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Carbonaceous materials used in the production of aluminium — Determination of density by gas pyknometry (volumetric) using helium as the analysis gas — Solid materials

Produits carbonés utilisés pour la production de l'aluminium — Détermination de la masse volumique vraie par la méthode pycnométrique gazeuse (volumétrique) utilisant de l'hélium comme gaz analytique — Matériaux solides

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 21687 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

Introduction

This International Standard is based on a DIN method: DIN 51913:2001, *Testing of carbon materials — Determination of density by gas pycnometer (volumetric) using helium as the measuring gas — Solid materials* prepared by Arbeitsausschuß NMP 281, *Test Methods for Carbon and Graphite*.

The density of calcined petrol coke permits an estimate of the degree of calcination of the analysed material.

Carbonaceous materials used in the production of aluminium — Determination of density by gas pyknometry (volumetric) using helium as the analysis gas — Solid materials

1 Scope

This International Standard specifies a method for the determination of the density of green and calcined petroleum coke and similar solid materials (e.g. electrodes). This standard is also suitable for hydrocarbons with a high-temperature boiling range and for other solid materials.

This method is not suitable for graphitized material.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-1:2000, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 6257:2002, *Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling*

ISO 6375:1980, *Carbonaceous materials for the production of aluminium — Coke for electrodes — Sampling*

ISO 8007-1:1999, *Carbonaceous materials used in the production of aluminium — Sampling plans and sampling from individual units — Part 1: Cathode blocks*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
density of a solid material
ρ
mass m per unit volume V

$$\rho = \frac{m}{V} \tag{1}$$

where

ρ is the density, expressed in grams per cubic centimetre;

m is the mass, in grams;

V is the volume of the solid, in cubic centimetres, excluding any pore volume.

NOTE Accessible and inaccessible pores contribute to the total pore volume of the solid. However, only accessible pores can be excluded from the volume of the solid.

4 Principle

A sample is ground and sieved to a size $\leqslant$ 0,063 mm. The density ρ is determined volumetrically in a gas pyknometer with helium as the analysis gas. The mass of the dry test portion is determined and the test portion is loaded into the analysis chamber. Helium gas is introduced into the analysis chamber and pressurized to a set value. The helium gas is subsequently expanded into an expansion chamber. The instrument records the equilibration pressures for both steps. The density ρ of the sample is calculated from the mass m of the dry sample and its volume V determined by gas pyknometry.

5 Apparatus and materials

5.1 Crusher, e.g. jaw-type**,** faced with a hard material that will not be abraded to minimize sample contamination.

5.2 Grinder, capable of grinding the sample to a particle size of less than 0,063 mm, and with the parts that come into contact with the sample made of a hard material that will not be abraded to minimize sample contamination.

5.3 Porcelain bowl, 50 mm to 60 mm in diameter and 30 mm to 40 mm in depth.

5.4 Copper plate.

5.5 Sieve, with a mesh width of 0,063 mm in accordance with ISO 3310-1.

5.6 Drying oven, (preferably with vacuum option) capable of operating at temperatures $\geqslant$ 110 °C.

NOTE Use of a drying oven without a vacuum option may reduce the precision given in Clause 9.

5.7 Dessicator, containing silica gel.

5.8 Gas pyknometer.

5.9 Calibration balls, with known volume.

5.10 Sample cup, tared.

5.11 Helium, with a minimum purity of 99,996 % (by volume fraction).

5.12 Analytical balance, accurate to 0,1 mg.

6 Sampling, sample preparation and drying

6.1 Sampling and sample preparation

6.1.1 Pitch

Take a sample of the material in accordance with ISO 6257.

Coarsely crush the pitch sample, place about 50 g in the porcelain bowl (5.3), weigh and dry for about 2 h in the oven (5.6) at a temperature of about 50 °C above the softening point and at least 110 °C. During the process, stir the sample to avoid air bubbles. Repeat weighing and drying until the mass remains constant.

Pour the sample onto the copper plate (5.4), allow to cool then grind the sample to a particle size of $\leqslant$ 0,063 mm using the grinder (5.2) and then sieve through the sieve (5.5).

Take a test portion of the sieved sample material $\leqslant$ 0,063 mm by filling the tared sample cup.

6.1.2 Coke

Take a sample of the material in accordance with ISO 6375.

Crush, grind and sieve the sample to a particle size of $\leqslant$ 0,063 mm using the grinder (5.2), and then sieve through the sieve (5.5). Store this sample in an airtight container until required for the determination.

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 using the grinder available. This can be done with any apparatus capable of determining the size of such particles.

6.1.3 Cathode blocks and prebaked anodes

Take a sample of the material in accordance with ISO 8007-1.

Drill a cylinder or cut a piece out of the carbon block.

Crush, grind and sieve the sample to a particle size of $\leqslant$ 0,063 mm using the grinder (5.2), and then sieve through the sieve (5.5). Store this sample in an airtight container until required for the determination.

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 (cathode blocks, prebaked anodes), using the grinder available. This can be done with any apparatus capable of determining the size of such particles.

6.2 Drying of coke, cathode blocks and prebaked anodes

Take a test portion of the sieved sample material $\leqslant$ 0,063 mm by filling the tared sample cup. Place the test portion and sample cup in the drying oven at a temperature of 110 °C and a pressure of 1 kPa (10 mbar) for at least 30 min. Remove from the oven, allow to cool in the desiccator (5.7), weigh, reheat again to 110 °C, and introduce the sample cup immediately into the gas pyknometer.

7 Procedure

Calibrate the apparatus and perform the analysis according to the operator's manual supplied by the equipment manufacturer.

8 Calculation

Calculate the density ρ, in grams per cubic centimetre (g/cm^3), using the equation

$$\rho = \frac{m}{V} \tag{2}$$

where

ρ is the density of the sample, expressed in grams per cubic centimetre (g/cm^3);

m is the mass of the sample, in grams (g);

V is the measured volume of the sample, in cubic centimetres (cm^3).

The volume of the sample is usually calculated by the analysis instrument, but may also be calculated from the analysis data using the equation

$$V = V_{\text{cell}} - \frac{V_{\text{exp}}}{\frac{P_1}{P_2} - 1} \tag{3}$$

where

V_{cell} is the calibrated volume of the empty analysis chamber, in cubic centimetres (cm^3);

V_{exp} is the calibrated volume of the expansion chamber, in cubic centimetres (cm^3);

P_1 is the filling pressure of the analysis chamber before the expansion, in kPa;

P_2 is the pressure after the expansion, in kPa.

9 Precision

9.1 Determination

The precision of the method was determined in three Round Robins:

a) green and calcined coke;

b) pitch;

c) baked anodes, cathode blocks and sidewall blocks;

and calculated by ASTM E 691:1999 under the following analysis conditions:

Gas pressure of helium	approximately 150 kPa
Purges	20
Purge/filling pressure	135 kPa
Equilibration rate	0,034 5 kPa/min

The precision is given for a 95 % confidence level.

9.2 Repeatability

The difference between two results, collected on the same sample by the same person with the same instrument under constant analysis conditions, should only in one out of 20 cases be larger than the mentioned value, if the method described is used under common and correct conditions.

Pitch: in the range 1,28 to 1,32 g/cm^3, the following precision has been obtained:
Repeatability (r): 0,003 g/cm^3

Coke: in the range 2,06 to 2,09 g/cm^3, the following precision has been obtained:
Repeatability (r): 0,004 g/cm^3

Anodes: in the range 2,06 to 2,12 g/cm^3, the following precision has been obtained:
Repeatability (r): 0,002 g/cm^3

9.3 Reproducibility

The difference between two single and independent results, collected on identical samples by different persons in different laboratories, should only in one out of 20 cases be larger than the mentioned value, if the method described is used under common and correct conditions.

Pitch: in the range 1,28 to 1,32 g/cm^3, the following precision has been obtained:
Reproducibility (R): 0,015 g/cm^3

Coke: in the range 2,06 to 2,09 g/cm^3, the following precision has been obtained:
Reproducibility (R): 0,014 g/cm^3

Anodes: in the range 2,06 to 2,12 g/cm^3, the following precision has been obtained:
Reproducibility (R): 0,006 g/cm^3

9.4 Examples of use

9.4.1 Repeatability

Given a pitch with real density in the range 1,28 to 1,32 g/cm^3, if two test portions are measured by the same operator in the same laboratory, the measurements are acceptable if they differ by less than 0,003 g/cm^3.

9.4.2 Reproducibility

Given a coke with real density in the range 2,06 to 2,09 g/cm^3, if two test portions are measured at different laboratories, the measurements are acceptable is they differ by less than 0,014 g/cm^3.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) details necessary for the complete identification of the material tested;

c) the type of pyknometer and its manufacturer;

d) the density of the sample, in grams per cubic centimetre (g/cm^3), rounded to 0,001 g/cm^2;

e) any agreed deviations from this International Standard;

f) the date of the test.

Bibliography

[1] ASTM E 691:1999, *Standard practice for conducting an interlaboratory study to determine the precision of a test method*

ICS 71.100.10

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