International Standard ISO 8003

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

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

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

1 Scope and field of application

This International Standard specifies two methods of determining the dynamic viscosity of a sample of pitch, which is used as a binder for the manufacture of electrodes required in the electrolytic production of aluminium. The two methods have the same validity for pitches of Newtonian nature and, for these pitches, they are presented as alternative methods.

— **Method A** (Ball displacement method)

The method is applicable to pitches of newtonian nature, i.e. containing no additional material. The measurement of the viscosity shall be carried out at a temperature greater than the softening temperature and not greater than 180 °C.

The measuring range of the method is 10^2 to 10^4 mPa·s.

— **Method B** (Rotation of a cylindrical body method)

The method is applicable to the pitches indicated for method A and to pitches for which a non-newtonian nature may be assumed, either because they have been treated at high temperature (presence of mesophase) or due to the necessity of being examined at high temperature (greater than 180 °C). This method allows the non-newtonian nature of a pitch to be characterized.

The method is applicable to pitches having a maximum viscosity of 3×10^6 mPa·s.

2 References

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

ISO 6388, *Surface active agents — Determination of flow properties using a rotational viscometer.*

3 Method A

3.1 Principle

Determination of the viscosity of a pitch at a given temperature determined using a sample of pitch having similar characteristics by measurement of the time a ball, drawn by a defined force, requires to traverse a fixed distance within a measuring tube filled with the sample of molten pitch.

The test results are given as dynamic viscosity in absolute units of "millipascal seconds" (mPa·s)*.

3.2 Apparatus and materials

3.2.1 Viscometer (see the figure)

The viscometer is designed similar to a two-arm lever balance consisting of the following items:

— a support (15) with a knife-edge bearing at the top;

— a ball (2), diameter 15,00 ± 0,02 mm, of stainless steel (C ⩽ 0,07 %, Si ⩽ 1,00 %, Mn ⩽ 2,00 %, P ⩽ 0,045 %, S ⩽ 0,03 %, 17,00 ⩽ Cr ⩽ 20,00 %, 8,50 ⩽ Ni ⩽ 10,00 %, coefficient of linear expansion 17×10^{-6} K^{-1});

— a rod (13), diameter 2 mm, of stainless steel [same composition as for the ball (2)];

— a measuring tube (1), internal diameter 16,15 ± 0,05 mm, external diameter 20 mm, length 200 mm, of stainless steel [same composition as for the ball (2)];

— a jacket tube (14) for thermostatic regulation, internal diameter 90 mm, of stainless steel [same composition as for the ball (2)];

— an entrance and exit nozzle (11);

— a screw cap with stopper (12);

— two precision thermometers (8):

range: 99 to 151 °C, maximum error: 0,1 °C,

range: 149 to 201 °C, maximum error: 0,1 °C,

length of the immersed part: 140 ± 5 mm;

— a balance lever (4) with knife-edge supports;

— a weight pan (3);

— a pointer (5), the length of which must be at least twice as long as the lever arm and which is chosen in such a way that the distance covered by the pointer is twice that covered by the ball;

* 1 mPa·s = 1 cP (centipoise)

— a scale (6) with a reflecting background and a millimeter graduation. The scale mark on the extreme left is 100 mm away from the zero mark. This pointer path, therefore, corresponds to a ball path of 50 mm, half of the pointer path corresponds to a vertical position of the pointer;

— a water level (7);

— levelling screws (10);

— an adjustable mark (9) which should be moved to a scale value S.

3.2.2 Thermostat (16)

Maximum operating temperature: 250 °C, temperature control accuracy: ±0,1 °C, capacity: at least 3 l, filled with a suitable heat-carrier oil (for example silicone oil).

3.2.3 Stop-watch

Graduation: 0,1 s.

3.2.4 Set of weights

Range: 5 to 500 g.

3.2.5 Lead shot

To tare the balance.

3.2.6 Calibration liquids

50 ml of a liquid product having a newtonian nature and a viscosity situated between 1 to 30 Pa·s.

3.3 Sampling and samples

Sample in accordance with ISO 6257.

3.4 Procedure

3.4.1 Preparation

Clean and dry the whole apparatus (3.2.1). Put the support for the balance lever (4 of the figure) in a vertical position by using the levelling screws (10 of the figure) and the water level (7 of the figure). Put the thermometer (8 of the figure) into the jacket tube (14 of the figure). Connect the thermostat (16 of the figure) to the entrance and exit nozzles (11 of the figure) as indicated in the figure (the exit nozzle has not been indicated). Raise the temperature of the thermostat to the measuring temperature. Heat the pitch sample to the measuring temperature. When the thermometer indicates that the measuring temperature has been reached pour the pitch sample into the measuring tube (1 of the figure) and fill it up to about 25 mm below the upper rim. Preheat the ball (2 of the figure) with the rod (13 of the figure) and then put it into the tube. Continue the heating to temper the apparatus for approximately 30 min.

From time to time move the ball up and down for faster temperature adjustment. During the last 10 min of tempering, keep the temperature within the range of the measuring temperature ±0,1 °C. Set the adjustable mark (9 of the figure) to the scale value S.

3.4.2 Taring the balance

After 30 min of tempering, put the lead shot (3.2.5) on the weight pan (3 of the figure). The necessary equilibrium is reached when the pointer remains in a vertical position, which corresponds to half of the pointer path.

The time required for taring the balance, i.e. the time required for the displacement of the ball between the two marks of the measuring tube in a liquid of known viscosity, shall be greater than the measuring time.

3.4.3 Measurement

For the measurement, place an additional weight (3.2.4) (not less than 5 g, not more than 500 g) on the weight pan. Choose the additional weight so that the pointer covers the distance between the two measuring marks (zero and point S) within 20 to 60 s.

Before measuring, sink the ball rod into the tube until the pointer position is about 20 mm before the zero mark. Then the additional weight on the weight pan pulls the ball upwards through the liquid sample. Note with the stop-watch (3.2.3) the time t (seconds) required by the pointer to cover the distance between the two measuring marks on the scale. Repeat the measuring five times.

3.5 Evaluation of the test results

Calculate the mean value t_m of five single test results. Then calculate the dynamic viscosity η of the pitch, expressed in millipascal seconds, using the equation

$$\eta = F \cdot m \cdot t_m$$

where

F is the ball factor, expressed in millinewtons per gram metre squared [F = 1 mN/(g·m^2)];

m is the additional weight, in grams;

t_m is the mean value of time, in seconds.

Using different additional weights, the viscosity measured for the same sample of pitch at a constant temperature may change between the precision limits of the method, i.e. not more than ±1 %.

3.6 Cleaning the apparatus

3.6.1 Cleaning the measuring tube

Take away the screw cap with stopper (12 of the figure). Collect the liquid pitch in a beaker. Draw through the measuring tube a piston-shaped rubber disk with a diameter of 16,5 mm. Wash away by a solvent the remaining pitch film on the wall of the tube.

3.6.2 Cleaning the rod and ball

Clean the rod and ball with a soft cloth when both are still hot. Do not use hard tools as the slightest damage to the ball leads to inaccuracies.

3.7 Calibration

Use a liquid of a known viscosity η_0. This viscosity should lie in the range of the viscosity of the pitch samples to be measured. Repeat the whole procedure as described in 3.4. Set the adjustable mark to 70 mm.

Calculate the scale value S, in millimetres, using the equation

$$S = \frac{\eta_0 \times 70}{F \cdot m \cdot t_m}$$

where

F, m and t_m have the same meanings as in 3.5;

η_0 is the viscosity of the calibration liquid, in millipascal seconds.

3.8 Precision

The limit error of the method is ± 1 %.

4 Method B

4.1 Definitions and symbols

The viscosity of a fluid sheared between two parallel planes, one of which moves in its own plane with linear and uniform motion relative to the other, is defined by Newton's equation:

$$\eta = \frac{\tau}{D}$$

where

η is the dynamic viscosity;

τ is the shear stress;

D is the rate of shear equal to

$$D = \frac{dv}{dz}$$

v being the velocity of one plane relative to the other;

z being the co-ordinate perpendicular to both planes.

Substituting in Newton's equation, the dynamic viscosity becomes

$$\eta = \frac{\tau}{D} = \frac{\tau}{\frac{dv}{dz}} = \frac{\tau}{dv} \times dz$$

The dynamic viscosity is expressed in pascal seconds or millipascal seconds.

4.2 Principle

Measurement of the shear stress of a substance at a selected rotational speed at a given temperature by means of a system composed of a cylindro-conical body revolving in the substance contained in a cylindrical vessel.

4.3 Apparatus

Ordinary laboratory apparatus, and

4.3.1 Revolving viscometer, including the principal following items:

— electric driving motor, with adjustable speed, capable of transmitting a revolving motion to the body immersed in the fluid. The rotating body and the vessel containing the fluid are coaxial and the system comprises an indicator giving the rotational speed of the immersed body;

— set of 4 cylindro-conical bodies each with an appropriate device for coupling to the driving motor;

— heating jacket and thermocouple;

— calculation tables, specific to the apparatus used, and supplied with it.

NOTE — Since these viscometers, which are commercially available, have some constructional differences, although based on the same principle, it is impossible to give more descriptive details. (However, see ISO 6388.)

4.3.2 Thermostatic bath, with automatic temperature regulation, filled with an appropriate oil (e.g. silicone oil).

4.4 Sampling and samples

Sample in accordance with ISO 6257.

4.5 Procedure

4.5.1 Test portion

Melt 150 g of the sample at a temperature near the measuring temperature and pour it in the cylindrical vessel.

4.5.2 Measurement

NOTE — The descriptive operation notices are delivered with the apparatus, and take the construction details into account. These directions must therefore be rigorously followed. Nevertheless, general principles of the procedure are given hereafter.

Introduce a rotating body having the proper characteristics for the presumed viscosity into the vessel containing the molten sample (4.5.1).

Preselect on the thermostatic bath scale (4.3.2) the temperature at which the measurement will be made.

Dip the thermocouple (4.3.1) into the molten sample, allow the system to reach the selected temperature and maintain at this temperature during a time varying with the softening point of pitch, but not less than 1 h.

Remove the thermocouple in order to avoid any friction during the measurement.

Select the rotational speed.

Set the rotating body in motion at the selected rotational speed.

Read on the indicator of the motor the rotational speed at regular intervals.

Take as the result the mean of at least two consecutive readings.

4.6 Expression of results

Following the relations indicated in 4.1, the calculation tables of the apparatus give, for each body measured, and for each rotational speed, either a coefficient allowing the calculation of the viscosity, or directly the viscosity expressed in pascal seconds.

4.7 Precision

The limit error of the method is ±1 %.

5 Test report

The test report shall include the following particulars:

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) the temperature at which the determination has been carried out;

e) any unusual features noted during the determination;

f) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

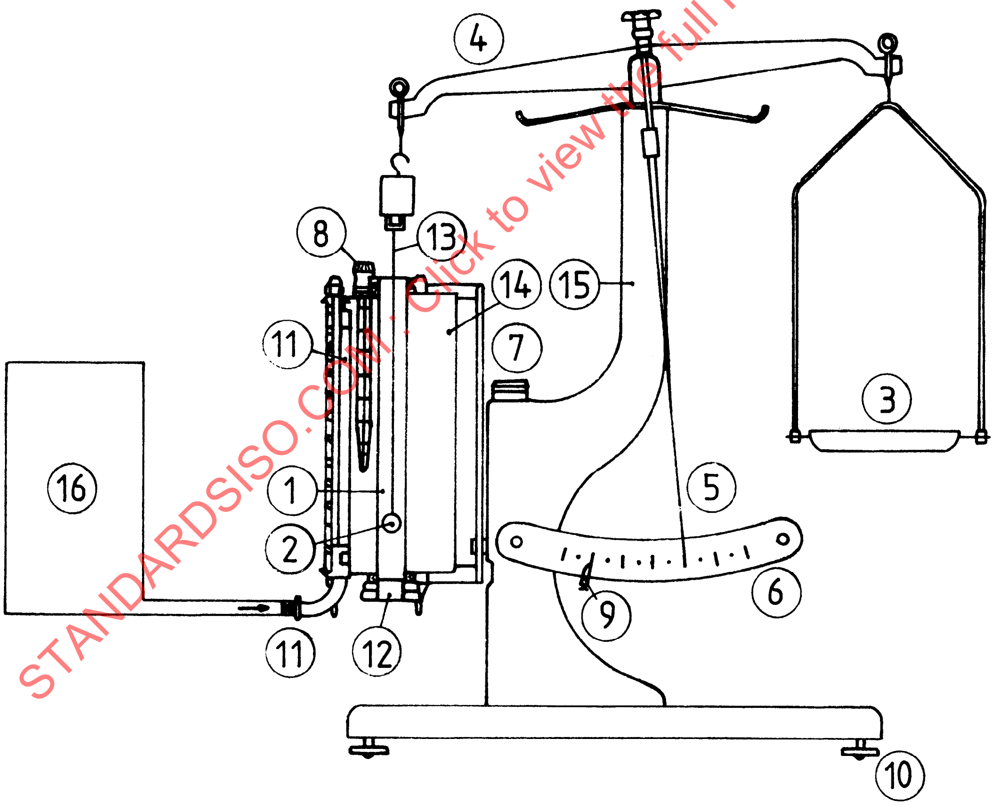

Figure — Apparatus for method A