International Standard 8004

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Carbonaceous materials for the production of aluminium — Calcined coke and calcined carbon products — Determination of the density in xylene — Pyknometric method

Produits carbonés utilisés pour la production de l'aluminium — Coke calciné et produits carbonés calcinés — Détermination de la masse volumique au xylène — Méthode pycnométrique

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Foreword

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Carbonaceous materials for the production of aluminium — Calcined coke and calcined carbon products — Determination of the density in xylene — Pyknometric method

0 Introduction

Calcined coke may be treated with different types of oil in order to avoid the formation of dust during loading and transportation.

The present method does not provide for the elimination of traces of oil which may be present in the sample.

An oil-free sample of coke may be derived from the coke obtained after the determination of oil by the extraction method specified in ISO 8723.

1 Scope and field of application

This International Standard specifies a pyknometric method for the determination of the density in xylene of calcined coke and calcined carbon products used for the production of aluminium.

2 References

ISO 3507, *Pyknometers.*

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

ISO 6375, *Carbonaceous materials for the production of aluminium — Cokes for electrodes — Sampling.*

ISO 8723, *Carbonaceous materials for the production of aluminium-calcined coke — Determination of oil content — Extraction method.*[1]

3 Principle

Measurement of the density at 25 °C of calcined coke and calcined carbon products by a pyknometric method after degassing under 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. Use this water immediately after degassing.

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

4.2 Acetone.

4.3 Xylene, commercial grade, ϱ about 0,860 g/ml.

WARNING — This product burns the skin and can be absorbed into the system through the skin. Inhalation of the vapour from hot material is to be avoided.

4.4 Sulfuric acid, ϱ approximately 1,84 g/ml, about 96 % (*m*/*m*) solution.

5 Apparatus

Ordinary laboratory apparatus, and

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

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

a) **Container** (5) for the pyknometer (5.1), consisting of a glass beaker (6) with removable lid (7) and O-ring (8), capable of containing the pyknometer without stopper. The outlet (10) is connected to the pumping device.

b) **Filling device** (11), fitted to the container by the conical ground joint (12). A tube (13) reaches into the pyknometer bottle. The reservoir (14) with ground stopper (15) contains the pyknometer liquid which is allowed to flow into the pyknometer bottle by the teflon valve (16).

c) **Support** (17), to maintain the beaker in place when no vacuum is applied to the degassing apparatus. The rod (18) with spring (19) allows the beaker (6) to be shaken with the pyknometer to facilitate the evolution of gas bubbles during evacuation.

The apparatus is made of glass. A rotary pump is connected via the outlet and the oil trap (20) to the apparatus. Between the pump and oil trap, a manometer (21) is connected to the vacuum system. The vacuum is adjusted so that the manometer, which is about 600 mm from the joint (12),

1) At present at the stage of draft.

registers 1,3 ± 0,3 kPa[1]. With the valve (22), the apparatus can be filled with air. This must be done slowly and with due care.

NOTE – At a pressure of 1,3 ± 0,3 kPa and an ambient temperature of 25 °C, a light vaporization of xylene may occur, until equilibrium is obtained, but this is of no consequence.

The above-mentioned apparatus is only an example; any other apparatus with these characteristics may be used.

5.3 Electric oven, capable of being controlled at 120 ± 2 °C.

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

5.5 Grinder, capable of grinding the sample to less than 63 µm size. The parts coming in contact with the sample are made of refractory hardmetals, to avoid contamination.

6 Sampling and samples

Sample in accordance with ISO 6375.

7 Procedure

7.1 General instructions

Weighing should be carried out with a precision of ±0,000 1 g.

The buoyancy correction is neglected so that apparent and real masses are assumed to be identical. This approximation is sufficient for the purpose and affects the results by less than 1×10^{-3}.

When the pyknometer contains a liquid, stabilize it in the thermostatically controlled bath (5.4) at 25 ± 0,05 °C.

7.2 Preparation of the sample

Grind the sample to a particular size < 63 µm using the grinder (5.5). Store the ground material in an air-tight container until the measurement is made. Before the measurement, dry the sample in the electric oven (5.3), controlled at 120 ± 2 °C for 8 h. Then cool the sample in a desiccator with silica gel as drying agent.

NOTE – In order to avoid the need for verifying the particle sizes of each sample, it is advisable to determine the most convenient grinding conditions which allow the desired particle size to be reached with each sample type (petroleum coke, anthracite, graphite) and with the available grinding apparatus. In particular, this control can be done with an apparatus of the "elutriateur" type.

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 the mass of the pyknometer

Wash the pyknometer (5.1) with the warm sulfuric acid solution (4.4), taking all necessary precautions. Wash carefully first with running water then with distilled water, then with the ethanol (4.1), and finally with the acetone (4.2). Eliminate the electrostatic charges by rubbing the pyknometer with a lint-free cloth moistened with acetone immediately before weighing. Weigh the dry pyknometer as specified in 7.1 (mass m_0).

7.3.2 Determination of the volume of the pyknometer

Fill the pyknometer with distilled water degassed at a temperature of 23 to 24 °C, with the ground stopper firmly inserted and the pyknometer cleaned from excess water with filter paper.

Place the filled pyknometer into the thermostatically controlled bath (5.4) and heat to a temperature of 25 ± 0,05 °C. During heating, remove the liquid which leaves the capillary bore carefully with filter paper. When no more water runs out, the pyknometer has reached the test temperature. Remove it from the thermostatically controlled bath and dry it carefully. To avoid running over due to the warmth of the hand or when the ambient temperature is greater than 25 °C, brief chilling in cooler water or with the acetone (4.2) can take place beforehand. Weigh the pyknometer, which is completely dry on the outside as specified in 7.1 (mass m_1).

The volume V, in millilitres, of the pycnometer is given by the formula

$$\frac{m_1 - m_0}{0{,}995\,87}$$

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;

0,995 87 is the apparent density of water, in grams per millilitre at 25 °C.

The volume V of the pyknometer is rounded off to 0,001 ml.

The calibration of the pyknometer should be repeated every 3 months and the mass m_0 should remain constant to ±0,001 g. The volume of the pyknometer, when calibration is repeated, shall be carried out several times and on different days to eliminate the effects of outside influences as well as the small differences in regulations of the thermostatically controlled

1) 1 kPa = 10 mbar

bath. Finally, it shall represent the mean of 8 to 10 determinations. The maximum permissible difference between two determinations is 0,0015 ml.

NOTE — During the period of validity of the calibration of the pyknometer (3 months), the pyknometer may be used for numerous determinations. In this case, it should be verified that the mass m_0 remains constant to within 0,001 g.

7.4 Determination of the density of commercial grade xylene

The procedure is the same as described in 7.3.2. The density of xylene ϱ_x, expressed in grams per millilitre, is given by the formula

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

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 (4.3).

The value of ϱ_x shall be the mean of 8 to 10 determinations.

The determinations shall be carried out several times and on different days to eliminate the effect of outside influences and also each time the thermostatically controlled bath has been stopped or changed.

7.5 Determination of the density of calcined coke and calcined carbon products

7.5.1 Test portion

Weigh, as specified in 7.1, 5 ± 0,1 g of the sample (see 7.2) (mass m_3) into the clean, dry, empty pyknometer, prepared according to 7.3.1.

7.5.2 Determination

Place the pyknometer without stopper, containing the test portion (7.5.1) in the container (5 of the figure) of the degassing apparatus (5.2). Before adding xylene, evacuate for 15 min at a residual pressure of 1,3 ± 0,3 kPa with the xylene feed closed (16 of the figure). This pressure is necessary for a precision of ±0,004 g/ml (see 8.2). If only a precision of ±0,01 g/ml is required, a pressure of 2,6 ± 0,3 kPa is sufficient. Thereafter add xylene drop by drop to the pyknometer. After the substance in the pyknometer is covered with 20 mm xylene at the most, interrupt the addition of xylene.

Continue the evacuation of air, occasionally shaking the pyknometer and suport 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 section.

Allow the solid material to settle for at least 30 min and then add xylene to fill the pyknometer before inserting the capillary ground stopper. Remove any excess xylene, which has been excluded, from the outside of the pyknometer.

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 as specified in 7.1 (mass m_4).

8 Expression of results

8.1 Method of calculation

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

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

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 (7.5.1);

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

Results rounded off to the third decimal place.

8.2 Precision (see ISO 5725, sub-clause 3.1)

Repeatability, r: ±0,004 g/ml

Reproducibility, R: ±0,006 g/ml

8.3 Checking the determination

Systematic errors can be checked by carrying out determinations on standard samples from time to time.

9 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) any unusual features noted during the determination;

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

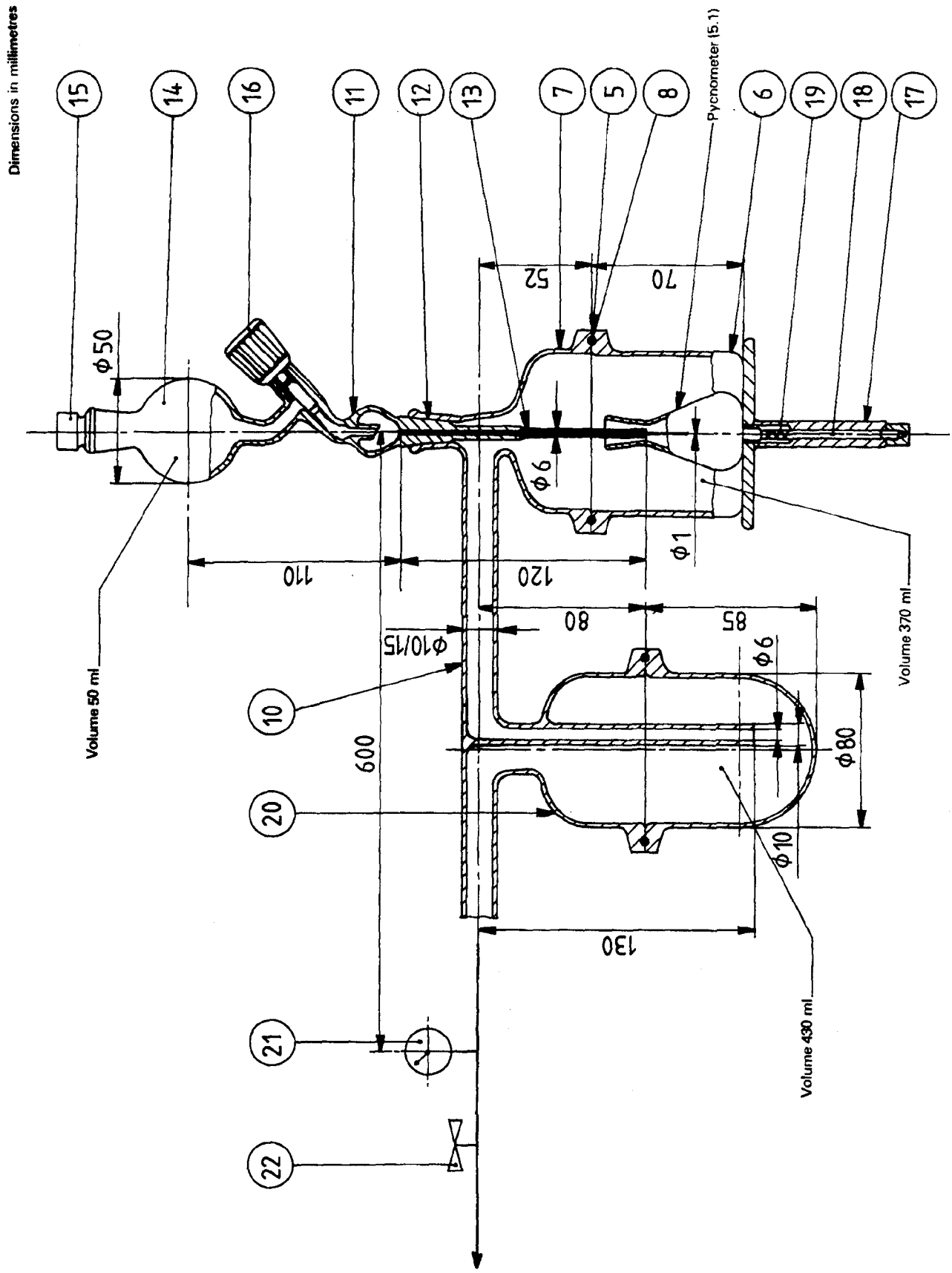

Figure — Typical degassing apparatus