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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 26930.7-2014

**Carbonaceous materials used in the production of aluminium -
Pitch for electrodes - Part 7: Determination of the softening
point (Mettler softening point method)**

原铝生产用炭素材料 煤沥青 第7部分：软化点的测定（Mettler法）

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Foreword Foreword

GB/T 26930, Carbonaceous materials used in the production of aluminium - Pitch for electrodes, is made up of 13 parts: Part 1: Determination of the water content - Azeotropic distillation method; Part 2: Determination of the softening point - Ring-and-ball method; Part 3: Determination of the density - Pyknometer method; Part 4: Determination of the quinoline-insoluble content; Part 5: Determination of the toluene-insoluble content; Part 6: Determination of ash; Part 7: Determination of the softening point (Mettler method); Part 8: Determination of the coking value; Part 9: Determination of the sulfur content by oxygen bomb combustion; Part 10: Determination of the sulfur content by the instrumental method; Part 11: Determination of the dynamic viscosity; Part 12: Determination of the volatile matter content; Part 13: Determination of the C/H atomic ratio in the quinoline insolubles. This part is Part 7 of GB/T 26930.

This part is drafted according to the rules given in GB/T 1.1-2009. It adopts ISO 5940-2:2007, Carbonaceous materials used in the production of aluminium - Pitch for electrodes - Part 2: Determination of the softening point (Mettler softening point method), by the translation method and is identical with it. The Chinese document having a consistent correspondence with the international document referred to normatively in this part is GB/T 26297.5-2010, Sampling method for carbonaceous materials used for aluminium - Part 5: Coal tar pitch (ISO 6257:2002, MOD).

This part is under the jurisdiction of the National Technical Committee on Nonferrous Metals of Standardization Administration of China (SAC/TC 243).

1 Scope

This part of GB/T 26930 fixes the determination of the softening point of coal tar pitch by the Mettler method, over the measuring range 50 °C to 180 °C.

This part applies to the determination of coal tar pitch whose Mettler softening point lies between 50 °C and 180 °C. It also applies to the determination of other organic materials used as binders or as impregnating agents whose Mettler softening point lies between 50 °C and 150 °C.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the edition cited applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

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; DIN 52011 Determination of the ring-and-ball softening point of bitumen; DIN 52025 Testing of carbonaceous materials - Determination of the Kraemer-Sarnow softening point; ASTM D2319 Standard test method for softening point of pitch (cube-in-air method); ASTM D3104 Standard Test Method for Softening Point of Pitches (Mettler Softening Point Method).

3 Principle of the method

The pitch test portion is placed above the outlet of a cylindrical sample cup and heated in an air bath until the test portion softens and flows out of the outlet, covering the light barrier 19 mm below it.

4 Apparatus and reagents

4.1 Measuring device (see Figure 1): made up of the parts given in 4.1.1 to 4.1.7.

4.1.1 Electric furnace.

4.1.2 Sample cup (see Figure 2): made of chromium-plated brass or of chromium-nickel steel, the diameter of the cup outlet being 6.35 mm.

4.1.3 Resistance thermometer: measuring temperature range 50 °C to 180 °C.

4.1.4 Sample cup holder.

4.1.5 Light barrier: sensing material able to sense the pitch flowing out of the sample cup.

4.1.6 Control unit with temperature indication: made up of the resistance thermometer

(4.1.3) and of an indicator lamp able to display the operating state of the equipment.

4.1.7 Collector: used to collect the test portion that falls after softening.

4.2 Bowl: made of ceramic, of polytetrafluoroethylene or of metal, of about 50 mL capacity.

4.3 Flat plate: with a smooth surface, made of metal or of glass.

4.4 Spatula.

4.5 Release agent: for example a mixture of glycerol and dextrin in the ratio 1:1.

4.6 Electric hydraulic press: such as the one described in ASTM D3104, or a suitable press able to force grindable pitch into the sample cup.

4.7 Punch press: suitable for pressing grindable pitch.

4.8 Balance: accurate to 0.1 g.

4.9 Benzoic acid.

Figure 1 is a schematic drawing of the measuring device, with the key naming the electric furnace, the sample cup, the resistance thermometer, the sample cup holder, the light barrier and the control unit with temperature indication. Figure 2 is a sectional drawing of the sample cup with dimensions in millimetres; besides the outlet diameter of 6.35 mm already given in 4.1.2, the remaining diameters, heights and the fillet radius appear only as dimension lines on the drawing and are not reproduced here.

5 Sampling

Sampling shall be carried out according to the provisions of ISO 6257.

6 Preparation of the test portion

6.1 Type of pitch. Pitch can be divided into grindable pitch and non-grindable pitch. Non-grindable pitch is treated by melting (see 6.2); grindable pitch is treated by melting or by moulding under pressure (see 6.3).

6.2 Preparation of the pitch test portion by melting. Weigh about 20 g of the air-dried test sample and melt it in the bowl (4.2), taking care that the bowl wall does not overheat and that no vapour is produced, and never heating any part of the sample more than 50 °C above the expected softening point. Stir the melted sample carefully to remove air bubbles, and remove the foam produced on the surface of the melted sample; foam indicates that the pitch contains water. Keep the sample warm until the foam in the pitch has disappeared. Apply the release agent (4.5) to the surface of the flat plate (4.3), set the sample cup (4.1.2) on the prepared flat plate, and pour the sample melted in the bowl (6.1) so as to fill the sample cup, with the melt standing 1 mm to 2 mm proud of the top of the cup. After cooling, scrape the cup mouth with a preheated spatula so that the surface of the sample is level

with the upper edge of the cup; this is not a simple cutting away of the protruding part of the sample, and cavities in the sample are to be avoided as far as possible. Finally remove the sample cup from the flat plate.

6.3 Preparation of the pitch test portion by moulding under pressure. Grind the sample to a particle size of 1 mm to 2 mm, place the sample cup in the sample box, fill the ground test portion into the sample cup and press the test portion in the sample cup gently by hand, then put the sample box into a hydraulic die. Apply to the test portion in the sample cup a force of 15 kN for at least 15 s, take the sample box out of the hydraulic die and open the head of the electric hydraulic press so as to release the sample cup. Note that when this preparation procedure is used the softening point measured is lower by 1 °C or more.

7 Determination procedure

7.1 Assembly of the sample box. The sample cup filled with the test portion, the sample cup holder and the collector sleeve are assembled into the sample box.

7.2 Instrument operation and expression of the result. Choose as starting temperature a temperature 20 °C to 25 °C below the expected softening point and set the heating rate of the equipment to 2 °C/min. When the instrument indicates that it is ready to run, put the sample box into the furnace at once and start the rotation until the collector sleeve is filled (about 30 s after the instrument has been switched on); at the end of the determination read the Mettler softening point from the results stored in the instrument, to 0.1 °C. Round the result to one decimal place as provided in DIN 1333.

7.3 Temperature calibration.

7.3.1 Calibration with a thermometer or a thermocouple. The temperature display of the control unit can be calibrated by the following method: insert the tip of a suitable thermometer or thermocouple into the sample cup and into the pitch, then put the lower part of the holder carrying the sample cup, together with the thermocouple or thermometer, into the furnace and heat it to a given temperature; 5 min after the preset temperature has been reached, read the thermometer or the thermocouple; its reading is lower than the temperature displayed by the control unit, but the difference is not greater than 0.5 °C. After a longer time the difference between the two readings should approach zero. If the furnace is heated at the constant rate of 2 °C/min, as in the heating programme used when the softening point is determined, the reading of a suitable thermometer or thermocouple is between 1 °C and 1.5 °C lower than the temperature displayed by the control unit.

7.3.2 Calibration with benzoic acid. The calibration test may be carried out every quarter, every half year or after servicing. Place a specimen holder on a flat surface and then fill it with benzoic acid. Tamp it with a glass rod 10 mm in diameter, select the calibration programme and start the analysis, with a starting temperature of 121 °C and a heating rate of 0.2 °C/min; under these conditions the result of the determination is 123.5 °C +/- 0.5 °C,