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Carbonaceous materials used for the production of aluminium — Cathode blocks and prebacked anodes — Determination of the density in xylene by a pyknometric method

Produits carbonés utilisés pour la production de l'aluminium — Blocs cathodiques et anodes précuites — Détermination par pycnométrie de la masse volumique en xylène

Foreword

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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.

International Standard ISO 9088 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

Carbonaceous materials used for the production of aluminium — Cathode blocks and prebaked anodes — Detemination of the density in xylene by pyknometric method

1 Scope

This International Standard specifies a pyknometric method for the determination of the true density in xylene of cathode blocks and prebacked anodes used for the production of aluminium.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3507:1976, *Pyknometers.*

ISO 8007:1985, *Carbonaceous materials used in the production of aluminium — Sampling from cathodic blocks and prebaked anodes — General.*

3 Principle

The true density of cathode blocks and prebaked anodes is measured in xylene in a pyknometer after degassing under a vacuum.

4 Reagents and materials

During the determination, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity, degassed by boiling for 1 h.

4.1 Ethanol, 95 % (*V/V*).

4.2 Acetone.

4.3 Sulfuric acid, density approx. 1,84 g/ml, about 98 % (*m/m*) solution.

4.4 Xylene, commercial grade, density about 0,86 g/ml.

WARNING — Xylene burns the skin and can also be absorbed into the system through the skin. In addition, inhalation of the vapour from hot material is to be avoided.

5 Apparatus

5.1 Pyknometer, type 3 as defined in ISO 3507 (Gay-Lussac), capacity 25 ml.

5.2 Degassing apparatus (see figure 1), comprising the following items:

5.2.1 Container (1) for the pyknometer, consisting of a glass beaker (2) with removable lid (3) and O-ring (4), capable of containing the pyknometer without its stopper. The container outlet (5) is connected to a pump (not shown).

5.2.2 Filling device (6), fitted to the container by a ground-glass joint (7). A tube (8) extends down into the pyknometer. A reservoir (9) with a ground-glass stopper (10) contains the pyknometer liquid which is allowed to flow into the pyknometer through a teflon valve (11).

5.2.3 Stand (12), to support the beaker when no vacuum is applied to the degassing apparatus. A rod (13) with a spring (14) allows the beaker (2),with the pyknometer in it, to be shaken to facilitate the evolution of a gas bubbles during degassing.

The apparatus is made of glass. A rotary pump is connected to the outlet of the apparatus via an oil trap (15). A manometer (16) is connected to the vacuum system between the pump and the oil trap. The vacuum is adjusted so that the manometer, which is about 600 mm from the joint (7) between the container and the filling device, registers 1,3 kPa ± 0,3 kPa. A valve (17) is fitted to allow air back into the apparatus. This has to be done slowly and with due care.

NOTE — At a pressure of 1,3 kPa ± 0,3 kPa and an ambient temperature of 25 °C, slight vapourization of xylene may occur until equilibrium is reached, but this is of no consequence.

The apparatus described above is only an example; other apparatus with the same characteristics may be used.

5.3 Drying oven, capable of being maintained at 120 °C ± 2 °C.

5.4 Thermostatically controlled bath, capable of being maintained at 25 °C ± 0,05 °C.

5.5 Grinder, capable of grinding the sample to a particle size of less than 63 μm. The parts which come into contact with the sample shall be made of hardmetal to avoid contamination.

5.6 Filter paper.

5.7 Jaw-type crusher.

6 Sampling

Sample in accordance with ISO 8007.

Dimensions in millimetres

Key

1) Container
2) Glass beaker
3) Lid
4) O-ring
5) Outlet
6) Filling device
7) Ground-glass joint
8) Tube
9) Reservoir

10) Ground-glass stopper
11) Teflon valve
12) Stand
13) Rod
14) Spring
15) Oil trap
16) Manometer
17) Valve

Figure 1 — Typical degassing apparatus

7 Procedure

7.1 General

Carry out all weighings to an accuracy of 0,1 mg. When the pyknometer contains a liquid, stabilize its temperature in the thermostatically controlled bath (5.4) at 25 °C ± 0,05 °C before weighing.

7.2 Preparation of the sample

Drill a cylinder or cut a piece out of the carbon block and crush it into small pieces using the jaw-type crusher (5.7).

Grind the sample to a particle size of < 63 μm using the grinder (5.5). Store the ground material in an airtight container until required for the determination. Before the determination, dry the sample in the drying oven (5.3) at 120 °C ± 2 °C for 8 h. Then cool in a desiccator containing silica gel.

NOTE — In order to avoid the need to verify the particle size of each sample, it is advisable to determine the grinding conditions which will enable the required particle size to be obtained with each sample type (carbon blocks, cathode blocks, prebaked anodes), using the grinder available. This can be done with any apparatus capable of determining the size of such particles.

7.3 Calibration of the pyknometer

Commercial pyknometers are usually calibrated at 20 °C, whereas the present determination is carried out at 25 °C. It is therefore necessary to calibrate the pyknometer at this temperature.

7.3.1 Determination of pyknometer mass

Wash the pyknometer (5.1) with warm concentrated sulfuric acid solution (4.3), taking all necessary precautions. Rinse carefully, first with running tap water, then with distilled water, then with ethanol (4.1), and finally with acetone (4.2). Weigh the perfectly dry pyknometer to the nearest 0,1 mg (mass m_0).

7.3.2 Determination of pyknometer volume

Fill the pyknometer with distilled water degassed at 23 °C to 24 °C, with the ground-glass stopper firmly inserted and the pyknometer cleaned of excess water with filter paper (5.6).

Place the filled pyknometer in the thermostatically controlled bath and heat to 25 °C ± 0,05 °C. During heating, remove the liquid which emerges from the capillary carefully with filter paper. When no more water runs out, the pyknometer has reached the temperature of the bath. Remove it from the bath and dry it carefully. To avoid the pyknometer overflowing due to the absorption of heat from the hand, it may be briefly chilled in cooler water or with acetone (4.2) before drying it. Weigh the pyknometer, which shall be completely dry on the outside, to the nearest 0,1 mg (mass m_1).

The volume V, in millilitres, of the pyknometer is given by the equation

$$V = \frac{m_1 - m_0}{0,997\,05}$$

where

m_0 is the mass, in grams, of the clean, dry, empty pyknometer;

m_1 is the mass, in grams, of the pyknometer filled with distilled and degassed water;

0,997 05   is the density of water, in grams per millilitre, at 25 °C.

Determine the pyknometer volume several times on different days to eliminate the effect of external influences, as well as any small differences in the temperature of the bath. The result shall be taken as the mean of 8 to 10 determinations. Individual values shall not deviate from the mean value by more than ± 0,001 5 ml.

Round the result to the nearest 0,001 ml.

7.3.3 Frequency of calibration

Repeat the calibration at least every 3 months. The mass m_0 shall remain constant to ± 1 mg.

NOTE — Since no two pyknometers have exactly the same volume, each pyknometer has to be calibrated individually. It is not necessary to check the volume every time the pyknometer is used for a series of density determinations, for instance, although it is usual to check the mass each time.

7.4 Determination of the density of the xylene (4.4)

Follow the procedure described in 7.3.2. The density of the xylene, ρ_x, expressed in grams per millilitre, is given by the equation

$$\rho_x = \frac{m_2 - m_0}{V}$$

where

m_0 and V are as defined in 7.3.2;

m_2 is the mass, in grams, of the pyknometer filled with xylene (4.4).

The result shall be taken as the mean of 10 determinations made over a number of days to eliminate the effect of external influences.

7.5 Determination of the density of the sample

7.5.1 Test portion

Weigh, to the nearest 0,1 mg, 5 g ± 0,1 g of the sample (see 7.2) (mass m_3) into the clean, dry, empty pyknometer, prepared in accordance with 7.3.1.

7.5.2 Determination

The determination shall not be carried out if the ambient temperature exceeds 30 °C.

Place the pyknometer, containing the test portion but without its stopper, in the container of the degassing apparatus (5.2). Before adding xylene, evacuate for 15 min to a residual pressure of 1,3 kPa ± 0,3 kPa with the xylene feed closed. This pressure is necessary for a repeatability of ± 0,004 g/ml (see 8.2). If a precision of only ± 0,01 g/ml is required, a pressure of 2,6 kPa ± 0,3 kPa is sufficient.

Add xylene drop by drop to the pykometer. When the test portion is covered with 20 mm of xylene at the most, interrupt the addition of xylene. Continue the evacuation of air, occasionally shaking the pyknometer and the support, until the evolution of air bubbles has stopped. In general, this takes up to 60 min.

Slowly allow air to enter the degassing apparatus. Remove the pyknometer and fill with xylene to the lower edge of the ground.

Allow the test portion to settle for at least 30 min, completely fill the pyknometer with xylene and insert the stopper. Remove from the outside of the pyknometer any xylene which overflows.

Repeat the procedure given in the second paragraph of 7.3.2 ("Place the filled pyknometer..."). Weigh the pyknometer containing the test portion and xylene to the nearest 0,1 mg (mass m_4).

7.6 Checking the determination

The apparatus can be checked for systematic errors by carrying out determinations on standard samples from time to time.

8 Expression of results

8.1 Method of calculation

The density ρ of the sample, expressed in grams per millilitre, is given by the equation

$$\rho = \frac{m_3}{V - \left[\dfrac{m_4 - (m_0 + m_3)}{\rho_x}\right]}$$

where

V and m_0 are as defined in 7.3.2;

ρ_x is as defined in 7.4;

m_3 is the mass, in grams, of the test portion (see 7.5.1);

m_4 is the mass, in grams, of the pyknometer containing the test portion and xylene.

Round the result to the third decimal place.

8.2 Precision

8.2.1 General

The precision data for this method were obtained in accordance with ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* (now withdrawn).

8.2.2 Repeatability, *r*

The results of two determinations carried out on the same sample under repeatability conditions (same operator, same apparatus, short time interval between the determinations) may be considered to be acceptable if they do not differ by more than 0,004 g/ml.

8.2.3 Reproducibility, *R*

The results of two determinations carried out on the same sample under reproducibility conditions (different operators, different apparatus, different times) may be considered to be acceptable if they do not differ by more than 0,006 g/ml.

9 Test report

The test report shall include the following particulars:

a)   all details necessary for identification of the sample;

b)   a reference to this International Standard;

c)   the result of the determination, and the units in which it has been expressed;

d)   details of any unusual features noted during the determination;

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