

Aluminium oxide used for the production of primary aluminium — Determination of attrition index —

ICS 71.100.10

National foreword

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**Aluminium oxide used for the production
of primary aluminium — Determination of
attrition index**

*Oxyde d'aluminium utilisé pour la production de l'aluminium primaire —
Détermination de l'index d'attrition*



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Foreword

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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 17500 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 2879.10-2003, *Alumina — Determination of attrition index*.

The Attrition Index reference material ASCRM 025 was released in December 2003 by SAI Global, along with a Technical Report on its preparation (TR 2.25-2003). These are available from SAI through their website:

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

Aluminium oxide used for the production of primary aluminium — Determination of attrition index

1 Scope

This International Standard specifies a procedure for the determination of the attrition index of smelter-grade alumina.

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 2926:2005, *Aluminium oxide used for the production of primary aluminium — Particle size analysis for the range 45 µm to 150 µm — Method using electroformed sieves*

AS 2879.6-1995, *Alumina — Determination of the mass distribution of particle sizes using electroformed sieves*

3 Principle

A test portion is attrited using a high velocity gas jet under controlled conditions using the attrition index apparatus. Flow in the apparatus is calibrated to a reference alumina. The particle size distribution is measured before and after attrition. The attrition index is the relative percentage decrease in the + 45 µm fraction under specific test conditions.

4 Apparatus

4.1 Attrition index apparatus.

A typical assembled apparatus is shown in Figure 1 which consists of:

a) Column

Stainless steel, aluminium and glass columns of length 1 500 mm to 1 600 mm and inside diameter 25 mm have been tested and found to be suitable. To minimize dust carryover, columns with larger-diameter upper sections are also suitable. A column of reduced overall length (700 mm) and upper diameter 64 mm has also been found to be suitable. In any case, modified columns shall maintain the 25 mm ID for a minimum of 200 mm from the orifice plate. Also, they shall be constructed with a smooth tapered transition between sections, to ensure that alumina is not held up in the upper section of the column. Columns may also be provided with a timed automated rapper to facilitate rapping the column [see 6.2 f)].

b) Orifice plate and assembly

The orifice plate assembly holds and seals the orifice plate ¹⁾ centrally to the base of the column. The orifice plate has a circular aperture of (400 ± 15) μm diameter cut through a hard material. A typical orifice plate and its correct orientation is shown in Figure 2.

Flow variations caused by minor dimensional variations between orifice plates are overcome by calibration with a reference alumina.

c) Dust filter and assembly

The dust filter assembly holds and seals the dust filter to the top of the column. Ideally the assembly should enclose the filter to allow subsequent flow measurement to the atmosphere, so that the flow can be monitored to ensure that it is maintained for the test period.

NOTE A cellulose Soxhlet thimble, with an internal diameter of 43 mm and length of 123 mm, has been found to be a suitable filter.

d) Flow measurement and control equipment

The flow measurement and control equipment shall include components to measure and control the airflow, to within 0,1 l/min, at the operating flows (typically 5 l/min to 8 l/min).

4.2 Balance.

Analytical balance capable of weighing 100 g to the nearest 0,01 g.

4.3 Particle sizing apparatus.

Electroformed sieves and apparatus as specified in ISO 2926:2005. For an alternative sizing technique, see Note to 6.3.

4.4 Sample splitter.

To provide representative portions, a rotary splitter is preferred.

4.5 Dried air or nitrogen supply.

A regulated system capable of supplying stable pressures, typically in the range 400 kPa to 800 kPa.

4.6 Sample containers.

Capable of being sealed and with a volume of between 60 ml and 125 ml.

4.7 Reference alumina.

With a certified attrition index value.

NOTE A suitable reference material is available from Standards Australia — (ASCRM 025 series).

1) A suitable orifice plate is obtainable from Societe des Filieres Balloffet, 13 rue de l'Industrie, BP 18, 01151 Lagnieu Cedex, France. 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.

5 Sample handling and preparation

5.1 Reference alumina

The reference alumina shall be split into 50 g to 60 g portions for attrition index and particle size analysis.

5.2 Test sample

The test sample shall be equilibrated to the laboratory atmosphere for a minimum of 2 h in a layer of maximum thickness 5 mm. Then the sample shall be split into 50 g to 60 g portions for attrition index and particle size analysis. Immediately after splitting, place the sample in containers (4.6) and seal.

6 Procedure

6.1 Flow calibration

The calibrated flow rate (F_c) required to produce the reference attrition index value shall be determined using several portions of reference alumina. F_o shall be subsequently used for the analysis of test samples. The procedure to determine F_o shall be as follows:

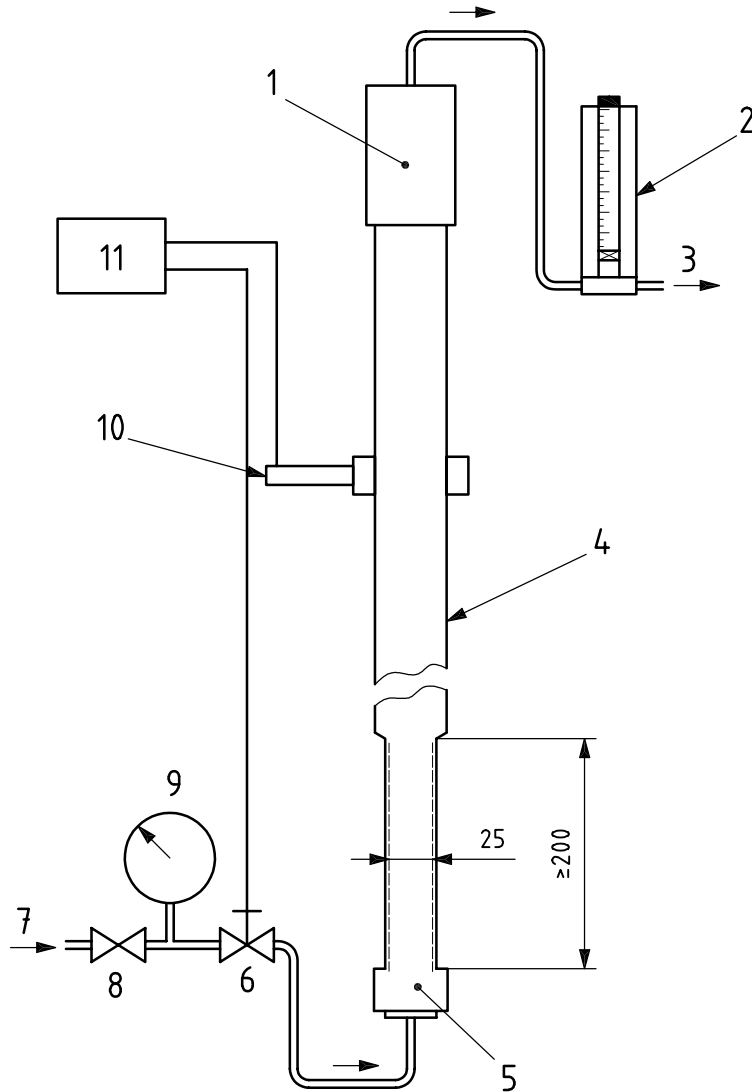
- a) Determine the percent + 45 μm size fraction of the unattrited reference alumina in accordance with 6.3. Perform this determination in triplicate. The mean of the three determinations is used in the calculation of the attrition index for all calibration portions.
- b) Weigh out six 50 g \pm 0,5 g portions of the reference alumina, using the balance (4.2), and record the actual mass to the nearest 0,01 g (m_1).
- c) Attrite the first of the six portions of the reference alumina in accordance with 6.2 c) to i), and determine the percent + 45 μm size fraction of the attrited material. Use a nominal exit flow rate within the range of 5 l/min to 8 l/min.
- d) Calculate the attrition index of this first portion of reference alumina according to Clause 7.
- e) Using this initial result, attrite three further portions of the reference alumina at different flow rates, determine the percent + 45 μm size fraction of each portion and calculate each portion's attrition index. Choose these flow rates such that:
 - 1) the determined attrition index values are greater and less than the recommended reference value; and
 - 2) the difference between the minimum and maximum flow rate is at least 1,0 l/min, but less than 3,0 l/min.
- f) Plot attrition index versus flow rate for these four portions and include a linear trend line of best fit. Add y-axis error bars of 5 % relative to the data points. If an error bar for a data point does not cross the trend line then repeat the analysis for that flow rate. Use the chart to determine the calibrated flow rate (F_c) required to obtain the attrition index of the reference alumina. Figure 3 shows an example of a typical calibration.

NOTE The attrition index has been shown not to be a linear function of flow, however linearity is assumed for the narrow range of flow rates used for calibration.

- g) Using the remaining two portions, repeat the attrition procedure [6.2 c) to i)] in duplicate at the calibrated flow rate. Determine the percent + 45 μm size fraction of each portion and calculate each portion's attrition index. The average of these results should produce the reference attrition index value \pm 0,8. If this value is not obtained, then the apparatus shall be checked and the calibration repeated.

NOTE The $\pm 0,8$ precision above is derived from $(r\sqrt{2})$, i.e. the repeatability of the method (see Table 1) as applied to duplicates.

Dimensions in millimetres

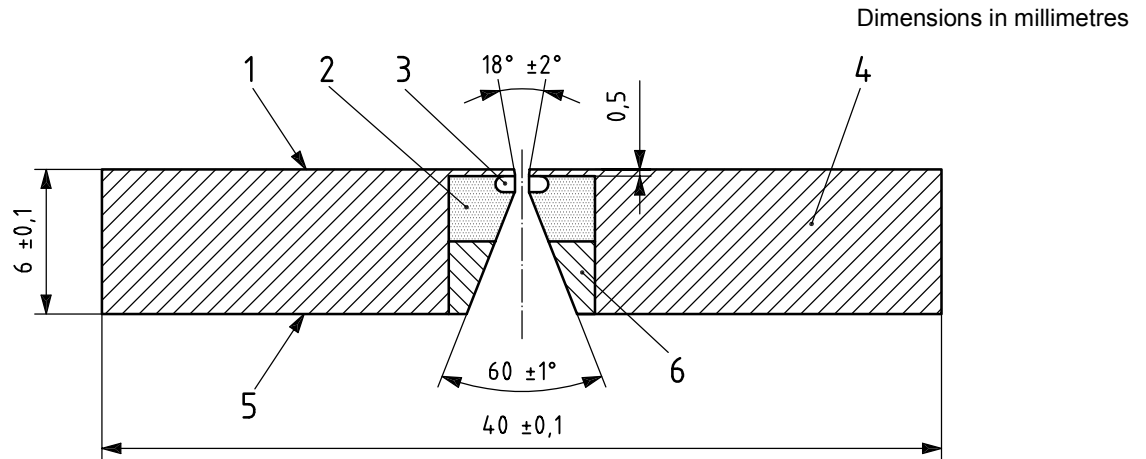


Key

- | | |
|---|----------------------------------|
| 1 dust-filter assembly | 6 on/off valve |
| 2 flow meter | 7 dry compressed air or nitrogen |
| 3 atmosphere | 8 flow-control valve |
| 4 optional expanded diameter to minimise dust carry-over, reduce overall height | 9 inlet air-pressure gauge |
| 5 orifice plate assembly | 10 rapper |
| | 11 timer |

NOTE The automatic rapper is optional.

Figure 1 — Typical attrition apparatus



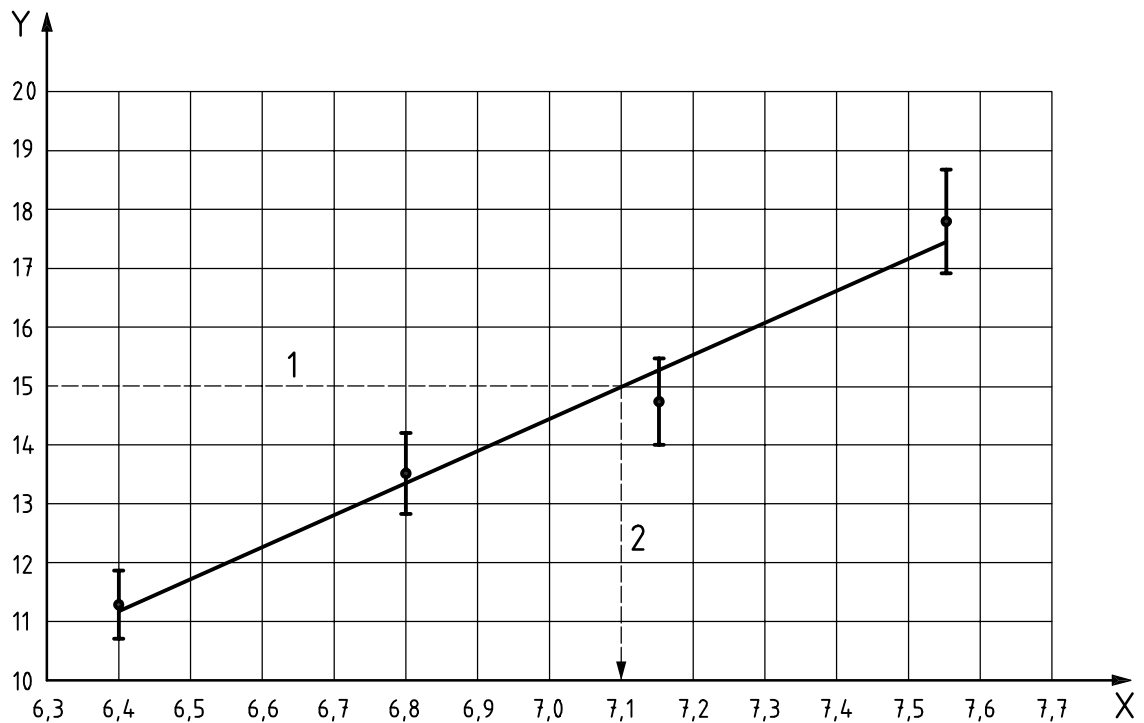
Key

- | | |
|-------------------|----------|
| 1 top | 4 mount |
| 2 epoxy | 5 bottom |
| 3 diamond orifice | 6 plug |

NOTE 1 Mount and plug: stainless steel 40 mm × 6 mm.

NOTE 2 Orifice: 0,400 mm ± 2 μm (as supplied by Balloffet) — laser drilled in natural diamond.

Figure 2 — Typical orifice plate



Key

- | | |
|---------------------|----------------------------------|
| X flow rate (l/min) | 1 reference value |
| Y attrition index | 2 calibrated flow rate (F_c) |

Figure 3 — Attrition index of reference material versus flow rate

6.2 Attrition procedure for test sample and reference alumina

The attrition procedure shall be carried out as follows.

- a) Determine the percent + 45 μm content of an unattrited sample portion using the method described in 6.3.
- b) Weigh out $50 \text{ g} \pm 0,5 \text{ g}$ of the alumina, using the balance (4.2), and record the actual mass to the nearest $0,01 \text{ g}$ (m_1).
- c) Set up the attrition apparatus without the dust-collector assembly.
- d) Using a funnel, slowly transfer this portion into the attrition tube, minimizing dust loss. Re-install the dust-collector assembly.
- e) Activate the air supply, set the flow rate and start the timer. The flow rate, when determining F_0 using the reference alumina, shall be as described in 6.1 c) and e), and for test samples shall be the calibrated flow rate (F_0). The flow rate shall be maintained within $\pm 0,1 \text{ l/min}$ of the set rate for the duration of the test.
- f) Rap the column at least every 3 min to dislodge alumina adhering to the column.

NOTE An optional automatic timed rapping device can be attached to the column.

After exactly 15 min, turn off the air supply, and stop the timer.

- g) Quantitatively transfer the contents of the dust collector and the attrition tube to a tared sample container (4.6). Remove any material from the inside of the apparatus and add to the container.
- h) Determine the total mass of the material recovered to $\pm 0,01 \text{ g}$. See next paragraph if this mass is more than $0,5 \text{ g}$ different from the initial mass (m_1).

The recovered mass will typically be less than the initial mass (m_1) due to loss of moisture. Typically, this loss of mass will be less than $0,5 \text{ g}$, but for samples with a high initial moisture content, this loss may be considerably higher. Care should be taken to ensure that the loss of mass is not due to loss of fines or incomplete recovery of material from the apparatus.

- i) Determine the percent + 45 μm content after attrition, using the same method for the unattrited sample in 6.3.

If the sizing procedure requires sub-sampling, the attrited material shall first be homogenized by passing through a sieve of aperture $200 \mu\text{m}$ to $500 \mu\text{m}$ (to break up agglomerates of fine material) and then tumbling.

6.3 Particle size distribution analysis

Determine the percent + 45 μm content of the alumina in accordance with AS 2879.6.

NOTE Alternative methods of size distribution analysis may be used, provided it can be demonstrated that they lead to attrition index results comparable with those obtained when particle size is determined according to AS 2879.6.

7 Calculation and reporting of results

The attrition index (I_A) is calculated as follows:

$$I_A = \frac{w_b - w_a}{w_b} \times \frac{100}{1}$$

where

I_A is the attrition index;

w_b is the mass fraction percent of + 45 μm before attrition;

w_a is the mass fraction percent of + 45 μm after attrition.

The results shall be reported to one decimal place.

8 Precision

A test programme of the method in this International Standard was carried out in accordance with AS 2850 [2]. From the results of this programme, a within-laboratory repeatability (r) and between-laboratory reproducibility (R) at the 95 % confidence level that should be achieved are given in Table 1.

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

Table 1 — Precision data for attrition index

Repeatability (r)	Reproducibility (R)
1,2	3,3

9 Quality control

If alterations are made to the apparatus and in particular if the orifice plate is changed, then recalibration as per 6.1 shall be performed.

It is recommended that internal standards be run on a frequent basis, and if internal standards exceed control limits then the apparatus shall be checked. If necessary, flow calibrations shall be performed using the reference alumina.

10 Test report

The test report shall contain the following information:

- an identification of the sample;
- a reference to this International Standard;
- the attrition index of the sample (this is reported without units);
- the date on which the test was carried out.

Annex A (informative)

Results of test programme

A test programme of the method in this International Standard was carried out in accordance with AS 2850 [2]. Alcan DWS 92 with a reference attrition index value of 15,0 was used as the flow-calibration alumina to maintain traceability to earlier calibrations. Samples of four smelter-grade aluminas from different refineries were analysed. Results were provided in triplicate by eight laboratories. The within-laboratory (r) and between-laboratory (R) precision data (at 95 % confidence limits) and mean attrition index values calculated from the results are given in Table A.1, as well as the overall precision.

Table A.1 — Precision data obtained using test samples

Sample	Mean attrition index	Repeatability (abs) r	Reproducibility (abs) R
S-120	5,2	1,0	3,3
S-121	26,0	1,5	3,7
ASCRM 025	18,8	1,1	3,8
ALU-11	11,4	1,0	2,1
Overall		1,2	3,3

Bibliography

- [1] ISO 802:1976, *Aluminium oxide primarily used for the production of aluminium — Preparation and storage of test samples*
- [2] AS 2850:1986, *Chemical analysis — Interlaboratory test programs — For determining precision of analytical method(s) — Guide to the planning and conduct*

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