ICS 71.100.10
CCS Q 52

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 26930.11-2014

Carbonaceous materials used in the production of aluminium - Pitch for electrodes - Part 11: Determination of dynamic viscosity

原铝生产用炭素材料 煤沥青 第11部分：动态粘度的测定

Issued on: July 24, 2014 **Implemented on: April 1, 2015**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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1 Scope

This part of GB/T 26930 lays down two methods for determining the dynamic viscosity of coal tar pitch used in the production of primary aluminium. For pitch with Newtonian flow behaviour the two methods are equally valid and either of them may be used to express the result.

Method A, the ball displacement method, applies to pitch with Newtonian flow behaviour, such as pitch to which no other material has been added when the softening point is determined, and is carried out at temperatures above the softening point but below 180 °C. Its measuring range runs from 100 mPa s to 10 000 mPa s.

Method B, the cylinder rotation method, applies both to pitch with Newtonian flow behaviour and to pitch that does not behave as a Newtonian fluid because a mesophase is present, and is also suitable whenever the measurement has to be made at a higher temperature, above 180 °C. The method makes it possible to tell whether a pitch behaves as a Newtonian fluid. Its measuring range is not more than 3 000 000 mPa s.

2 Normative references

The documents listed below cannot be dispensed with for the application of this part. For dated references only the edition corresponding to that date, including any amendments, applies; for undated references the latest edition applies.

ISO 6257 Carbonaceous materials used in the production of aluminium - Pitch for electrodes - Sampling.

ISO 6388 Surface active agents - Determination of flow properties using a rotational viscometer.

3 Method A - principle, apparatus and procedure

3.1 Principle. At a given temperature the time is measured that a ball takes, under a given force, to travel a fixed distance through the measuring tube filled with the molten pitch; the viscosity is obtained from that time.

3.2.1 The viscometer (see Figure 1) is built like a two-arm balance and takes in: a support frame with knife-edge bearings at the top; a balance beam with knife edges; a weighing pan; a pointer at least twice the length of the beam arm, so that the ball can travel twice that distance; a dial with a reflecting background graduated to 1 mm, whose measuring span is 50 mm; a spirit level; a levelling screw; a movable index that can be set to the mark S; a stainless steel ball 15.00 mm +/- 0.02 mm in diameter; a stainless steel rod 2 mm in diameter; a stainless steel measuring tube of inside diameter 16.15 mm +/- 0.05 mm, outside diameter 20 mm and length 200 mm; a stainless steel jacket of 90 mm inside diameter for temperature calibration; two precision thermometers covering 99 °C to 151 °C and 149 °C to 201 °C, readable to 0.1 °C, with an immersed length of 140 mm +/- 5 mm.

3.2.2 Thermostatic bath: highest working temperature 250 °C, control accuracy +/- 0.1 °C, capacity at least 3 L, filled with a heat transfer oil such as silicone oil.

3.2.3 Stopwatch: accurate to 0.1 s.

3.2.4 A set of weights, of masses from 5 g to 500 g.

3.2.5 Lead shot, used for balancing.

3.2.6 Calibration liquid: 50 mL of a liquid with Newtonian flow behaviour whose dynamic viscosity lies between 1 Pa s and 30 Pa s.

3.3 Sampling is carried out in accordance with ISO 6257.

3.4.1 Preparation. The whole apparatus (3.2.1) is cleaned and dried and is levelled with the spirit level, using the levelling screw. The viscometer is placed in the thermostatic bath and the thermometer is connected. When the temperature of the outlet of the spray tube reaches the measuring temperature, the temperature of the bath is raised to the measuring temperature, the pitch is poured into the measuring tube to about 25 mm from its upper end, and the preheated stainless steel ball and rod are then placed in the measuring tube;

circulating heating is continued for about 30 min. The ball is moved up and down continuously to speed up the adjustment of the temperature; after 10 min of circulating heating the measuring temperature is held stable to +/- 0.1 °C and the movable index is set to the mark S.

3.4.2 Balancing the beam. After 30 min of circulating heating, lead shot (3.2.5) is placed in the weighing pan until the pointer stands in the vertical position, that is at half of the span of the dial.

3.4.3 Measurement. Weights (3.2.4) with masses between 5 g and 500 g are placed in the weighing pan, chosen so that the time taken by the pointer to travel from the zero point to the mark S is between 20 s and 60 s. Before the measurement, the ball and the rod are preheated and placed in the measuring tube, and the time in seconds is recorded with the stopwatch for the pointer to pass between the two marks on the dial while the ball travels through the tube filled with the liquid of known viscosity. The measurement is repeated five times.

3.5 Calculation of the results. The mean of the five measured times is calculated and the dynamic viscosity of the pitch, in millipascal seconds, is obtained from formula (1). The legend printed under the formula is: the dynamic viscosity of the pitch, in millipascal seconds; a constant; the mass of the weights, in grams; and the mean of the times of the five measurements, in seconds. Weights of different mass used on the same pitch sample at the same temperature may give somewhat different results, but the deviation is not to exceed +/- 1 %.

3.6.1 Cleaning the measuring tube. The screw cap is taken off and the liquid pitch is collected in a beaker; a piston fitted with a rubber disc of 16.5 mm diameter is drawn through the measuring tube, and the tube wall is then cleaned with a solvent.

3.6.2 Cleaning the ball and the rod. The ball and the rod are wiped with a cloth while they are hot and soft. Hard objects are not to be used, since even a slight scratch affects the accuracy of the mass of the ball.

3.7 Calibration. A liquid of known viscosity is used, the steps of 3.4 are followed and the movable index is set at 70 mm. The scale value S, in millimetres, is calculated from formula (2), whose legend is: the scale value, in millimetres; the viscosity of the calibration liquid, in millipascal seconds; and the same three quantities as in formula (1).

3.8 Precision. The maximum permissible error of the method is +/- 1 %.

4 Method B - definitions, principle and apparatus

4.1 Definitions and symbols. The shear viscosity of a fluid between two parallel plates, that is when the movement of one plate relative to the other is approximately linear, is defined by the Newton equation (3). The legend printed under it is: the dynamic viscosity; the shear

stress; and the shear rate. The shear rate is itself defined by equation (4), whose legend is: the velocity of one plate relative to the other; and the perpendicular distance between the two plates. Substituting in the Newton equation gives the dynamic viscosity as equation (5). The unit of dynamic viscosity is the pascal second or the millipascal second.

4.2 Principle. Use is made of a system in which a conical body rotates inside a cylindrical vessel holding the substance, so that the shear stress of the substance is measured at a given temperature and at a chosen speed of rotation.

4.3.1 Rotational viscometer, whose main parts are: a variable-speed drive motor, able to transmit the rotary movement to a rotor immersed in the liquid, with an indicator of the speed of rotation of the rotor immersed in the liquid; the rotor and the vessel holding the liquid are to be coaxial; a set of four conical elements, each fitted with a suitable coupling so that it can be driven by the motor; a heating jacket and a thermocouple; and the calculation tables that come with the equipment.

Relationship Relationship to other documents

The foreword states that GB/T 26930, Carbonaceous materials used in the production of aluminium - Pitch, is divided into thirteen parts, and that this text is Part 11.

The part is a translation-identical adoption of ISO 8003:1985, whose foreword has been deleted. The foreword also records the corresponding national document for the international document normatively referred to: GB/T 26297.5-2010, which corresponds to ISO 6257:2002 (MOD), on sampling of pitch.