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

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Dosage du calcium

Reference number
ISO 2070:1997(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2070 was prepared by the British Standards Institution (BSI) (as BSI 4140: Part 14:1990) and was adopted under a special "fast-track procedure", by Technical Committee ISO/TC 47, *Chemistry*, in parallel with its approval by the ISO member bodies.

It cancels and replaces ISO 2070:1981 of which it constitutes a technical revision.

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

1 Scope

This International Standard describes 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, of not less than 0,002 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 802:1976, *Aluminium oxide primarily used for the production of aluminium — Preparation and storage of test samples.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 2073:1976, *Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by hydrochloric acid attack under pressure.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Principle

A test portion is dissolved in hydrochloric acid under pressure.

The solution is aspirated into a dinitrogen oxide (nitrous oxide) acetylene flame and the calcium content determined by spectrometric measurement of the absorption at a wavelength of 422,7 nm using a calcium hollow-cathode lamp.

4 Reagents

Except where otherwise stated, use only reagents of recognized analytical grade and water complying with grade 2 of ISO 3696. Do not use leaded-glass reagent vessels.

4.1 Aluminium oxide, of purity greater than 99,95 %, containing less than 0,000 1 % (*m/m*) of CaO.

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

4.3 Aluminium, acid solution, having an aluminium concentration equivalent to that of a 20 g/l solution of alumina (Al_2O_3).

Wash approximately 11 g of extra-pure aluminium shavings [99,999 % (*m/m*)], in 2 ml to 3 ml of nitric acid solution [$\rho \approx 1{,}4$ g/ml, about 68 % (*m/m*)]. Rinse the shavings with water, wash them with acetone in a fume cupboard and blow or suck dry.

Weigh, to the nearest 1 mg, 10,585 g of the dried shavings, place them in a beaker of suitable capacity (for example 500 ml and add 144 ml of hydrochloric acid solution (4.2). Add 1 drop of extra-pure mercury to aid the attack. Wait until the reaction subsides 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 (5.2), dilute to the mark and mix.

4.4 Standard calcium solution, $c(CaO) = 0{,}020$ g/l.

Heat about 1 g of calcium carbonate in an oven at 110 °C ± 5 °C for 16 h and cool in a desiccator. Weigh, to the nearest 0,000 1 g, 0,178 5 g of the calcium carbonate and transfer to a beaker of suitable capacity (for example 100 ml) and dissolve in 5,5 ml of hydrochloric acid solution (4.2). Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask (5.2), dilute to the mark and mix.

Transfer, by pipette, 100 ml of this solution to a 500 ml one-mark volumetric flask (5.2), dilute to the mark and mix.

4.5 Strontium chloride solution, $c(SrCl_2) = 100$ g/l.

Weigh, to the nearest 1 mg, 25,000 g of strontium chloride of calcium content less than 0,001 % (*m/m*) and transfer to a beaker of suitable capacity (for example 250 ml) and dissolve in about 100 ml of water. Transfer the solution quantitatively to a 250 ml one-mark volumetric flask (5.2), dilute to the mark and mix.

5 Apparatus

Only borosilicate glassware or plastics materials shall be used. Only ground-glass or plastic stoppers shall be used, and not rubber.

Ordinary laboratory apparatus, the apparatus described in 3.3.2 of ISO 802:1976, in clause 5 of ISO 2073:1976 and the following are required.

5.1 One-mark pipettes, complying with ISO 648, class A.

5.2 One-mark volumetric flasks, complying with ISO 1042, class A.

5.3 Spectrometer, atomic absorption type, fitted with a burner fed from cylinders of acetylene and dinitrogen oxide (nitrous oxide), and with a calcium hollow-cathode lamp.

6 Preparation of test sample

Prepare a test sample in accordance with ISO 802.

7 Procedure

7.1 Test portion

Weigh, to the nearest 1 mg, 2 g of the test sample prepared in clause 6.

7.2 Determination

7.2.1 Preparation of test solution

Take the test portion prepared in 7.1 and follow the procedure described in ISO 2073. Transfer the resulting solution quantitatively to a 100 ml one-mark volumetric flask. Add, using a pipette (5.1), 5 ml of strontium chloride solution (4.5), dilute to the mark and mix.

7.2.2 Preparation of blank test solution

Follow the procedure described in 7.2.1 but replace the test portion with the same mass of aluminium oxide (4.1), weighed to the nearest 1 mg.

7.2.3 Preparation of calibration solutions

Into each of a numbered series of six 100 ml one-mark volumetric flasks (5.2), transfer, using a 50 ml pipette (5.1), 50 ml of the acid solution of aluminium (4.3) followed by the corresponding portions of the calcium solution (4.4) shown in table 1, using pipettes (5.1). Add, using a pipette (5.1), 5 ml of strontium chloride solution (4.5), dilute to the mark and mix.

7.2.4 Adjustment and operation of the spectrometer

Operate the spectrometer (5.3) throughout the determination in accordance with the manufacturer's instructions. Adjust the wavelength to 422,7 nm and alter the sensitivity and the aperture of the slit according to the characteristics of the instrument. Adjust the pressure of the dinitrogen oxide (nitrous oxide) and acetylene according to the characteristics of the burner, to obtain an oxidizing flame.

7.2.5 Spectrometric measurements

Carry out measurements at regular intervals in a random order of the test solution (see 7.2.1), the blank test solution (see 7.2.2) and the calibration solutions (see 7.2.3), taking care to maintain a constant aspiration rate. Take repeat readings of all solutions in the reverse order to compensate for drift, and calculate the mean of the duplicate absorbance values.

Table 1 — Calibration solutions

No.	Volume of calcium solution ml	Corresponding mass of calcium oxide mg
1 [1)]	0	0
2	5	0,10
3	10	0,20
4	15	0,30
5	20	0,40
6	25	0,50

1) Calibration blank solution.

8 Expression of results

8.1 Calibration curve

Correct each measurement obtained in 7.2.5 by subtracting the value given by the blank calibration solution numbered "1". Prepare a calibration curve by plotting corrected absorbance against the corresponding mass, in milligrams, of CaO.

Determine the quantities, in milligrams, of CaO corresponding to the absorbance measurements for the test solution and the blank test solution.

8.2 Calculation

The calcium content w, expressed as a percentage by mass of calcium oxide (CaO), is given by the following equation:

$$w = \frac{(m_0 - m_1)}{1\ 000} \times \frac{100}{2}$$

where

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

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

Express the result to three decimal places.

9 Precision

The precision of this method is not known because interlaboratory data are not available. Interlaboratory data are being obtained, and a precision statement will be added at the next revision.

10 Test report

The test report shall include the following information:

a) a complete identification of the sample;

b) reference to this International Standard, i.e. ISO 2070:1997;

c) the results, expressed in accordance with clause 8;

d) details of any unusual features noted during the determination;

e) details of any operation not included in this International Standard or in the International Standard to which reference is made, as well as any operation regarded as optional.