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# Aluminium oxide primarily used for the production of aluminium — Determination of alpha alumina content — Method using X-ray diffraction net peak areas

*Oxyde d'aluminium principalement utilisé pour la production d'aluminium — Dosage de la teneur en alumine alpha — Méthode utilisant la diffraction à rayons X des surfaces de pic net*



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# Foreword

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The committee responsible for this document is ISO/TC 226, *Materials for the production primary aluminium*.



 

# Introduction

This International Standard is based on an Australian Standard AS 2879.3-2010, *Alumina — Determination of alpha alumina content by X-ray diffraction*.

# Aluminium oxide primarily used for the production of aluminium — Determination of alpha alumina content — Method using X-ray diffraction net peak areas

## 1 Scope

This International Standard sets out an X-ray diffraction method for the determination of the alpha alumina content of smelter grade alumina. The method is applicable to smelter grade alumina containing alpha phase at levels up to 50 %. The percentage by mass of alpha alumina is determined on an "as received" basis.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

AS 4538.2, *Guide to the sampling of alumina — Preparation of samples*

## 3 Principle

The integrated peak areas of the (012) and (116) lattice plane reflections (nominal *d* spacings 0,348 nm and 0,160 nm) are measured for the test sample and a 100 % alpha alumina calibration standard. The ratio between the net peak area intensities for the test sample and for the standard is determined and the alpha alumina content calculated from this ratio.

## 4 Reagents

### 4.1 General

During the analysis, only reagents of recognized analytical reagent grade and only distilled water, or water of equivalent purity, shall be used.

**4.2 100 % alpha alumina calibration standard**, prepared in accordance with either 4.2.1 or 4.2.2.

**4.2.1** A commercially available, certified 100 % alpha alumina calibration standard suitable for diffraction analysis.

**4.2.2** An in-house produced 100 % alumina calibration standard prepared in accordance with 6.1 and using the following reagents.

**4.2.2.1 Hydrochloric acid (100 g/l)**.

**4.2.2.2 Smelter grade alumina**.

NOTE The type of alumina used as the base for the 100 % alpha alumina calibration standard can affect the peak areas measured. Grades of the base alumina other than smelter grade can yield different peak area intensities.

# 5 Apparatus

**5.1 X-ray diffractometer**.

**5.2 Hydraulic press**, suitable for preparing mounts.

**5.3 Rotary divider or riffle**.

**5.4 Grinding mill**, capable of grinding alumina to a particle size 0f 90 % less than 45 µm.

**5.5 In-house alpha aluminium apparatus**, prepared as follows (see 5.5.1 to 5.5.5). Necessary only if alpha aluminium standard (4.2.2) is to be prepared in accordance with 6.1.

**5.5.1 Platinum dish and lid**.

**5.5.2 Coarse filter paper**.

**5.5.3 Drying oven**, capable of being controlled at (105 ± 5) °C.

**5.5.4 Magnetic stirrer**.

**5.5.5 Electric furnace**, capable of being controlled at (1 300 ± 50) °C. Necessary only if the 100 % alpha alumina calibration standard (4.2.2) is to be prepared in-house in accordance with 6.1.

# 6 Procedure

## 6.1 Preparation of 100 % alpha alumina calibration standard

The calibration standard shall be prepared as follows.

a) Weigh out approximately 100 g of smelter grade alumina (4.2.2.2).

b) Transfer to a 600 ml beaker and add 250 ml of hydrochloric acid (4.2.2.1). Stir the solution at ambient temperature for 3 h using the magnetic stirrer (5.5.4).

c) Filter the alumina slurry through a coarse filter paper (5.5.2). Wash the paper and the residue thoroughly three or four times with water to remove entrained hydrochloric acid.

NOTE 1 This washing procedure reduces the soda and other impurities in the alumina. Soda will react to form a beta alumina phase on calcining which will reduce the alpha phase content.

d) Dry the alumina residue in the drying oven (5.5.3) at (105 ± 5) °C for 2 h. Transfer the dried alumina residue to a platinum dish (5.5.1). Cover the dish with the platinum lid.

e) Transfer the dish into the electric furnace (5.5.5) at 300 °C. Raise the furnace temperature over a period of at least one hour to (1 300 ± 50) °C. Maintain at (1 300 ± 50) °C for 48 h.

NOTE 2 Raising the temperature gradually will prevent the ejection of material from the dish.

f) Remove the dish from the furnace and allow to cool to room temperature. Transfer the 100 % alpha alumina calibration standard to a sealed container.



 

### 6.2 Preparation of the test portion and calibration standard

The test portion and calibration standard shall be prepared as follows.

a) Prepare a portion of the test sample of a suitable mass for the grinding mill (5.4) by using the riffle or rotary divider (5.3) in accordance with AS 4538.2. Take particular care to avoid loss of fine particles through dusting.

b) Grind the test portion and if necessary the calibration standard, to a particle size of 90 % less than 45 µm in the grinding mill (5.4).

c) Prepare duplicate mounts of the test portion and the calibration standard for XRD analysis (see Note 2). A hydraulic press (5.2) may be used for this.

d) Visually inspect each mount for imperfections such as cracks or areas with an uneven surface finish and discard if any imperfections can be seen. In this case prepare a new mount [(see 6.2 c)].

NOTE 1 Commercially available, certified 100 % alpha alumina calibration standards typically have particle sizes less than 45 µm however in-house produced calibration standards will typically need to be ground.

NOTE 2 Instructive information on sample preparation can be found in the References [1] and [2].

### 6.3 Measurement

The measurement procedure shall be as follows.

a) Set the X-ray diffractometer (5.1) conditions for the determination of alpha alumina phase (corundum) content.

b) Typical diffractometer settings and 2 theta (2θ) angular scan settings are given in Table 1 and Table 2.

c) Ensure that the scanning ranges and background positions are corrected for the actual 2 theta (2θ) diffractometer alignment.

d) Measure the integrated peak intensity and individual 2θ background intensities to allow calculation of net peak area of the (012) and (116) reflections as per [Clause 7 a)], for each mount.

e) Ensure a calibration standard (4.2) mount is measured at least at the beginning and end of each batch.

**Table 1 — Typical diffractometer settings**

| Excitation radiation | Co Kα1 | Cu Kα1 |
|---|---|---|
| **Tube voltage** | 40 kV | 45 kV |
| **Tube amperage** | 40 mA | 40 mA |
| **Measurement** | Fixed area method | Fixed area method |
| **Time per step** | 3 seconds | 3 seconds |
| **Step size** | 0,02° | 0,02° |
| **Background measurement time** | 6 seconds per peak | 6 seconds per peak |

**Table 2 — Typical 2θ scan settings**

| hkl | *d*-spacing (Å) | Theoretical peak position (°2θ)[a] | | Scanning range (°2θ) | | Background (°2θ) | |
|---|---|---|---|---|---|---|---|
| | | Cu Kα1 | Co Kα1 | Cu Kα1 | Co Kα1 | Cu Kα1 | Co Kα1 |
| 012 | 3,48 | 25,58 | 29,79 | 25,2 to 26,0 | 29,3 to 30,2 | 24,9 to 26,4 | 28,9 to 30,7 |
| 116 | 1,60 | 57,50 | 67,91 | 57.0 to 58,1 | 67,3 to 68,6 | 56,7 to 58,4 | 66,9 to 68,9 |

[a] The alpha $Al_2O_3$ (corundum) theoretical peak positions are from the ICDD file no 46–1212, year 2003 (see Reference [3]).

NOTE 1 The background 2θ angle positions are outside the scanning ranges used to determine the integrated peak area. This is to ensure that for the 100 % alpha alumina calibration sample measurement, the background positions are clear of the very large alpha alumina peak.

NOTE 2 Background intensities will be much higher for smelter grade aluminas than for 100 % alpha alumina calibration samples. This is due to the significant contribution to background from intermediate alumina phases in smelter grade aluminas.

## 7 Calculation and expression of results

The alpha alumina content of each test sample shall be calculated separately from each of the two peaks (012) and (116) separately, as below. They are then averaged.

a) Calculate the net integrated peak area, $I_{net}$ for each test portion from Formula (1):

$$I_{net} = I_p - I_b \quad (1)$$

where

$I_p$ is the integrated peak intensity, i.e the sum of the individual step measurements, in cps;

$I_b$ is the integrated background intensity, in cps.

$I_b$ is calculated using:

$$I_b = \left( \frac{2\theta_{diff}}{2\theta_{step}} + 1 \right) \times \left( \frac{I_{b1} + I_{b2}}{2} \right) \quad (2)$$

where

$I_{b1}$ is the intensity at the low-angle background position b1, in cps;

$I_{b2}$ is the intensity at the high-angle background position b2, in cps;

$2\theta_{diff}$ is the scanning range, the difference between the 2θ angle finish and start positions, in °;

$2\theta_{step}$ is the scanning step size, in °.

b) Calculate the average intensity for the 100 % alpha alumina calibration standard, $I_{net(100),av}$, from Formula (3):

$$I_{\text{net(100),av}} = \frac{\left(\sum_{i=1}^{n} I_{\text{net}}\right)}{n} \quad (3)$$

where

$I_{\text{net,100}}$ net peak intensity in cps, for an individual calibration standard measurement, calculated as per Formula (1);

$n$ number of measurements carried out on the calibration standards.

c) Calculate the alpha content for each peak of each test portion, as a percentage by mass, from Formula (4):

$$\%\ \text{alpha alumina} = \frac{I_{\text{net}}}{I_{\text{net(100),av}}} \times 100 \quad (4)$$

d) Average the alpha content for both peaks (012) and (116) of each test portion and express to the nearest integer.

## 8 Precision

A test program to determine precision of the method was carried out in accordance with AS 2850[5] by one analyst from each of eight different laboratories. Results of the interlaboratory test program are shown in Annex A. Four repetitions were carried out by each laboratory on each of four test samples of different refinery aluminas. The means within-laboratory repeatability "*r*" and between-laboratory reproducibility "*R*" at the 95 % confidence level were calculated for the four samples. These are given in Table 3.

**Table 3 — Alpha alumina results from test program**

| Sample ID | Mean $\alpha$ alumina content, % | Repeatability *r* | Reproducibility *R* |
|---|---|---|---|
| S-123 | 2,9 | 0,8 | 1,4 |
| S-124 | 24,5 | 0,8 | 2,9 |
| S-125 | 1,5 | 0,8 | 1,1 |
| S-126 | 9,2 | 1,2 | 1,8 |

From this data, the overall precision expected of this method at the 95 % confidence level is given in Table 4. Note that reproducibility "*R*" was shown to be a function of the alpha content of the sample.

Repeatability "*r*" was typically 0,8, but the "*r*" of 1,2 obtained for sample S-126 indicates a likely sample dependency for "*r*". The S-126 sample was obtained by mixing two process streams (rotary calcined and flash calcined aluminas) from the same refinery. The other samples were from single process streams at other refineries.

**Table 4 — Precision data for alpha alumina determination**

| | |
|---|---|
| Repeatability *r* | Typically 0,8 |
| Reproducibility *R* | $R = 0{,}08\ \alpha + 1{,}0$<br>Where "$\alpha$" is % alpha alumina content of the sample |

## 9 Test report

The test report shall contain the following information:

a) identification of the sample;

b) date on which sample was taken;

c) date on which the test was carried out;

d) alpha alumina content of the test sample, expressed as a percentage by mass of the sample;

e) the number of acceptable results from which the mean value has been calculated;

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

g) a reference to this International Standard, i.e. ISO 19950.



 