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Aluminium oxide primarily used for the production of aluminium — Determination of loss of mass at 300 °C and 1 000 °C

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Détermination de la perte de masse à 300 °C et à 1 000 °C

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

This second edition cancels and replaces the first edition (ISO 806:1976) together with ISO 803:1976, which have been technically revised. This International Standard is based on AS 2879.1-2000 prepared by the Standards Australia Committee MN/9, Alumina and Materials used in Aluminium Production, as a revision of AS 2879:1986, *Alumina — Determination of loss of mass at 300 °C and 1 000 °C*.

Introduction

The objective of this revision is to incorporate sample preparation procedures, improve the description of the method and to provide a method for determination of loss of mass by automatic procedures.

Aluminium oxide primarily used for the production of aluminium — Determination of loss of mass at 300 °C and 1 000 °C

1 Scope

This International Standard specifies a method for the determination of loss of mass on heating of aluminium oxide at 300 °C and further loss of mass on ignition at 1 000 °C. By industry convention, these mass losses are often referred to as “moisture (MOI)” and “loss on ignition (LOI)” respectively.

This method is suitable for calcined alumina in the range 0,2 % to 5 % loss of mass at 300 °C and 0,1 % to 2 % loss of mass at 1 000 °C.

This method provides for samples to be treated on an “as-received” basis for determination of actual MOI and LOI in alumina samples. To improve precision of analysis in cases where “as-received” results are not required, samples can be “air-equilibrated” prior to analysis. “Air-equilibration” can greatly affect MOI results and significantly alter LOI results. The “air-equilibration” procedure and its effects are discussed in Annex A.

Instrumental methods are also discussed.

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.

AS 2850, *Chemical analysis — Interlaboratory test programs — For determining precision of analytical method(s) — Guide to the planning and conduct*

3 Principle

The test portion of aluminium oxide is dried at 300 °C for 2 h and the loss of mass is determined by mass difference. The test portion is then ignited at 1 000 °C for 2 h and the further loss of mass is determined.

4 Desiccants

WARNING — Because of the risk of explosion, do not attempt regeneration of magnesium perchlorate by oven drying. Magnesium perchlorate and phosphorus pentoxide are hazardous and reference should be made to appropriate material safety information.

One of the following desiccants shall be used:

a) phosphorus pentoxide;

b) activated alumina;

c) magnesium perchlorate.

If alumina is to be used as a desiccant it shall be freshly activated by heating for 12 h at (300 ± 10) °C and shall then be cooled for at least 4 h in the desiccator before use. The alumina shall be activated daily.

5 Apparatus

5.1 Vacuum desiccator (see Figure 1), containing an aluminium heat sink (5) with positions for four crucibles and tray of desiccant.

Figure 2 shows a suitable design for a heat sink. A metal tray of approximate dimensions 150 mm diameter and 30 mm depth and containing approximately 250 g of desiccant is suitable. The desiccator should be of such dimensions that the free circulation of air is not restricted (see Figure 1 for a suitable configuration). The desiccator lid inlet should also be fitted with a moisture trap containing a granular desiccant.

5.2 Platinum crucibles with lids, of 25 ml capacity and having approximate dimensions of 35 mm diameter and 40 mm depth.

5.3 Electric oven, capable of being controlled at (300 ± 2) °C, and fitted with mechanical air circulation.

NOTE Ovens utilizing natural air convection are not likely to achieve the required temperature control.

5.4 Electric furnace, capable of being controlled at (1 000 ± 10) °C.

5.5 Balance, capable of weighing to the nearest 0,000 1 g.

5.6 Thermogravimetric instrument, if required (see Clause 11).

6 Sample handling and preparation

Aluminium oxide used for aluminium production is a mixture of phases, most of which are active and will rapidly absorb moisture from the atmosphere. Consequently, great care needs to be taken to minimize exposure to atmosphere.

Seal samples in an airtight container immediately after collection. Leave space in the container to allow tumble mixing. Unless samples are prepared promptly and with a minimum of exposure to the laboratory atmosphere, inaccurate values for both moisture content and loss on ignition on an “as-received” basis will result.

Tumble the sample bottle to mix the sample prior to analysis. Remove the test portion and seal immediately after the test portion has been taken from it. Do not use any sub-sampling or mixing technique that involves removing all the bottle contents.

7 Procedure

7.1 Preparation of crucible and lid

Prepare the crucible and lid as follows.

a) Heat the crucible and lid in the furnace (5.4) at (1 000 ±10) °C for 15 min.

b) Remove the crucible and lid from the furnace, place in the desiccator (5.1) and allow to cool for 10 min.

c) Weigh the crucible and lid and record the mass to the nearest 0,000 1 g (m_1).

7.2 Determination of loss of mass at 300 °C (moisture content)

Determine the loss of mass at 300 °C as follows.

a) Transfer a (5 ± 0,5) g test portion from the test sample into the crucible, cover with the lid, weigh and record the total mass to the nearest 0,000 1 g (m_2).

b) Immediately transfer the crucible and test portion into the oven (5.3). Remove the lid from the crucible and place in the desiccator or leave in the oven. Allow the oven to regain an operating temperature of (300 ± 2) °C and maintain this temperature for 120 min.

c) Remove the crucible from the oven, place in the heat sink in the desiccator (5.1) and cover with the lid. Immediately evacuate the desiccator and allow to cool for 10 min.

d) Slowly release the vacuum in the desiccator through a moisture trap without disturbing the test portion. Immediately weigh the crucible and lid and record the mass to the nearest 0,000 1 g (m_3).

Minimize exposure to atmosphere, e.g. while the desiccator lid is off or while weighing, so as to prevent rapid moisture absorption of dried test portions.

7.3 Determination of loss of mass at 1 000 °C (loss on ignition)

Determine the loss of mass at 1 000 °C as follows.

a) Transfer the crucible and lid containing the dried test portion (see 7.2) to the furnace (5.4). Remove the lid from the crucible and place in the desiccator or leave in the furnace. Allow the furnace to regain the operating temperature of (1 000 ± 10) °C. Maintain this temperature for 120 min.

b) Remove the crucible from the furnace, place in the heat sink in the desiccator (5.1) and cover with the lid. Immediately evacuate the desiccator and allow to cool for 30 min.

c) Slowly release the vacuum in the desiccator through a moisture trap without disturbing the test portion. Immediately weigh the crucible and lid and record the mass to the nearest 0,000 1 g (m_4).

Minimize exposure to atmosphere, e.g. while the desiccator lid is off or while weighing, so as to prevent rapid moisture absorption of dried test portions.

8 Calculation

Calculate the loss of mass at 300 °C, w_{300}, expressed as a percentage using Equation (1)

$$w_{300} = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \qquad (1)$$

where

m_1 is the mass, expressed in grams, of the empty crucible plus lid after conditioning (see 7.1), expressed in grams;

m_2 is the mass, expressed in grams, of the crucible, lid and test portion;

m_3 is the mass, expressed in grams, of the crucible, lid and dried test portion.

The loss of mass on ignition, i.e. between 300 °C and 1 000 °C, may be reported either on an undried or on a dried basis (300 °C) and shall be calculated as a percentage using Equations (2) or (3).

Calculate the percent loss of mass on ignition (300 °C to 1 000 °C, denoted by Δ1000) on an undried basis, $w_{\Delta 1000,u}$, using Equation (2):

$$w_{\Delta 1000,u} = \frac{m_3 - m_4}{m_2 - m_1} \times 100 \quad (2)$$

where

m_1, m_2, m_3 are as defined in Equation (1);

m_4 is the mass, expressed in grams, of the crucible, lid and ignited test portion.

Calculate the percent loss of mass on ignition (300 °C to 1 000 °C, denoted by Δ1000) on a dried basis, $w_{\Delta 1000,d}$, using Equation (3):

$$w_{\Delta 1000,d} = \frac{m_3 - m_4}{m_3 - m_1} \times 100 \quad (3)$$

where m_1, m_2, m_3 and m_4 are as defined in Equations (1) and (2).

Results shall be reported to the nearest 0,01 %.

9 Precision

A planned trial of the method was carried out in accordance with AS 2850. Samples of five smelter grade aluminas were analysed on an “as-received” basis ranging from 0,5 % to 3,0 % moisture and 0,7 % to 0,9 % loss of mass on ignition. Results were provided in quadruplicate by six laboratories. Loss of mass on ignition results were calculated on a dried basis. The within laboratory (r) and between laboratory (R) precision data (at 95 % confidence limits) calculated from the results are given in Table 1.

Table 1 — Precision data for loss of mass

Values in percent

Loss of mass	Range of results	Manual method		Instrumental method	
		Repeatability r	Reproducibility R	Repeatability r	Reproducibility R
At 300 °C (Moisture)	< 1	0,07 (5)	0,21	0,04	0,22
	⩾ 1	0,05	0,12	0,04	0,20
At 1 000 °C (Loss on ignition)	All	0,06	0,12	0,03	0,07

10 Test report

The test report shall include the following information:

a) identification of the sample;

b) reference to this International Standard (ISO 806:2004);

c) the loss of mass at 300 °C (moisture) and 1 000 °C (loss on ignition), expressed as a percentage by mass of the test portion, and a statement as to whether the sample was handled on an “as-received” or “air-equilibrated” basis;

d) whether the loss of mass at 1 000 °C (loss on ignition) percentage was calculated on a dried (300 °C) or undried basis;

e) the date on which the test was carried out;

f) any unusual observations made during the course of the test which may have had an effect on the result.

11 Instrumental analysis

The development of modern thermogravimetric instruments has allowed the determination of the moisture content and loss on ignition of smelter-grade alumina to be automated.

To ensure that the use of thermogravimetric instruments does not result in any loss of either analytical accuracy or precision, the following aspects of the determination have to be understood before they can be successfully applied.

Parameters that are critical in the manual determination, such as the time at 300 °C and 1 000 °C, the ability of an oven to be controlled at (300 ± 2) °C, a furnace that can be controlled at (1 000 ± 10) °C and a balance capable of weighing to the nearest 0,000 1 g and capable of weighing the total mass of crucible plus test portion, are just as significant for the determinations to be carried out in a thermogravimetric instrument.

In addition, thermogravimetric instruments take time to reach 300 °C. While reaching this temperature, the atmosphere in the furnace needs to be dry and it is necessary to purge the furnace with dry air. Even under a dry atmosphere, the time to reach 300 °C should not exceed 15 min and the time to increase from 300 °C to 1 000 °C should not exceed 20 min in order not to deviate significantly from the manual procedure.

The use of thermogravimetric instruments is acceptable, provided that it can be demonstrated that the results so generated have an equivalent accuracy to those obtained by the manual method and have, for the particular material under test, a precision as given in Table 1.