

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 2069

ALUMINIUM OXIDE PRIMARILY USED
FOR THE PRODUCTION OF ALUMINIUM
DETERMINATION OF CALCIUM CONTENT
ATOMIC ABSORPTION METHOD

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 2069, *Aluminium oxide primarily used for the production of aluminium – Determination of calcium content – Atomic absorption method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 2069, which was circulated to all the ISO Member Bodies for enquiry in July 1970. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

One Member Body opposed the approval of the Draft :

New Zealand

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

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**ALUMINIUM OXIDE PRIMARILY USED
FOR THE PRODUCTION OF ALUMINIUM
DETERMINATION OF CALCIUM CONTENT
ATOMIC ABSORPTION METHOD**

1. SCOPE

This ISO Recommendation describes an atomic absorption method for the determination of calcium in aluminium oxide primarily used for the production of aluminium.

2. FIELD OF APPLICATION

The method is applicable to calcium contents, expressed as CaO, greater than 0.003 %.

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.

Nebulization of the solution in the centre of an acetylene/nitrous oxide flame, and determination of the calcium by photometric 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 %), an acetylene/air flame can be used.

4. REAGENTS

For the analysis, use only water doubly distilled from a borosilicate glass apparatus fitted with ground joints, or water of equivalent purity. Avoid the use of lead glass.

4.1 Lanthanum chloride heptahydrate ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$)

or, alternatively,

Triethanolamine (CH_2OHCH_2)₃N, ρ 1.130 g/ml approximately.

4.2 Aluminium oxide, of greater than 99.95 % purity, containing less than 0.0005 % of CaO.**4.3 Hydrochloric acid**, ρ 1.19 g/ml, approximately 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 1.40 g/ml approximately (about 68 % (m/m) solution).

Wash the pickled shavings with water and then dry them by washing with acetone. Weigh, to the nearest 1 mg, 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 one drop of pure mercury to aid the attack. Wait until the reaction quiets down, then place the beaker on a sand bath and maintain at 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 thoroughly.

4.5 Calcium, standard solution, containing 1.000 g/l of CaO.

Weigh, to the nearest 0.1 mg, 1.7847 g of calcium carbonate, extra pure, previously dried at 250 °C 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 1000 ml one-mark volumetric flask. Dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 1.000 mg of CaO.

NOTE. - Store this solution in a plastics flask.

4.6 *Calcium*, standard solution, containing 0.100 g/l of CaO.

Take 100.0 ml of the standard calcium solution (4.5), place in a 1000 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 0.100 mg of CaO.

NOTE. - Store this solution in a plastics flask.

4.7 *Calcium*, standard solution, containing 0.020 g/l of CaO.

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 thoroughly.

1 ml of this standard solution contains 20 µg of CaO.

NOTE. - Prepare this solution just before use.

4.8 *Sodium*, solution containing 8 g/l of Na₂O.

Weigh, to the nearest 0.1 mg, 3.770 g of sodium chloride previously dried at 110 °C 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 thoroughly.

1 ml of this solution contains 8 mg of Na₂O.

NOTE. - Store this solution in a plastics flask.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Apparatus* as specified in ISO Recommendation R 2073, *Aluminium oxide primarily used for the production of aluminium - Preparation of sample solution for analysis by means of attack by hydrochloric acid under pressure*.

5.2 *Burette*, graduated in 0.05 ml conforming to ISO Recommendation R 385, *Burettes*.

5.3 *Spectrophotometer*, atomic absorption type, fitted with a burner fed from cylinders of acetylene and nitrous oxide, or acetylene and air (see section 3).

5.4 *Calcium hollow cathode lamp*.

NOTE. - All glassware, including reagent flasks, should be of borosilicate glass or glass of other types not releasing calcium, or as an alternative, plastics material. Ground glass or plastics stoppers should be used exclusively.

6. PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.001 g, 2 g of the sample dried at 300 °C, prepared according to clause 2.3 of ISO Recommendation R 802, *Aluminium oxide primarily used for the production of aluminium - Preparation and storage of test samples*.

6.2 Preparation of the calibration curve

Prepare a calibration curve 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 lanthanum chloride or 10 ml of triethanolamine (4.1) followed respectively by the volumes of standard solution of calcium (4.7) indicated in the following table, measured with the aid of the burette (5.2).

Standard solution of calcium (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 the reagents used for the preparation of the calibration curve.

Dilute to the mark and mix.

6.2.2 Photometric measurements

6.2.2.1 ADJUSTMENT OF THE APPARATUS, FITTED WITH CALCIUM HOLLOW CATHODE LAMP (5.4). Switch on the current to the apparatus (5.3) a sufficient time in advance to ensure stabilization. Adjust the wavelength to around 422.7 nm and also the sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the pressure of the acetylene and nitrous oxide according to the characteristics of the burner nebulizer, so as to have a clear, non-luminous oxidizing flame.

6.2.2.2 PHOTOMETRIC MEASUREMENTS. Nebulize the series of standard matching solutions (6.2.1) in the centre of the flame and for each measure the absorbance. Take care to keep the rate of nebulization constant throughout the preparation of the calibration curve.

NOTE. - Pass water through after each measurement.

6.2.3 Preparation of calibration chart. Plot a curve showing, for example, on the x-axis the values, expressed in milligrammes, of the quantities of CaO contained in 100 ml standard matching solution, and on the y-axis, the corresponding values of the measured absorbances, reduced 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 curve).

6.3 Determination

6.3.1 Preparation of the sample solution. Prepare the sample solution according to the method described in ISO Recommendation R 2073, placing the reaction solution in a 100 ml one-mark volumetric flask. Add to the solution $(5 - 2.5x)$ ml of the sodium solution (4.8), where x is the Na_2O content of the test sample, expressed as a percentage, determined according to ISO Recommendation R 1617, *Aluminium oxide primarily used for the production of aluminium - Determination of sodium - Flame emission spectrophotometric method*. Add 45.6 g of lanthanum chloride or 10 ml of triethanolamine (4.1) to the solution, make up to volume and mix thoroughly.

NOTE. - The quantity of sodium solution (4.8) added brings the Na_2O content of the aluminium oxide tested to a constant value of 2 %.

6.3.2 Blank test. Carry out a blank test in parallel, following the same procedure and using the same quantities of all the reagents as 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 Photometric measurements. Carry out the measurement of the sample solution (6.3.1), the blank test solution (6.3.2) and the standard matching solutions (6.2.1) following the procedure described in clause 6.2.2.2. Take care to include with the measurement of the sample 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 chart (see clause 6.2.3) determine the quantities of CaO corresponding to the measured absorbances for the sample solution and for the blank test solution.

Calcium oxide (CaO) content is given, as a percentage by mass, by the following formula :

$$(m_1 - m_2) \times 50$$

where

m_1 is the mass, in grammes, of calcium oxide found in the sample solution;

m_2 is the mass, in grammes, of calcium oxide found in the blank test solution.

The result should be expressed to three places of decimals.

8. TEST REPORT

The test report should 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 ISO Recommendation or regarded as optional.

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