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Aluminium oxide used for the production of aluminium — Determination of particles passing a 20 micrometre aperture sieve

Oxyde d'aluminium utilisé pour la production de l'aluminium — Détermination de la finesse des particules: mesure du passant à 20 micromètres

Reference number
ISO 23202:2006(E)

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Contents

Page

Foreword ... iv
Introduction ... v
1 Scope ... 1
2 Normative references ... 1
3 Principle ... 1
4 Safety ... 1
5 Reagents ... 1
6 Apparatus ... 2
7 Sampling and sample preparation ... 3
8 Procedure ... 3
9 Calculation and expression of results ... 5
10 Precision ... 5
11 Quality control ... 5
12 Test report ... 6
Annex A (informative) **Results of test programme** ... 7
Annex B (normative) **Determination of effective aperture of the test sieve** ... 8
Bibliography ... 10

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.

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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 23202 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

Introduction

This International Standard is based on Australian method, AS 2879.2-2003, *Alumina — Determination of particles passing a 20 micrometre aperture sieve*.

The Minus 20 Micron reference material ASCRM 026 was released in December 2003 by SAI Global, along with a Technical Report on its preparation (TR 2.26-2003, *Certified reference materials — Alumina — Preparation and certification of ASCRM 026*). These are available from SAI through their website:

http://www.standards.com.au/

Aluminium oxide used for the production of aluminium — Determination of particles passing a 20 micrometre aperture sieve

1 Scope

This International Standard sets out a wet-sieving procedure for the determination of the percentage by mass of particles of smelter-grade alumina passing a 20 µm aperture sieve.

This procedure is applicable for aluminas with a −20 µm content up to 4 %.

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 802:1976, *Aluminium oxide primarily used for the production of aluminium — Preparation and storage of test samples*

ISO 806:2004, *Aluminium oxide primarily used for the production of aluminium — Determination of loss of mass at 300 °C and 1 000 °C*

ISO 3310-3, *Test sieves — Technical requirements and testing — Part 3: Test sieves of electroformed sheets*

3 Principle

A test sample of alumina is sieved on a 20 µm electroformed sieve, using acetone, and the retained material is determined gravimetrically after drying at 300 °C.

4 Safety

Chemicals used may be hazardous or toxic and reference should be made to the appropriate Material Safety Data Sheets.

CAUTION — ACETONE PRESENTS A FLAMMABILITY RISK AND SHOULD BE USED IN A SUITABLE EXTRACTION HOOD.

5 Reagents

5.1 Acetone, analytical reagent grade.

5.2 Desiccant.

Phosphorous pentoxide, activated alumina and molecular sieves have been found to be suitable. Silica gel is not a suitable desiccant.

WARNING — PHOSPHOROUS PENTOXIDE IS A HAZARDOUS MATERIAL AND REFERENCE SHOULD BE MADE TO THE APPROPRIATE MATERIAL SAFETY DATA SHEET.

5.3 Ethanol or methanol, technical grade.

6 Apparatus

Ordinary laboratory equipment and the following.

6.1 Test sieve, consisting of a frame, nominally of diameter 75 mm to 150 mm, with an electroformed 20 μm aperture mesh constructed and tested in accordance with ISO 3310-3. The aperture shape shall be round and the sieving medium shall be supported by a suitable grid to provide adequate strength. The construction materials shall be such that the sieve is resistant to chemical corrosion and no physical damage shall occur as a result of heating to 110 °C. The mesh shall be attached to the frame of the sieve such that particles cannot lodge in any part of the joining seam.

NOTE Two suitable mesh sizes are commonly available, 317# and 570#. The open area of the mesh is approximately 17 % for the 570#, and 3,5 % for the 317#. Thus, the 570# sieve is more efficient but more fragile than the 317# sieve.

6.2 Sieve brush, which is acetone-compatible, of high quality with an unpainted handle and soft bristles.

Any paint on the brush shall be removed.

NOTE 1 Westart Akrilik Filbert #6 or #8 brushes have been found suitable. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

NOTE 2 A stiff-bristled brush (e.g. hog's bristle) is not suitable, as it may bias the analysis high and may also damage the sieve mesh.

NOTE 3 A dark-bristled brush is useful, as any alumina particles adhering to the bristles are easily seen.

6.3 Ovens, fitted with mechanical air circulation and capable of being controlled at (300 ± 10) °C.

6.4 Vacuum desiccator, containing an aluminium heat sink, in accordance with ISO 806, and a tray containing desiccant. A tray containing approximately 250 g of desiccant is suitable.

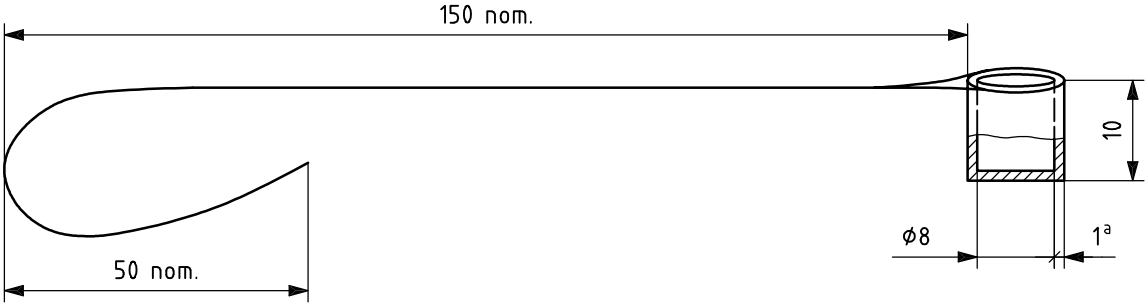
6.5 Platinum crucibles, of 25 ml capacity and approximate dimensions of 35 mm diameter and 40 mm depth and fitted with lids. Two crucible and lid sets are required for each determination. The crucible and lid sets shall be conditioned by placing in an oven maintained at (300 ± 10) °C for 30 min, then cooled and stored in the heat sink in the desiccator.

6.6 Wash bottle, made of polyethylene and filled with reagent-grade acetone.

6.7 Ultrasonic bath.

6.8 Sample scoop, made of stainless steel or brass with a handle. Nominally this scoop shall take up to 0,5 g of alumina when loaded. (See Figure 1.)

Dimensions in millimetres



a Wall thickness (nominal).

Figure 1 — Typical sampling scoop

7 Sampling and sample preparation

A 50 g test sample shall be prepared from the laboratory sample as specified in ISO 802, taking particular care to avoid loss of fine particles through dusting. The test sample shall be mixed well by manual or mechanical tumbling in a sealed container that is not more than 75 % full. After tumbling, let the container rest to allow fines to settle. Each test portion shall be extracted with the sample scoop (6.8), taking a number of grabs to make up the required mass. Manual tumbling shall be repeated between extraction of test portions.

A flat-bladed spatula or vibrating spatula should not be used, as segregation may occur.

8 Procedure

8.1 Check the sieve

The sieve shall be checked to ensure that the mesh is not ruptured and that there is not excessive aperture blinding. When holding the sieve up to a light source, areas of blinding are visible as being darker in appearance. If more than 30 % of the sieve mesh is blinded, the sieve shall be cleaned as specified in 8.2. Larger ruptures in the sieve may be seen by visual inspection. To see smaller ruptures, magnification is required. Inspection using a stereo-microscope is recommended; scan the entire mesh area and the edges to check for ruptures.

NOTE Ruptures can be successfully repaired using silver solder. When a repair is carried out, check the repaired area under magnification to ensure that the rupture is covered and that the solder is bonded to the mesh.

8.2 Cleaning sieve

The procedure shall be as follows.

a) Submerge the sieve in a beaker containing 50 % ethanol or methanol in water. The beaker should be of sufficient diameter to hold the sieve on its side.

b) Place the beaker in an ultrasonic bath and sonicate for 10 min.

c) Remove the sieve and inspect for aperture blinding.

d) Repeat sonication if blinding is still greater than 30 %.

NOTE The sieve is placed on its side during sonication to prevent damage.

8.3 Test procedure

The procedure shall be as follows:

a) Using the sample scoop (6.8), transfer a (2 ± 0,1) g test portion of the test sample into a tared weighing vessel. Record the mass to the nearest 0,000 1 g (m_1) and transfer to a clean 20 μm test sieve (6.1).

b) Weigh two conditioned platinum crucible and lid sets (A and B), and record their masses (m_2 and m_3, respectively). Using the sample scoop, transfer another (2 ± 0,1) g test portion to crucible A. Reweigh crucible set A and record the mass (m_4). Record all masses to the nearest 0,000 1 g.

   Crucible set A holds the test portion used for moisture correction and is retained in a desiccator until Step i). Crucible set B is used to dry the sieved test portion. Both test portions should be weighed out at the same time.

NOTE 1 The crucible and lid set conditioning procedure is given in 6.5.

c) Place the sieve with the test portion in a fume cupboard.

d) Wet the test portion in the sieve with approximately 50 ml of acetone poured from the wash bottle (6.6), and then sieve the material by flooding with a jet of acetone delivered from the wash bottle whilst brushing (see 6.2). Wash any alumina that collects on the inside frame wall of the sieve onto the sieve mesh with acetone. Collect the spent acetone and dispose of or recycle in accordance with environmental good practice.

   Pressure on the brush should be just sufficient to cause slight bending of the bristles. The purpose of brushing is to suspend the fines in the acetone, to enable them to be washed through the sieve.

NOTE 2 Take care not to lose any sample as the jet of acetone impinges on the sieve. Splashing may result in a loss of sample.

e) Continue sieving for 10 min in a manner such that all of the alumina and all parts of the sieve mesh receive equal washing and brushing.

f) Remove any alumina from the brush by pressing the brush firmly against the inside wall of the sieve, simultaneously washing with a jet of acetone and rotating the brush.

   The total volume of acetone passing through the sieve in a single determination should be at least 400 ml. If it is less, not all the fines may be washed through the sieve. A low volume of acetone indicates that the sieve is blinded due to inadequate cleaning and inspection (see 8.2) or the sieve may be overloaded with fines. In the case of the latter, clean the sieve (see 8.2) and repeat the analysis using nominally a 1 g sample.

g) Quantitatively transfer the contents of the sieve to crucible B and wash any adhering alumina from the sieve into the crucible with a jet of acetone.

NOTE 3 This step is conveniently accomplished using a funnel held on a retort stand over the receiving crucible.

h) Carefully evaporate to dryness in a fume hood, avoiding sample loss.

NOTE 4 An infrared lamp is suitable for evaporating acetone.

i) Heat crucible sets A and B in the oven (6.3) maintained at (300 ± 10) °C for 2 h.

WARNING — DO NOT TRANSFER THE CRUCIBLES TO THE OVEN UNTIL THE ACETONE HAS EVAPORATED COMPLETELY.

j) Remove the crucible sets from the oven, place in the heat sink in the desiccator (ensuring the lids cover the crucible) and immediately evacuate the desiccator. Allow to cool.

k) Reweigh crucible sets A and B and record the masses (m_5 and m_6, respectively), to the nearest 0,000 1 g.

9 Calculation and expression of results

The mass fraction, in percent of material passing the 20 μm aperture sieve (w_{20}), shall be calculated from the following equation and expressed to one decimal place on an 'as-received' basis:

$$w_{20} = 100 - \left[\frac{(m_6 - m_3) \times (m_4 - m_2) \times 100}{m_1 \times (m_5 - m_2)} \right]$$

where

w_{20} is the mass fraction, in percent of the material passing the 20 μm aperture sieve;

m_6 is the mass of crucible set B and the +20 μm fraction of the test portion after heating at 300 °C, in grams;

m_3 is the mass of empty crucible set B, in grams;

m_4 is the mass of crucible set A and sample prior to heating at 300 °C, in grams;

m_2 is the mass of empty crucible set A, in grams;

m_1 is the mass of sample taken for the sieve test, in grams;

m_5 is the mass of crucible set A and sample after heating at 300 °C, in grams.

10 Precision

A test programme of the method in this International Standard was carried out according to 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.

NOTE The results of the test programme are given in Annex A.

Table 1 — Precision data for –20 μm analysis

Mass fraction, % (absolute)	
Repeatability (r)	**Reproducibility** (R)
0,24	0,47

11 Quality control

A reference sample, such as ASCRM 026 [1)], should be run with each batch of samples. Particular care should be taken in the tumble mixing and sampling procedures (see Clause 7) to maintain the integrity of successive portions of reference material taken from one bottle.

If the reference sample result is outside the certified range then the sieve should be checked for excessive blinding or rupture (see 8.1). If the sieve appears to be in good condition, then the test should be repeated. If required, repeat a further determination using a fresh bottle of reference material.

1) Available from Standards Australia International.

Consistent out-of-specification reference sample results may be due to the effective aperture of the sieve departing significantly from 20 μm. The effective aperture shall then be checked according to the procedure given in Annex B.

12 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) identification of the test sample;

c) the date on which the sample was taken;

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

e) the −20 μm content of the test sample, expressed as the mass fraction in percent of the test sample, reported to the nearest 0,1 %.

Annex A
(informative)

Results of test programme

A test programme of the method in this International Standard was carried out according to AS 2850 using sieves from three different manufacturers. Samples of four smelter-grade aluminas from different refineries were analyzed. Results were provided in quadruplicate by seven laboratories. The within laboratory (r) and between laboratory (R) precision data (at 95 % confidence limits) and mean values of the particles passing a 20 µm aperture sieve calculated from the results are given in Table A.1, along with the overall precision.

Table A.1 — Precision data obtained using test samples

Mass fraction, % (absolute)			
Sample	**Mean –20 µm content**	**Repeatability** r	**Reproducibility** R
S-112	0,69	0,24	0,30
S-113	2,08	0,21	0,46
S-114	3,74	0,32	0,71
ASCRM 026	1,02	0,20	0,39
Overall		0,24	0,47

Annex B
(normative)

Determination of effective aperture of the test sieve

B.1 General

This Annex sets out a procedure for determining the effective aperture of the 20 μm test sieve.

B.2 Effective aperture

The effective aperture is the size at which the sieve 'cuts' the particle-size distribution of the sample. It is governed by the larger apertures in the sieve. The effective aperture is useful for trouble-shooting purposes. Provided blinding and mesh rupture have been eliminated as a cause of a problem, such as consistently biased certified reference material (CRM) determinations, the effective aperture of the sieve may diagnose a sieve fault. As an example, if a reference material's −20 μm content is high, the effective aperture of the sieve may be greater than 20 μm, or there may be aperture damage.

B.3 Principle

A well-characterized spherical particulate reference material is sieved. The effective aperture is derived from the mass percent passing through the sieve, and relating this to the particle-size distribution of the reference material.

B.4 Reagents and apparatus

B.4.1 Apparatus

The apparatus is in accordance with Clause 6.

B.4.2 Reagent

Certified reference material (CRM). A suitable NIST spherical particulate CRM, SRM 1003b or NIST traceable equivalent.

NOTE Whitehouse Scientific (UK), Endecotts (UK) and Duke Scientific (USA) are suppliers of spherical particulate NIST traceable particle-size CRMs. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

B.5 Procedure

The procedure shall be carried out in duplicate as follows.

a) Thoroughly mix the CRM and extract $1 \pm 0{,}1$ g by taking several increments from various parts of the bottle, using a small scoop similar to that shown in Figure 1 but with dimensions to contain approximately 0,2 g of CRM.

b) Alternatively, micro-rotary riffle split the entire mass into test portions.

c) Dry the reference-material test portion in a tared (m_1), clean, dry platinum crucible for a minimum of 2 h at (110 ± 5) °C. Cool in a desiccator, reweigh, and record the mass (m_2) to the nearest ± 0,000 1 g.

d) Transfer the cooled test portion to the sieve and carry out wet-sieving as described in 8.3, Steps c) to h). Transfer the retained material to the weighed crucible.

e) Dry the retained material for a minimum of 2 h at (110 ± 5) °C. Cool in a desiccator, reweigh, and record the mass (m_3) to the nearest 0,000 1 g.

B.6 Calculation

The mass fraction in percent passing through the 20 μm sieve (w_{20}) shall be calculated from the equation:

$$w_{20} = \frac{(m_2 - m_3 + m_1)}{m_2} \times 100$$

where

w_{20} is the mass fraction in percent of the material passing the 20 μm aperture sieve;

m_1 is the mass of the dried test portion, in grams;

m_3 is the mass of the crucible and the dried portion retained on the sieve, in grams;

m_2 is the mass of the crucible, in grams.

The effective aperture shall be determined as follows:

a) If a cumulative particle-size distribution plot is not supplied with the CRM, plot a graph of the percentage of undersize against particle diameter for the range 16 μm to 24 μm using the CRM's certified particle-size data. Draw a smooth curve through the points.

b) Read off the diameter corresponding to the mass percentage passing the sieve to the nearest 0,1 μm. Duplicate results should agree to within 1,0 μm; if not, then a single effective aperture determination shall be repeated and data within 1 μm used for the average calculation.

c) Calculate the average result, and report to one decimal place. If the average effective aperture falls between 19 μm and 21 μm, the sieve is suitable for use.

Bibliography

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

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