

Methods of test for

# Aluminium oxide —

## Part 16: Determination of zinc content by flame atomic absorption

[ISO title: Aluminium oxide primarily used for the production of aluminium — Determination of zinc content — Flame atomic absorption method]

NOTE It is recommended that this Part be read in conjunction with the general information given in BS 4140-0 “*General introduction*” which is issued separately.

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The committees responsible for this British Standard are shown in Part 0.

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# National foreword

This Part of BS 4140 is identical with ISO 2071:1976 “*Aluminium oxide primarily used for the production of aluminium — Determination of zinc content — Flame atomic absorption method*” published by the International Organization for Standardization (ISO).

This method supersedes clause 16 of Addendum No. 2 (1974) to BS 4140:1967. Part 11 to 16<sup>1)</sup> of this standard collectively supersede Addendum No. 2 (1974) to BS 4140:1967, which is withdrawn.

**Terminology and conventions.** The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards, it is current practice to use a full point on the baseline as the decimal marker.

In British Standards it is current practice to use the symbol “L” for litre (and its submultiples) rather than “l”.

Wherever the words “International Standard” appear, referring to this standard, they should be read as “British Standard”.

## Cross-references

International Standard	Corresponding British Standard
	BS 4140 <i>Methods of test for aluminium oxide</i>
ISO 802:1976	Part 1:1986 <i>Preparation and storage of test samples</i> (Identical)
ISO 804:1976	Part 4:1986 <i>Preparation of sample solution by alkaline fusion</i> (Identical)
ISO 2073:1976	Part 13:1986 <i>Preparation of sample solution by treatment with hydrochloric acid under pressure</i> (Identical)
ISO 2927:1973	Part 20:1980 <i>Sampling</i> (Identical)

NOTE The other International Standards listed in the annex are for information only. Their correspondence with British Standards is summarized in BS 4140-0 “*General introduction*”.

**This standard prescribes methods of test only, and should not be used or quoted as a specification defining limits of purity. Reference to this Part should indicate that the method of test used complies with BS 4140-16:1986.**

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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## Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

<sup>1)</sup> Part 14 and 15 are in preparation.

## 1 Scope and field of application

This International Standard specifies a flame atomic absorption method for the determination of the zinc content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products having a zinc content, expressed as ZnO, equal to or greater than 0,002 % (*m/m*).

## 2 References

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

ISO 804, *Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by alkaline fusion*.

ISO 2073, *Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by hydrochloric acid attack under pressure*.

ISO 2927, *Aluminium oxide primarily used for the production of aluminium — Sampling*.

## 3 Principle

Dissolution of a test portion by attack with hydrochloric acid under pressure.

Aspiration of the solution in an acetylene/air flame and determination of the zinc content by spectrophotometric measurement of the absorption of the 213,8 nm line emitted by a zinc hollow-cathode lamp.

The dissolution of the test portion can also be effected by alkaline fusion (see Annex A).

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water doubly distilled from a borosilicate glass apparatus with ground joints, or water of equivalent purity. Avoid the use of leaded glass.

**4.1 Aluminium oxide**, purity greater than 99,95 %, containing less than 0,001 % (*m/m*) of ZnO.

**4.2 Hydrochloric acid**,  $\rho$  approximately 1,19 g/ml, about 38 % (*m/m*) solution.

**4.3 Aluminium**, acid solution (main solution).

Pickle 11 g of extra-pure aluminium (99,999 % purity), in the form of shavings obtained by milling or drilling, in a little nitric acid solution,  $\rho$  approximately 1,40 g/ml, about 68 % (*m/m*) solution. Wash the pickled shavings with water and then dry them by washing with acetone.

Weigh, to the nearest 0,001 g, 10,558 g of these dried shavings, place them in a beaker of suitable capacity (for example 500 ml) and add 144 ml of the hydrochloric acid solution (4.2). Add 1 drop of extra-pure mercury to aid the attack. Wait until the reaction subsides, then place the beaker on a sand bath and maintain a gentle heat until all the aluminium has dissolved. Allow to cool, transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

**4.4 Zinc**, standard solution, corresponding to 0,100 g of ZnO per litre.

Weigh, to the nearest 0,000 1 g, 0,100 g of zinc oxide, previously calcined at 1 000 °C for 1 h and cooled in a desiccator. Place this in a beaker of suitable capacity (for example 100 ml) and dissolve in 5,5 ml of the hydrochloric acid solution (4.2). Dilute the solution and transfer it quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 0,100 mg of ZnO.

**4.5 Zinc**, standard solution, corresponding to 0,020 g of ZnO per litre.

Take 100,0 ml of the standard zinc solution (4.4), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,020 mg of ZnO.

## 5 Apparatus

Ordinary laboratory apparatus and

**5.1 Apparatus** as specified in ISO 2073.

**5.2 Burette**, graduated in 0,05 ml, conforming to ISO/R 385.

**5.3 Spectrophotometer**, atomic absorption type, fitted with a burner fed from cylinders of acetylene and compressed air.

**5.4 Zinc hollow-cathode lamp**

NOTE All glassware, including reagent flasks, shall be of borosilicate glass or glass of another type not releasing zinc, or as an alternative, plastics material. Rubber stoppers shall not be used; use exclusively ground glass or plastics stoppers.

## 6 Procedure

### 6.1 Test portion

Weigh, to the nearest 0,001 g, 2 g of the test sample dried at 300 °C (see 3.3 of ISO 802).

## 6.2 Preparation of the calibration graph

### 6.2.1 Preparation of the standard matching solutions

Into each of a series of six 100 ml one-mark volumetric flasks, place 50 ml of the acid solution of aluminium (4.3) followed by the volumes of standard zinc solution (4.5) shown in the following table, measured with the aid of the burette (5.2):

Standard zinc solution (4.5)	Corresponding mass of ZnO
ml	mg
0 <sup>a</sup>	0
5,0	0,10
10,0	0,20
15,0	0,30
20,0	0,40
25,0	0,50

<sup>a</sup> Blank test on the reagents used for the preparation of the calibration graph.

Dilute to the mark and mix.

### 6.2.2 Photometric measurements

#### 6.2.2.1 ADJUSTMENT OF THE INSTRUMENT, FITTED WITH ZINC HOLLOW CATHODE LAMP (5.4)

Switch on the current to the instrument (5.3) a sufficient time in advance to ensure stabilization. Adjust the wavelength to around 213,8 nm and also the sensitivity and the aperture of the slit according to the characteristics of the instrument. Adjust the pressure of the air and acetylene according to the characteristics of the burner, so as to obtain an oxidizing flame.

#### 6.2.2.2 PHOTOMETRIC MEASUREMENTS

Aspirate the series of standard matching solutions (6.2.1) in the flame, and for each measure the absorbance. Take care to keep the rate of aspiration constant throughout the preparation of the calibration graph.

Spray water through the burner after each measurement.

### 6.2.3 Plotting the calibration graph

Plot a graph having, for example, as abscissae the values, expressed in milligrams, of the quantities of ZnO contained in 100 ml of standard matching solution, and as ordinates the corresponding values of the absorbances, decreased by the measured value for the zero term of the standard matching solutions (blank test on the reagents used for the preparation of the calibration graph).

## 6.3 Determination

### 6.3.1 Preparation of the test solution

Prepare the test solution according to the method specified in ISO 2073, placing the reaction solution in a 100 ml one-mark volumetric flask. Dilute to the mark and mix.

### 6.3.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as for the determination, replacing the test portion with 2 g of the aluminium oxide (4.1) weighed to the nearest 0,001 g.

### 6.3.3 Photometric measurements

Carry out the measurements of the test solution (6.3.1), the blank test solution (6.3.2) and the standard matching solutions (6.2.1), in accordance with the procedure specified in 6.2.2.2. Take care to include with the measurement of the test solution and blank test solution respectively, two standard matching solutions containing quantities of ZnO as close as possible to those to be determined.

## 7 Expression of results

By means of the calibration graph (see 6.2.3), determine the quantities of ZnO corresponding to the values of the photometric measurements of the test solution and of the blank test solution.

The zinc content, expressed as a percentage by mass of zinc oxide (ZnO), is given by the formula

$$\frac{(m_0 - m_1) \times 50}{1\ 000}$$

where

$m_0$  is the mass, in milligrams, of zinc oxide found in the test solution;

$m_1$  is the mass, in milligrams, of zinc oxide found in the blank test solution.

Express the result to three places of decimals.

## 8 Test report

The test report shall include the following particulars:

- the reference of the method used;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

## Annex A Dissolution of the test portion by alkaline fusion

The following particulars and modifications, peculiar to the dissolution of the test portion by alkaline fusion, shall be applied to the method already specified.

### A.1 Reagents

Delete the reagent 4.2 and add the following:

**A.4.6** *Sodium carbonate*, anhydrous.

**A.4.7** *Boric acid* ( $\text{H}_3\text{BO}_3$ ).

**A.4.8** *Hydrochloric acid*, approximately 8 N solution.

Dilute 670 ml of a hydrochloric acid solution,  $\rho$  approximately 1,19 g/ml, about 38 % (*m/m*) solution, with water to 1 000 ml and mix.

### A.2 Apparatus

Replace the apparatus specified in 5.1 by the following:

**A.5.1** *Apparatus* as specified in ISO 804.

### A.3 Procedure

Delete 6.1, 6.3.1 and 6.3.2 and replace them respectively by the following:

#### A.6.1 *Test portion*

Weigh, to the nearest 0,001 g, 5 g of the test sample dried at 300 °C (see 3.3 of ISO 802).

#### A.6.3 *Determination*

##### A.6.3.1 *Preparation of the test solution*

Prepare the test solution in accordance with the method specified in ISO 804, taking care to effect the recovery of the fused mass (see 6.2 and 6.3 of ISO 804) by 90 ml of the hydrochloric acid solution (A.4.8), adjusting the final volume of the principal solution P to 250 ml.

##### A.6.3.2 *Blank test*

Carry out a blank test at the same time as the determination, and following the same procedure, in the presence of extra-pure aluminium oxide, in accordance with the method specified in 6.4.1 of ISO 804.

## Annex B ISO Publications relating to aluminium oxide primarily used for the production of aluminium

ISO 802, *Preparation and storage of test samples*.

ISO 803, *Determination of loss of mass at 300 °C (conventional moisture)*.

ISO 804, *Preparation of solution for analysis — Method by alkaline fusion*.

ISO 805, *Determination of iron content — 1,10-Phenanthroline photometric method*.

ISO 806, *Determination of loss of mass at 1 000 and 1 200 °C*.

ISO 900, *Determination of titanium content — Diantipyrylmethane photometric method*.

ISO 901, *Determination of absolute density — Pycnometer method*.

ISO 902, *Measurement of the angle of repose*.

ISO 903, *Determination of untamped density*.

ISO 1232, *Determination of silica content — Reduced molybdosilicate spectrophotometric method*.

ISO 1617, *Determination of sodium content — Flame emission spectrophotometric method*.

ISO 1618, *Determination of vanadium content — N-Benzoyl-N-phenylhydroxylamine photometric method*.

ISO 2069, *Determination of calcium content — Flame atomic absorption method*.

ISO/R 2070, *Determination of calcium content — Spectrophotometric method using naphthalhydroxamic acid*.

ISO 2071, *Determination of zinc content — Flame atomic absorption method*.

ISO/R 2072, *Determination of zinc content — PAN photometric method*.

ISO 2073, *Preparation of solution for analysis — Method by hydrochloric acid attack under pressure*.



ISO 2828, *Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method.*

ISO 2829, *Determination of phosphorus content — Reduced phosphomolybdate spectrophotometric method.*

ISO 2865, *Determination of boron content — Curcumin spectrophotometric method.*

ISO 2926, *Particle size analysis — Sieving method.*

ISO 2927, *Sampling.*

ISO 2961, *Determination of an adsorption index.*

ISO 3390, *Determination of manganese content — Flame atomic absorption method.*



## Publications referred to

See national foreword.

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