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Carbonaceous materials for the production of aluminium — Baked anodes — Determination of the air permeability

Produits carbonés utilisés pour la production de l'aluminium — Anodes cuites — Détermination de la perméabilité à l'air



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Foreword

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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 15906 was prepared by Technical Committee ISO/TC 226, Materials for the production of primary aluminium.

ISO 15906:2007(E)

Introduction

Anode performance in aluminium reduction cells can be partially characterized by the measurement of the gas permeability. Higher permeability allows attack and consumption of the anode by air and ${\rm CO}_2$, thus shortening the life and overall performance of the anode.

Carbonaceous materials for the production of aluminium — Baked anodes — Determination of the air permeability

1 Scope

This International Standard describes a method for the measurement of the air permeability of baked anodes, by determining the resistance to airflow of a specimen of specified volume at room temperature, within the range of air permeability between 0,01 nPm (nanoperms) and 10 nPm.

"Green" anodes are considered to be impermeable to gas. Therefore, this test is most applicable to "baked" carbon material.

NOTE This method can also be used for measuring other carbon materials (e.g. microporous carbon materials), but precision statements are not available for other carbon materials.

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 8007-2:1999, Carbonaceous materials used in the production of aluminium — Sampling plans and sampling from individual units — Part 2: Prebaked anodes

3 Terms and definitions

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

3.1

sample

portion of carbon from a baked anode

3.2

test specimen

article prepared from a sample

4 Principle

The air permeability of a sample is determined by a comparative method. The time necessary to draw a specified volume of air through the test specimen is measured. This time is compared to the time necessary to draw a specified volume of air through a standard with known air permeability. This comparison yields the air permeability of the sample material.

5 Apparatus

5.1 Callipers, with 0,01 mm reading precision.

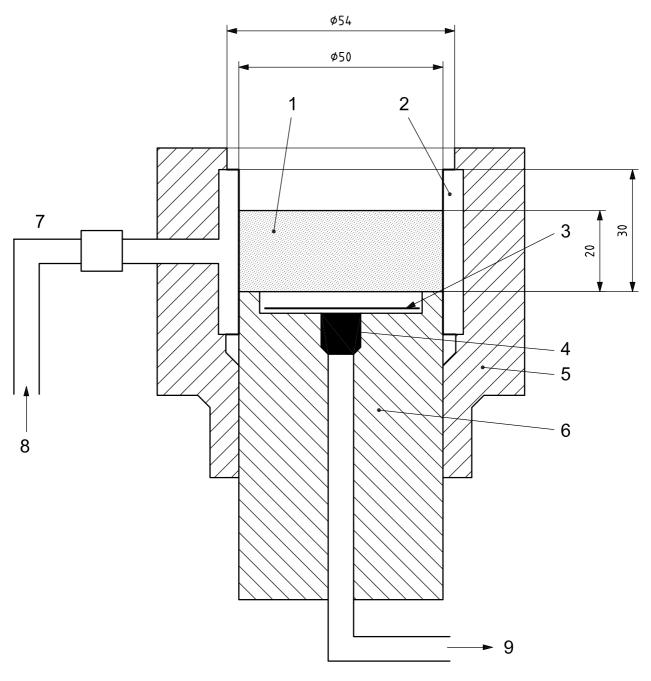
- 5.2 Air permeability unit equipped as in 5.2.1 to 5.2.3.
- Membrane vacuum pump, with an inlet to generate vacuum down to 100 mbar, and an outlet to 5.2.1 generate high pressure up to 4 bar. The maximum flow rate has to reach 25 l/min.
- Permeability cell, as shown in Figure 1. The cell is designed to ensure an airtight sealing around the periphery of the sample. A rubber tube is forced as a seal against the cylindrical test specimen by means of compressed air. The porous filter-paper and the sponge-rubber protect the vacuum pump from particles and/or dust from the sample.
- 5.2.3 **U-tube manometer**, with light fork sensors as shown in Figure 2.

The U-tube is directly linked with the vacuum pump and is placed parallel to the permeability cell. It has a working length of 220 mm, an internal diameter of 16 mm ± 1 mm and an external diameter of 20 mm. It is filled to the equilibrium mark with a non-volatile, non-hygroscopic liquid of low viscosity and density, such as dibutylphtalate or a light-grade mineral oil. Thereby, the vacuum and the air volume flowing through the test specimen can easily be measured by following the oil level inside the U-tube.

There are three light fork sensors. The lowest one (sensor 3) is located at 145 mm below the mark line of equilibrium. The two other sensors are located at 30 mm (sensor 1) and at 80 mm (sensor 2) below the equilibrium mark.

The connection to the vacuum pump can be closed by a valve, which then leads to a decrease of the vacuum by air flowing through the test specimen due to its air permeability.

Dimensions in millimetres

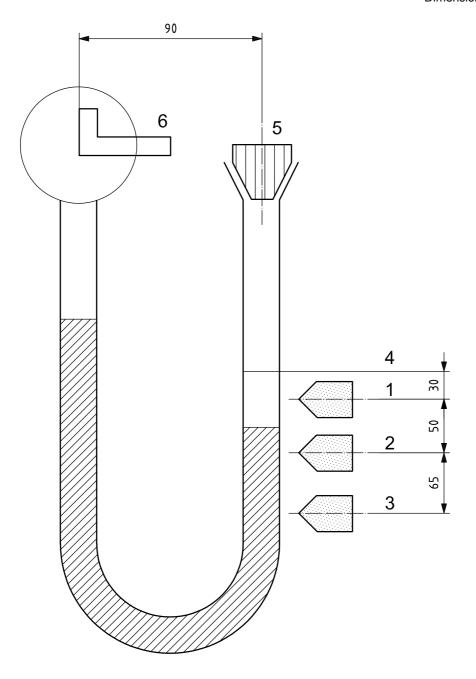


Key

- 1 test specimen
- 2 rubber tube
- 3 porous filter-paper
- 4 rubber sponge
- 5 movable clamping device
- 6 stationary piston
- 7 plastic hose
- 8 compressed air intake
- 9 vacuum system

Figure 1 — Permeability cell

Dimensions in millimetres



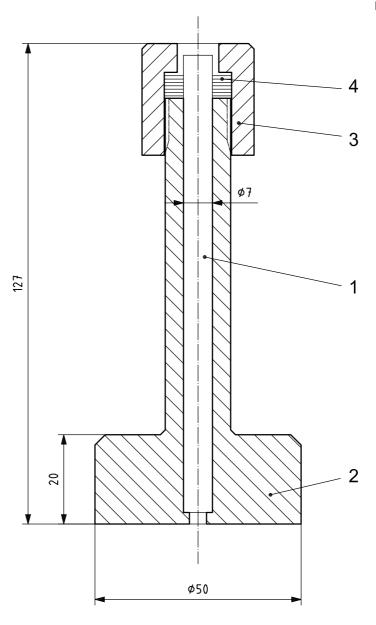
Key

- 1 proximity sensor 1
- 2 proximity sensor 2
- 3 proximity sensor 3
- 4 mark line of equilibrium
- 5 air inlet
- 6 vacuum system

Figure 2 — U-tube manometer

5.3 Standards with known air permeability, high-precision capillaries with known air permeability suitable for calibrating the air permeability unit. (1) A range of permeability values for this standard of between 1 and 2 nPm is appropriate. The capillaries are mounted in a holder which fits onto the cell clamping device (50 mm diameter) of the air permeability unit, see Figure 3. A proper seal guarantees that there is no air leakage between the capillary and the metallic holder.

Dimensions in millimetres



Key

- 1 capillary
- 2 holder
- 3 screw cap
- 4 seal

Figure 3 — Standard with known air permeability

5.4 Impermeable disc, having the same dimensions as the test specimen.

¹⁾ Standards with known air permeability can be certified from the Netherlands Measurements Institute, NMI, in Dordrecht, The Netherlands.

Sampling and preparation of test specimens 6

- Prepare samples taken as defined in ISO 8007-2:1999 in the shape of a cylinder.
- 6.2 Select, at random, one representative core. Cut at least 20 mm from the outermost end of the core. Discard this portion.
- Carefully cut a 20 mm portion from the adjacent area of the core. Blow any dust or particulate off the 20 mm section and dry it in an oven at 110 °C for 4 h.
- Measure the height at four points, 90° apart, on the periphery of the circular end faces, to the nearest 6.4 0,01 mm, using the callipers (5.1). Make two sets of diameter measurements, to the nearest 0,01 mm, using the callipers (5.1). Each set shall consist of four measurements, one at each end and two at intermediate points along an axial line. These sets shall lie at 90° to each other. Determine the mean length and the mean of each of the two sets of diameter measurements.
- The diameter of the test specimen shall be 50 mm \pm 0,4 mm, and the height of the test specimen shall be 20 mm with the two faces parallel within \pm 0,05 mm.

Calibration and standardization 7

Calibrate the apparatus by measuring the air permeability of a suitable standard (5.3) according to the procedure given in Clause 8.

Procedure

- Check all connections, make sure they are airtight and verify that there are no leaks by performing a leak check using the impermeable disc (5.4).
- Place either the calibration standard (5.3) or test specimen (Clause 6), in the test apparatus and expand the rubber tube to ensure an airtight seal.
- 8.3 Evacuate the system until the liquid in the U-tube reaches the sensor 3. Then close the connection to the vacuum tube, using the valve, so causing a decrease of the vacuum by air flowing through the standard or test specimen due to its air permeability. When the liquid level reaches sensor 2, record the time. When the liquid level reaches sensor 1, record the time again and calculate the time difference between sensors 2 and 1.

Calculation of results

Determination of the calibration factor f_c by using the standard of known air permeability (5.3)

Calculate the calibration factor f_c according to Equation (1).

$$f_{\rm c} = t_{\rm c} \cdot D_{\rm c} \cdot \frac{1963}{20} \tag{1}$$

where

is the calibration factor; $f_{\rm c}$

is the time difference, in s; t_{C}

is the air permeability of the standard, in nPm; D_{c}

- 1963 corresponds to the cross-sectional surface area of a 50 mm diameter test specimen, to be tested later;
- corresponds to the nominal height of the test specimen.

9.2 Determination of the air permeability $D_{\rm S}$ of the test specimen

Calculate the air permeability $D_{\rm S}$ of the sample according to Equation (2).

$$D_{S} = \frac{f_{C}}{t_{S}} \cdot \frac{h_{S}}{A_{S}} \tag{2}$$

where

 D_s is the air permeability of the sample, expressed in nPm;

 t_s is the time difference, in s;

 h_s is the height of the test specimen, in mm;

 $A_{\rm s}$ is the cross-sectional surface area of the test specimen, in mm².

10 Precision and bias

10.1 General

Precision statements are valid for anode samples in the range 0,7 nPm to 3,4 nPm.

10.2 Repeatability

According to document ISO/TC 47/SC 7 N 1369:

$$r = 0.06 \text{ (nPm)}$$

10.3 Reproducibility

According to document ISO/TC 47/SC 7 N 1369:

$$R = 0.04X \cdot (nPm) + 0.09 (nPm)$$

where *X* is the material average.

11 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) the names of the producer and customer;
- c) the number and reference marking of the batches;
- d) the date and place of test;
- e) the test results.



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