INTERNATIONAL STANDARD ISO 2069

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

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Dosage du calcium — 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 2069 and found it technically suitable for transformation. International Standard ISO 2069 therefore replaces ISO Recommendation R 2069-1971 to which it is technically identical.

ISO Recommendation R 2069 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 2069 into an International Standard :

United Kingdom

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Determination of calcium 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 calcium content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products having a calcium content, expressed as CaO, equal to or greater than 0,003 % (*m/m*).

2 REFERENCES

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

ISO 1617, *Aluminium oxide primarily used for the production of aluminium — Determination of sodium content — Flame emission spectrophotometric method.*

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.

Addition of sodium ions to the solution to stabilize the promotion of the emission of calcium, and of lanthanum ions or triethanolamine to increase the sensitivity.

Aspiration of the solution into an acetylene/dinitrogen monoxide flame, and determination of the calcium content by spectrophotometric measurement of the absorption of the 422,7 nm line emitted by a calcium hollow-cathode lamp. For the determination of relatively high CaO contents [greater than approximately 0,03 % (*m/m*)], an acetylene/air flame can be used.

4 REAGENTS

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

4.1 Lanthanum chloride heptahydrate ($LaCl_3.7H_2O$), or

4.1.1 Triethanolamine $[(CH_2OHCH_2)_3N]$, ρ approximately 1,130 g/ml.

4.2 Aluminium oxide, of purity greater than 99,95 %, containing less than 0,000 5 % (*m/m*) of CaO.

4.3 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution.

4.4 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, 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, 10,588 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 (4.3). 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 is dissolved. Allow to cool, transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

4.5 Calcium, standard solution, corresponding to 1,000 g of CaO per litre.

Weigh, to the nearest 0,000 1 g, 1,784 7 g of extra-pure calcium carbonate, previously dried at 250 °C for 2 h and cooled in a desiccator. Place this in a beaker of suitable capacity (for example 600 ml), and dissolve carefully in 10 ml of the hydrochloric acid solution (4.3) and 15 ml of water. 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 1,000 mg of CaO.

Store this solution in a bottle of material free from calcium.

4.6 Calcium, standard solution, corresponding to 0,100 g of CaO per litre.

Take 100,0 ml of the standard calcium solution (4.5), place in 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 CaO.

Store this solution in a bottle of material free from calcium.

4.7 Calcium, standard solution corresponding to 0,020 g of CaO per litre.

Take 100,0 ml of the standard calcium solution (4.6), 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 CaO.

Prepare this solution just before use.

4.8 Sodium, solution corresponding to 8 g of Na_2O per litre.

Weigh, to the nearest 0,000 1 g, 3,770 g of sodium chloride previously dried at 110 °C for 2 h and cooled in a desiccator. Dissolve in about 200 ml of water and transfer the solution quantitatively to a 250 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this solution contains 8 mg of Na_2O.

Store this solution in a bottle of material free from calcium and sodium.

5 APPARATUS

Ordinary laboratory apparatus of material free from calcium, 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 an aspirator burner fed with acetylene and dinitrogen monoxide or with acetylene and air (see clause 3).

5.4 Calcium hollow-cathode lamp.

6 PROCEDURE

6.1 Test portion

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

6.2 Preparation of the calibration graph

Prepare a calibration graph each time a set of samples is analysed.

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 (4.4), 5 ml of the sodium solution (4.8), 45,6 g of the lanthanum chloride (4.1) or 10 ml of triethanolamine (4.1.1) followed respectively by the volumes of the standard calcium solution (4.7) indicated in the following table, measured with the aid of the burette (5.2) :

Standard calcium solution (4.7)	**Corresponding mass of CaO**
ml	mg
0*	0
5,0	0,10
12,5	0,25
20,0	0,40
27,5	0,55
35,0	0,70

* Blank test on reagents for preparation of the calibration graph.

Dilute to the mark and mix.

6.2.2 *Spectrophotometric measurements*

6.2.2.1 ADJUSTMENT OF THE INSTRUMENT, FITTED WITH CALCIUM 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 about 422,7 nm and also the sensitivity and the aperture of the slit according to the characteristics of the instrument. Adjust the pressure of the acetylene and of the dinitrogen monoxide according to the characteristics of the aspirator burner.

6.2.2.2 SPECTROPHOTOMETRIC 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 aspiration rate constant throughout the preparation of the calibration graph.

Aspirate water through the burner after each measurement.

6.2.3 *Plotting the calibration graph*

Plot a graph having, for example, as abscissae the numbers of milligrams of CaO contained in 100 ml of standard matching solution, and as ordinates, the corresponding values of the absorbances, reduced by the value for the zero term of the standard matching solutions (blank test on reagents 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. Add to the solution $(5 - 2{,}5\,x)$ ml of the sodium solution (4.8), where x is the Na_2O content of the test sample, expressed as a percentage by mass, determined according to ISO 1617. Add 45,6 g of the lanthanum chloride (4.1) or 10 ml of the triethanolamine (4.1.1) to the solution, dilute to the mark and mix.

NOTE – The volume of the sodium solution (4.8) added brings the Na_2O content of the aluminium oxide tested to a constant value of 2 % (*m/m*).

6.3.2 *Blank test*

Carry out a blank test following the same procedure and using the same quantities of all the reagents as used for the determination, except that 5 ml of the sodium solution (4.8) should be added and the test portion should be replaced by 2 g of the pure aluminium oxide (4.2) weighed to the nearest 0,001 g.

6.3.3 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements of the test solution (6.3.1), the blank test solution (6.3.2) and the standard matching solutions (6.2.1) following 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 CaO as close as possible to those to be determined.

7 EXPRESSION OF RESULTS

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

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

$$\frac{(m_0 - m_1) \times 50}{1\,000} = \frac{m_0 - m_1}{20}$$

where

m_0 is the mass, in milligrams, of calcium oxide found in the test solution (6.3.1);

m_1 is the mass, in milligrams, of calcium oxide found in the blank test solution (6.3.2).

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

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.