International Standard 6999

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Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of density — Pyknometric method

Produits carbonés utilisés pour la production de l'aluminium — Brai pour électrodes — Détermination de la masse volumique — Méthode pycnométrique

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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.

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It has been approved by the member bodies of the following countries:

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United Kingdom

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Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of density — Pyknometric method

0 Introduction

The density of pitch used for preparation of electrodes for the production of aluminium is a property which gives a guide to its consistency of quality. Knowledge of this parameter is essential for optimum use of pitch.

1 Scope and field of application

This International Standard specifies a pyknometric method for determination of density of pitch used for preparation of electrodes for use in the aluminium industry.

2 References

ISO 3507, *Pyknometers.*

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

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

3 Principle

Measurement, by a pyknometric method, of the density of pitch at 25 °C, after degassing under vacuum.

4 Reagents and materials

During the determination, use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

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

4.2 Acetone.

4.3 Non-ionic wetting agent, 0,1 % (*m*/*m*) aqueous solution.

5 Apparatus

Ordinary laboratory material and

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

5.2 Vacuum pump, fitted with safety valve and filter, capacity about 1 m^3/h, capable of producing a residual pressure of 33 mbar.

5.3 Airtight container, to hold the pyknometer (5.1) capable of being maintained under vacuum during the determination and fitted with a suitable separating funnel.

WARNING — When under vacuum, place behind a suitable safety screen.

A typical assembled apparatus is shown in the figure.

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

5.5 Water bath, capable of being controlled at 25 ± 0,05 °C.

6 Sampling

Sampling shall be carried out according to the method specified in ISO 6257.

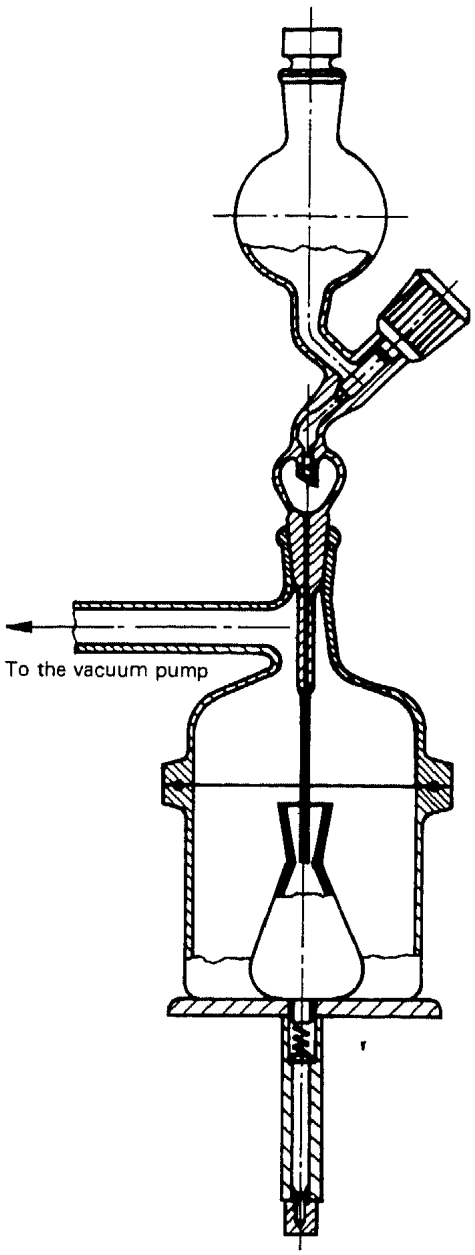

Figure — Typical apparatus for pyknometric determination of density

7 Procedure

7.1 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 latter temperature.

7.1.1 Determination of the mass of the pyknometer

WARNING — Sulfo-chromic mixture causes severe burns, is carcinogenic and in contact with combustible material, may cause fire. Avoid contact with eyes, skin and clothing. Do not breathe vapour. Wear protective clothing and goggles when using this material.

Wash the pyknometer (5.1) with a warm sulpho-chromic mixture, taking all necessary precautions. Wash carefully first with tap water then with distilled water, then with the ethanol (4.1) and finally with the acetone (4.2). Dry the pyknometer in an air stream and weigh to the nearest 0,000 1 g (m_0).

7.1.2 Determination of the volume of the pyknometer

Fill the pyknometer with distilled water at a temperature of 23 to 24 °C, insert its stopper, making sure that water escapes through the orifice in the stopper, and wipe the pyknometer with filter paper.

Place the filled pyknometer in the water bath (5.5), controlled at 25 ± 0,05 °C, and allow it to reach thermal equilibrium at this temperature. Level off the water to the top of the stopper,

Remove the pyknometer from the water bath, cool slightly with the acetone (4.2) and wipe carefully with filter paper.

Weigh the filled pyknometer to the nearest 0,000 1 g (m_1).

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

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

where

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

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

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

NOTES

1 The volume V of the pyknometer is rounded off to 0,000 1 ml. The calibration should be repeated every 3 months; the mass m_0 should remain constant to 0,001 g.

2 It is essential that the volume of the pyknometer be determined several times on different days, to eliminate the effect of outside influences as well as the small differences in regulation of the water bath. The volume V should represent the mean of 8 to 10 determinations.

7.2 Determination of density

7.2.1 Test portion

Weigh to the nearest 0,000 1 g, 5 ± 0,1 g of the laboratory sample (clause 6) with a particle size between 1 and 2 mm (m_2) into the dry, empty pyknometer, prepared according to 7.1.1.

7.2.2 Determination

Fill the separating funnel fitted to the airtight container (5.3), with the wetting solution (4.3). Place the unstoppered pyknometer containing the test portion (7.2.1) in the container and, with the stopcock of the separating funnel closed, run the vacuum pump (5.2) for about 5 min until a residual pressure of 33 to 35 mbar is obtained.

Slowly open the stopcock of the separating funnel and allow the wetting agent solution to fall, drop by drop, until about 20 ml have been added to the pyknometer. Close the stopcock. Maintain the vacuum until any air bubbles in the pyknometer have disappeared. Restore the pressure within the container to ambient level, open the container, remove the pyknometer and fill it completely with the wetting agent solution. Close the pyknometer and continue the determination according to the conditions specified in 7.1.2, starting from the paragraph: "Place the filled pyknometer in the water bath..."

Weigh the filled pyknometer to the nearest 0,000 1 g (m_3).

8 Expression of results

8.1 Calculations

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

$$\frac{m_2}{V - \dfrac{m_3 - (m_2 + m_0)}{0{,}997\ 05}}$$

where

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

m_2 is the mass, in grams, of the test portion (7.2.1);

m_3 is the mass, in grams, of the pyknometer containing the test portion and water;

0,997 05 is the density of the wetting agent solution (4.3), pratically equal to that of the water.

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

Standard deviation of repeatability: 0,004 g/ml.

Standard deviation of reproducibility: 0,006 g/ml.

Results rounded off to the third place of decimals.

8.3 Checking the determination

Systematic errors can be checked by carrying out determinations on reference materials 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 operations not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.