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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*



Reference number
ISO 9088:1997(E)

Foreword

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

1 Scope

This International Standard specifies a pycnometric method for the determination of the true density in xylene of cathode blocks and prebaked 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, *Pycnometers*.

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 pycnometer 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 $1,3 \text{ kPa} \pm 0,3 \text{ kPa}$. 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 $1,3 \text{ kPa} \pm 0,3 \text{ kPa}$ and an ambient temperature of $25 \text{ }^{\circ}\text{C}$, 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 $120 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$.

5.4 Thermostatically controlled bath, capable of being maintained at $25 \text{ }^{\circ}\text{C} \pm 0,05 \text{ }^{\circ}\text{C}$.

5.5 Grinder, capable of grinding the sample to a particle size of less than $63 \text{ }\mu\text{m}$. The parts which come into contact with the sample shall be made of hardmetal to avoid contamination.

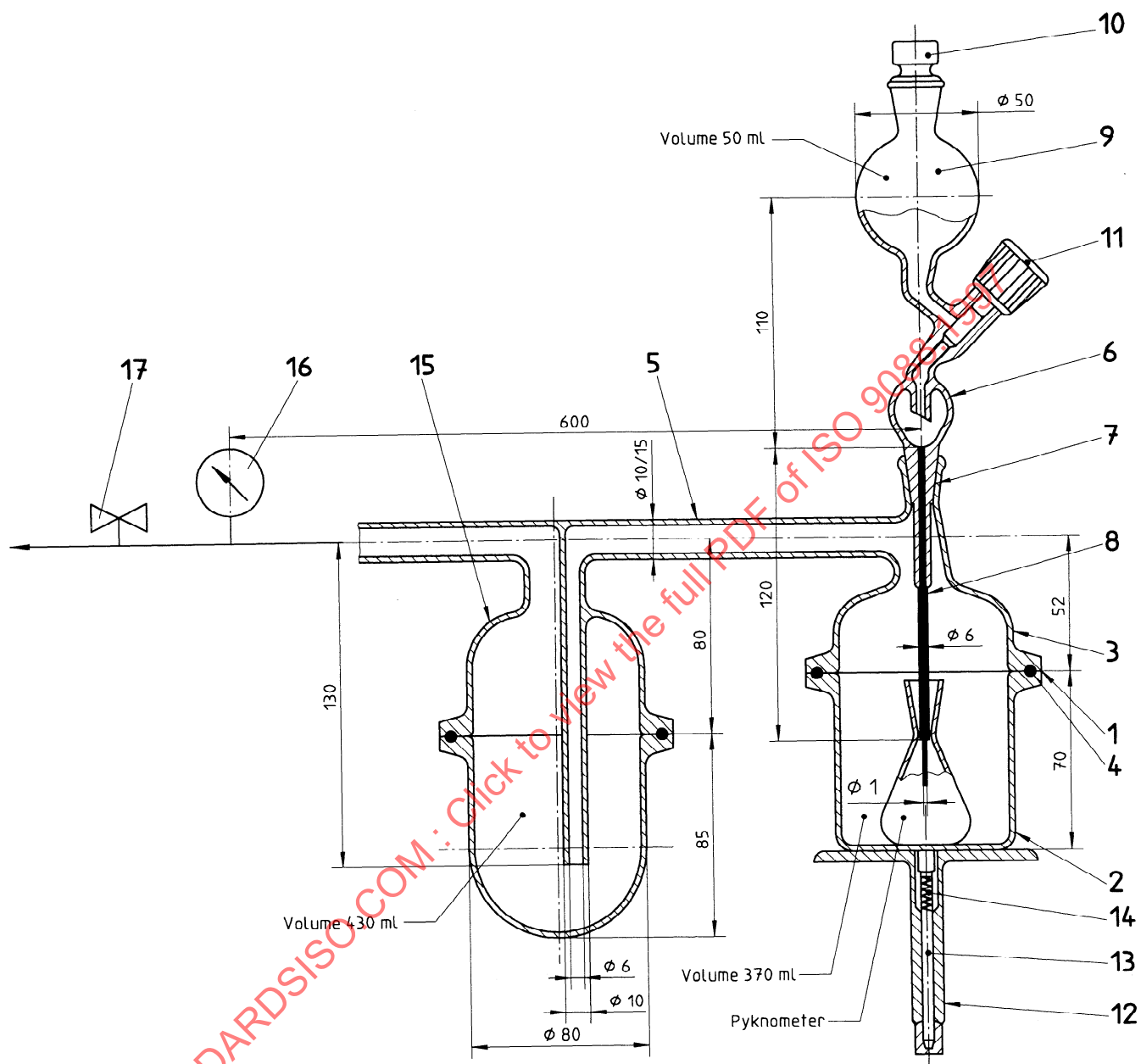
5.6 Filter paper.

5.7 Jaw-type crusher.

6 Sampling

Sample in accordance with ISO 8007.

Dimensions in millimetres

**Key**

- | | |
|-----------------------|--------------------------|
| 1) Container | 10) Ground-glass stopper |
| 2) Glass beaker | 11) Teflon valve |
| 3) Lid | 12) Stand |
| 4) O-ring | 13) Rod |
| 5) Outlet | 14) Spring |
| 6) Filling device | 15) Oil trap |
| 7) Ground-glass joint | 16) Manometer |
| 8) Tube | 17) Valve |
| 9) Reservoir | |

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 $25\text{ }^{\circ}\text{C} \pm 0,05\text{ }^{\circ}\text{C}$ 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 $< 63\text{ }\mu\text{m}$ 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 $120\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ 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 $20\text{ }^{\circ}\text{C}$, whereas the present determination is carried out at $25\text{ }^{\circ}\text{C}$. 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 $23\text{ }^{\circ}\text{C}$ to $24\text{ }^{\circ}\text{C}$, 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 $25\text{ }^{\circ}\text{C} \pm 0,05\text{ }^{\circ}\text{C}$. 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 $25\text{ }^{\circ}\text{C}$.

Determine the pycnometer 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 $\pm 0,0015$ 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 pycnometers have exactly the same volume, each pycnometer has to be calibrated individually. It is not necessary to check the volume every time the pycnometer 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 pycnometer 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, $5 \text{ g} \pm 0,1 \text{ g}$ of the sample (see 7.2) (mass m_3) into the clean, dry, empty pycnometer, 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 pycnometer, 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 $1,3 \text{ kPa} \pm 0,3 \text{ kPa}$ with the xylene feed closed. This pressure is necessary for a repeatability of $\pm 0,004 \text{ g/ml}$ (see 8.2). If a precision of only $\pm 0,01 \text{ g/ml}$ is required, a pressure of $2,6 \text{ kPa} \pm 0,3 \text{ kPa}$ is sufficient.

Add xylene drop by drop to the pycnometer. 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 pycnometer 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 pycnometer 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 pycnometer with xylene and insert the stopper. Remove from the outside of the pycnometer 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

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

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.

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