INTERNATIONAL STANDARD 2071

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Aluminium oxide primarily used for the production of aluminium — Determination of zinc content — Flame atomic absorption method

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Dosage du zinc — Méthode par absorption atomique dans la flamme

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47 has reviewed ISO Recommendation R 2071 and found it technically suitable for transformation. International Standard ISO 2071 therefore replaces ISO Recommendation R 2071-1971 to which it is technically identical.

ISO Recommendation R 2071 was approved by the Member Bodies of the following countries :

Austria	Iran	Spain
Belgium	Israel	Sweden
Czechoslovakia	Italy	Switzerland
Egypt, Arab Rep. of	Korea, Rep. of	Thailand
France	Netherlands	United Kingdom
Germany	New Zealand	U.S.A.
Greece	Poland	U.S.S.R.
Hungary	Portugal	
India	South Africa, Rep. of	

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 2071 into an International Standard.

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Determination of zinc content — Flame atomic absorption method

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, ρ 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, ρ 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*	0
5,0	0,10
10,0	0,20
15,0	0,30
20,0	0,40
25,0	0,50

* 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 :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) 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 (H_3BO_3).

A.4.8 Hydrochloric acid, approximately 8 N solution.

Dilute 670 ml of a hydrochloric acid solution, ρ 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 – Pyknometer 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.