INTERNATIONAL STANDARD

ISO 8008

Second edition
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Aluminium oxide primarily used for the production of aluminium — Determination of specific surface area by nitrogen adsorption

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Détermination de la surface spécifique par adsorption d'azote

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Contents

Page

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 8008 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 8008:1986), which has been technically revised.

Introduction

This International Standard is based on Australian Standard AS 2879.4-2003, *Alumina — Determination of specific surface area by nitrogen adsorption*.

Aluminium oxide primarily used for the production of aluminium — Determination of specific surface area by nitrogen adsorption

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies an instrumental method for the determination of specific surface area (SSA) of smelter-grade alumina (SGA) by nitrogen adsorption by a single- or multi-point method. A multi-point method is recommended due to the higher accuracy obtained; if a single-point method is used, a lower result will be obtained.

NOTE Annex A provides an explanation of the difference between single- and multi-point determined BET SSA.

This International Standard is applicable to aluminas having a surface area between 50 m^2/g and 90 m^2/g.

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 2850-1986, *Chemical analysis — Interlaboratory test programs — For determining precision of analytical method(s) — Guide to the planning and conduct*

3 Principle

The method is based on the ability of a material to adsorb nitrogen molecules on its surface at the boiling point of liquid nitrogen. The instrument determines the quantity of nitrogen gas necessary to form a monolayer of gas molecules on the sample surface. The surface area can then be calculated using the basic theory developed by Brunauer-Emmett-Teller[1)]. A test sample is degassed at 150 °C. The degassing process can be carried out using either vacuum or a flowing nitrogen stream. After degassing, the sample is weighed. This mass, and the monolayer volume determined by the instrument, is used to calculate the specific surface area.

4 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

1) S. Brunauer, P.H. Emmett and E. Teller, *J. Am Chem. Soc*. 60, p.309 (1938).

4.1 Liquid nitrogen, with a boiling point of −196 °C at 101.3 kPa[2].

CAUTION — Particular care should be taken when handling cryogenic liquids.

4.2 Nitrogen gas, high purity.

4.3 Other gases, as specified by the instrument manufacturers.

4.4 Reference alumina

Alcan International Limited[3] Alumina Surface Area Standard ALU-11[4]: which is a commercial product sold by Alcan International Limited as part of their in-house commitment to supply reference materials to the alumina/aluminium industry. The certified value at 95 % confidence level of ALU-11 for single-point analysis is 67,8 ± 2,9 m^2/g and for multi-point analysis is 69,1 ± 2,3 m^2/g.

NOTE Annex A provides an explanation of the difference between single- and multi-point determined BET SSA.

5 Apparatus

5.1 Surface-area analyser, employing low temperature (−196 °C) nitrogen adsorption. The instrument should be capable of multi-point or single-point analysis.

5.2 Degassing equipment, suitable for degassing a sample at an elevated temperature (150 °C) using either a flowing nitrogen stream or a vacuum system capable of maintaining a vacuum of $<$ 0,2 mbar.

Sample tubes shall include sealing devices to prevent contact between air and the sample after degassing.

5.3 Analytical balance, capable of weighing to an accuracy of ± 0,000 1 g.

6 Sampling and sample preparation

A 50 g test sample is prepared from the laboratory sample using a riffle or a rotary divider, taking particular care to avoid loss of fine particles through dusting. A representative test portion of optimum mass (depending on the requirements of the instrument and anticipated surface area) shall be taken from the test sample. The mass of sample used should be such that the total surface area is in accordance with the recommendations of the manufacturer of the surface-area analyser (5.1) and with a minimum sample mass of 0,2 g.

Clean all sample tubes prior to each batch of samples analysed. It is recommended that the tubes be cleaned in an ultrasonic bath. To speed up the drying process, it is recommended that the tubes be rinsed with ethanol before being put into the drying oven.

2) 101,3 kPa = 1 atm. The unit atm is deprecated.

3) Alcan International Limited,
Arvida R&D Centre, 1955 Mellon Blvd,
P.O. Box 1250, Jonquière,
Québec, Canada G7S 4 K8,
Tel. 418-699-6585 ext. 2481 or 2828,
FAX: 418-699-2919

4) Alumina ALU-11 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

7 Procedure

7.1 Equipment preparation

The equipment shall be prepared as follows.

a) The surface-area analyser and degassing equipment shall be configured and prepared for degassing and analysis according to the manufacturer's instructions and allowed to warm up. The degassing temperature shall be set to 150 °C.

b) Ensure that the operating parameters of the instrument are entered. For multi-point determinations, relative pressures (P/P_o) of between 0,10 and 0,30 shall be used. For single-point determinations, a relative pressure of 0,30 shall be used.

NOTE 1 For multi-point determinations, these relative pressures ensure that the determination is carried out within the linear portion of the adsorption isotherm for smelter grade alumina. Similarly, a relative pressure of 0,30 is most appropriate for single-point determinations.

NOTE 2 The molecular area of nitrogen is 0,162 nm^2.

c) If required, determine the saturation pressure (P_o) or atmospheric pressure in accordance with the manufacturer's instructions.

The saturation or atmospheric pressure should be determined just prior to analysis and at 6 h intervals during analysis.

7.2 Determination of specific surface area of test sample

The specific surface area of the test sample shall be determined as follows.

a) Weigh a clean, dry sample tube, including its sealing device, on a balance (5.3) and record the mass to the nearest 0,000 1 g (m_1).

b) Add the test sample to the sample tube. The mass of the added test sample shall be in accordance with Clause 6.

c) Insert the test sample into the degassing equipment (5.2) and degas at 150 °C for a minimum of 2 h by using either flowing nitrogen or vacuum degassing methods.

d) Remove the sample tube from the heat source of the degassing unit and cool to room temperature, whilst still purging with nitrogen or maintaining a vacuum. Ensure that air cannot enter the sample tube, both during and at the completion of cooling, until the tube and sample are weighed.

e) Weigh the sample tube, sealing device and sample, and record the mass of the assembly to the nearest 0,000 1 g (m_2). If vacuum degassing is used, purge with nitrogen immediately before weighing.

f) Calculate the degassed test sample mass by:

$$m_3 = m_2 - m_1$$

where

m_1 is the mass of empty sample tube and sealing device, in grams;

m_2 is the mass of sample tube, sealing device and test sample after degassing, in grams;

m_3 is the mass of degassed test sample, in grams.

g) Using sample mass (m_3), and the procedures recommended by the instrument manufacturer, determine the specific surface area of the test sample. Record the specific surface area reported by the instrument, to the nearest 0,01 m^2/g.

8 Calculation and reporting of results

If the instrument reports only the total surface area, calculate the specific surface area of the test sample by:

$$A_{SSA} = \frac{S}{m_3}$$

where

A_{SSA} is the specific surface area of the test sample, in square metres per gram;

S is the total surface area of the test sample, in square metres, reported by the instrument;

m_3 is the mass of degassed test sample, in grams.

Record the calculated specific surface area to the nearest 0,01 m^2/g.

Report the specific surface area thus calculated, or the specific surface area recorded in 7.2 g) to the nearest whole unit.

9 Precision

A test programme of the method in this International Standard was carried out in accordance with AS 2850. From the results of this programme, a within-laboratory repeatability (r) and between-laboratory reproducibility (R), at the 95 % confidence level as given in Table 1, should be achieved.

The results of the test programme are given in Annex B.

Table 1 — Precision data for specific-surface-area determinations

Determination	**Repeatability** (r) m^2/g	**Reproducibility** (R) m^2/g
Single-point	0,8	2,1
Multi-point	1,0	2,5

10 Quality control

The specific surface area of the reference alumina (4.4) shall be determined for every batch of samples, by repeating the procedure in 7.2 and substituting the test sample with the reference material.

If this result does not lie between 67,8 $\pm$ 2,9 m^2/g for single-point determinations, or 69,1 $\pm$ 2,3 m^2/g for multi-point determinations, then it shall be noted in the test report along with the certified value.

11 Test report

The test report shall contain the following information:

a) reference to this International Standard, i.e. ISO 8008;

b) identification of the sample;

c) date on which the sample was taken;

d) date on which the test was carried out;

e) specific surface area of the test sample, expressed in square metres per gram, to the nearest whole unit;

f) whether the analysis was single-point or multi-point;

g) information in accordance with Clause 10, if required;

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

Annex A
(informative)

Difference between single- and multi-point-determined BET SSA

Differences between single- and multi-point SSA for SGA are typically between 1 m^2/g and 3 m^2/g. The single-point solution of the BET equation is a simplification of the theory which results in a lower SSA than the multi-point solution. This difference can be calculated provided the constant C is known for the material being analysed. The constant C in the BET equation is a term that relates to the energy of interaction between the surface and the adsorbate. For the single-point solution, the assumption is made that C is large (greater than 100) resulting in terms in the BET equation simplifying. The BET-plot intercept goes to zero and the slope is inversely proportional to the monolayer volume. This simplification enables the BET equation to be solved by measuring the volume of adsorbate at a single partial pressure.

The difference arising from the assumptions applied in the single-point formulation is:

$$\text{Relative percentage difference} = 100\left\{1 - Cx/\left[Cx + (1 - x)\right]\right\}$$

where

x is the partial pressure used in the single-point measurement;

C is the adsorbate/surface-interaction energy term determined in the multi-point equation.

For smelter grade aluminas, C is typically between 100 and 200.

Hence for $C = 150$ and $x = 0{,}3$, the difference is 1,5 % at the 70 m^2/g level. This equates to the single-point analysis being 1,1 m^2/g lower than the multi-point analysis.

Annex B
(informative)

Results of test programme

A test programme of the method in this International Standard was carried out in accordance with AS 2850. Samples of the Alcan ALU-11 reference material and two other samples from different refineries were analysed. Results were provided in quadruplicate by eleven, nine and six laboratories respectively for samples ALU-11, ALU-10 and S-119. The within-laboratory (r) and between-laboratory (R) precision data (at 95 % confidence limits) and mean-specific-surface-area determinations calculated from the results are given in Table B.1.

Table B.1 — Precision data for specific-surface-area determinations

Test samples	**Specific surface area (mean value)** m^2/g		**Repeatability** (r) m^2/g		**Reproducibility** (R) m^2/g	
	sp	mp	sp	mp	sp	mp
ALU-11	67,8	69,1	0,8	1,2	2,9	2,3
ALU-10	52,9	54,1	0,4	0,6	1,1	3,1
S-119	85,0	88,1	1,1	1,1	2,0	2,0

From these results, an absolute repeatability of 0,8 and an absolute reproducibility of 2,1 should be achieved for single-point determinations, and similarly 1,0 and 2,5 for multi-point determinations.

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