.

7.3.2 Using benzoic acid on units with a calibration program

This test can be run every quarter or half year, or after adjustments. Place the test-portion holder on an even surface and fill with benzoic acid. Pack firmly using a 10 mm diameter glass rod. Select the calibration program and run the analysis, but with a start temperature of 121 °C and a temperature increase of 0,2 °C/min. The results should be 123,5 ± 0,5 °C. If not, the apparatus must be adjusted.

NOTE Pitch on the outside of the sample cup or inside the furnace will fume during the measurement and the fumes can disturb the photocell. This can cause a measurement result that is too low.