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INTERNATIONAL STANDARD ISO 2829

G-01-33

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION •МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Aluminium oxide primarily used for the production of aluminium — Determination of phosphorus content — Reduced phosphomolybdate spectrophotometric method

First edition — 1973-12-15

UDC 661.862.22 : 546.18 : 543.42

Ref. No. ISO 2829-1973 (E)

Descriptors : aluminium oxide, chemical analysis, determination of content, phosphorus, spectrophotometry.

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Price based on 3 pages

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.

International Standard ISO 2829 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in June 1972.

It has been approved by the Member Bodies of the following countries :

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This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

The Member Body of the following country expressed disapproval of the document on technical grounds :

New Zealand

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Determination of phosphorus content — Reduced phosphomolybdate spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method using reduced ammonium phosphomolybdate for the determination of the phosphorus content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to phosphorus contents, expressed as P_2O_5, greater than 0,000 5 %.

2 REFERENCE

ISO/R 804, *Aluminium oxide primarily used for the production of aluminium — Preparation of sample solution for analysis.*

3 PRINCIPLE

Preparation of a solution from a test portion by alkaline fusion, either with a mixture of sodium carbonate and boric acid, or with a mixture of sodium carbonate and sodium tetraborate. Dissolution of the melt in nitric acid and adjustment of an appropriate aliquot portion to pH 2.

Formation of the phosphomolybdic complex and extraction by 2-methylpropan-1-ol in a sulphuric acid medium.

Reduction of the complex by tin(II) chloride in the organic phase and spectrophotometric measurement of the reduced complex contained in the organic phase at a wavelength of about 730 nm.

4 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

4.1 Sodium carbonate, anhydrous.

4.2 Boric acid (H_3BO_3), or alternatively

4.2.1 Sodium tetraborate, anhydrous ($Na_2B_4O_7$).

4.3 2-Methylpropan-1-ol (*iso*butyl alcohol), ρ approximately 0,805 g/ml.

4.4 Nitric acid, approximately 8 N solution.

Dilute 540 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution, with water to 1 000 ml.

4.5 Sulphuric acid, approximately 10 N solution.

Carefully add, in small portions, 280 ml of sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, to about 500 ml of water and, after cooling, dilute to 1 000 ml.

4.6 Sulphuric acid, approximately N solution.

Take 100 ml of the sulphuric acid solution (4.5) and dilute with water to 1 000 ml.

4.7 Sulphuric acid, approximately 0,5 N solution.

Take 250 ml of the sulphuric acid solution (4.6) and dilute with water to 500 ml.

4.8 Ammonium acetate, 500 g/l solution.

4.9 Iron(III) sulphate, acid solution.

Dissolve 0,5 g of iron(III) sulphate nonahydrate [$Fe_2(SO_4)_3.9H_2O$] in 50 ml of water containing 2 ml of perchloric acid solution, ρ approximately 1,60 g/ml, about 64,5 % (*m/m*) solution, and dilute to 100 ml.

1 ml of this solution contains about 0,001 g of Fe(III).

NOTE — If iron(III) sulphate nonahydrate of recognized analytical reagent quality is not available, ammonium iron(III) sulphate dodecahydrate may be used. In this case, to obtain a solution with the same concentration of iron(III), dissolve, instead of 0,5 g of iron(III) sulphate nonahydrate, 0,86 g of ammonium iron(III) sulphate dodecahydrate [$FeNH_4(SO_4)_2.12H_2O$].

4.10 Ammonium iron(II) sulphate, acid solution.

Dissolve 0,5 g of ammonium iron(II) sulphate hexahydrate [$Fe(NH_4)_2(SO_4)_2.6H_2O$] in 50 ml of water containing 0,5 ml of perchloric acid solution, ρ approximately 1,60 g/ml, about 64,5 % (*m/m*) solution, and dilute to 100 ml.

1 ml of this solution contains about 0,000 7 g of Fe(II).

Prepare this solution just before use.

4.11 Ammonium molybdate, 25 g/l acid solution.

Dissolve 5 g of ammonium molybdate tetrahydrate [$(NH_4)_6Mo_7O_{24}.4H_2O$] in water at 60 °C. Cool and dilute to 100 ml. Add to the solution 100 ml of the sulphuric acid solution (4.5) and mix.

Store the solution in a plastics bottle.

4.12 Washing solution.

Saturate, at ambient temperature, approximately 500 ml of the sulphuric acid solution (4.7) with the 2-methylpropan-1-ol (4.3).

4.13 Tin(II) chloride, 2,38 g/l hydrochloric solution.

Dissolve 1,19 g of tin(II) chloride dihydrate ($SnCl_2.2H_2O$) in 85 ml of hydrochloric acid solution, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution, dilute to 500 ml and transfer the solution into a plastics bottle.

Prepare this solution just before use.

4.14 Phosphorus standard solution corresponding to 0,400 g of P_2O_5 per litre.

Weigh, to the nearest 0,000 1 g, 0,766 8 g of anhydrous potassium dihydrogen phosphate (KH_2PO_4), previously dried over about 12 N sulphuric acid. Dissolve in water, quantitatively transfer the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,4 mg of P_2O_5.

4.15 Phosphorus standard solution corresponding to 0,010 g of P_2O_5 per litre.

Place 25,0 ml of the standard phosphorus solution (4.14) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,01 mg of P_2O_5.

4.16 Phosphorus standard solution corresponding to 0,001 0 g of P_2O_5 per litre.

Place 25,0 ml of the standard phosphorus solution (4.15) in a 250 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,001 mg of P_2O_5.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Apparatus specified in ISO/R 804.

5.2 Burette, graduated in 0,05 ml divisions (see ISO/R 385).

5.3 Separating funnels, 200 ml, with ground glass stoppers.

5.4 pH meter, fitted with a glass electrode.

5.5 Spectrophotometer.

NOTE — All glassware, including the reagent bottles, shall be of borosilicate or other quality of glass unaffected by phosphorus.

Alternatively, plastics apparatus may be used. Wash all the glassware carefully with about 6 N hydrochloric acid solution, then rinse thoroughly with water.

6 PROCEDURE

6.1 Preparation of the test solution (principal solution P).

Using the apparatus specified in 5.1, follow the instructions given in clauses 5.1, 5.2 and 5.3 of ISO/R 804, to give a volume of 250 ml of principal solution P.

6.2 Blank test

At the same time as the determination, carry out a blank test in the absence of the pure aluminium oxide, according to the instructions of clause 5.4.2 of ISO/R 804. Then proceed using the same quantities of all reagents used for the determination and following the same procedure.

6.3 Preparation of the calibration curve

6.3.1 *Preparation of standard colorimetric solutions,* referring to spectrophotometric measurements made with an optical path length of 1 cm.

6.3.1.1 TAKING ALIQUOT PORTIONS OF THE STANDARD SOLUTION

Into a series of six of the separating funnels (5.3) measure, by means of the burette (5.2), the volumes of the standard phosphorus solution (4.16) shown in the following table :

Standard phosphorus solution (4.16)	Corresponding mass of P_2O_5
ml	mg
0*	0
5,0	0,005
10,0	0,010
15,0	0,015
20,0	0,020
25,0	0,025

* Compensation solution.

Add to each separating funnel the quantity of water required to increase the volume to 60 ml, then add 1 ml of the iron(III) sulphate solution (4.9) and 1 ml of the ammonium iron(II) sulphate solution (4.10) to each funnel and mix.

6.3.1.2 FORMATION OF THE OXIDIZED PHOSPHOMOLYBDATE COMPLEX AND EXTRACTION OF THE REDUCED COMPLEX

Add to each separating funnel 3 ml of the sulphuric acid solution (4.6), then 7,5 ml of the ammonium molybdate solution (4.11), mix and leave to stand for 10 min. Add 25 ml of the 2-methylpropan-1-ol (4.3) and shake vigorously for 1 min. Allow the layers to separate, run off the aqueous phase and discard it. To the organic phase add 30 ml of the washing solution (4.12), shake for 1 min, run off the aqueous phase and discard it. Repeat this washing once more.

Then add 30 ml of the sulphuric acid solution (4.6) and 0,5 ml of the tin(II) chloride solution (4.13), shake for 30 s, allow to settle, run off the aqueous phase and discard it.

Transfer the organic phase to a 25 ml one-mark volumetric flask which has been previously dried. Wash the separating funnel with 1 to 2 ml of the 2-methylpropan-1-ol (4.3), transferring the washings to the same volumetric flask. Then dilute to the mark with the same 2-methylpropan-1-ol (4.3). Mix and leave to stand, shielded from light, for at least 10 min.

6.3.2 *Spectrophotometric measurements*

After allowing to stand for at least 10 min but not more than 60 min, carry out the spectrophotometric measurements using the spectrophotometer (5.5) at a wavelength of about 730 nm after having adjusted the instrument to zero absorbance against the compensation solution.

6.3.3 *Preparation of the calibration chart*

Plot a graph having, for example, the values, expressed in milligrams, of the masses of P_2O_5 per 25 ml of the standard colorimetric solutions on the abscissae and the corresponding values of absorbance on the ordinates.

6.4 Determination

6.4.1 *Treatment of the test solution*

Take a volume of the test solution (6.1) containing a mass of phosphorus corresponding to 0,015 to 0,025 mg of P_2O_5. Adjust the pH to 2 ± 0,1 by the addition, in small portions, of the ammonium acetate solution (4.8), checking by means of the pH meter (5.4). Transfer the solution quantitatively to one of the separating funnels (5.3).

Increase the volume of the solution to about 60 ml and then add 1 ml of the iron(III) sulphate solution (4.9), 1 ml of the ammonium iron(II) sulphate solution (4.10) and mix.

6.4.2 *Formation of the oxidized phosphomolybdic complex and extraction of the reduced complex*

Follow the instructions in 6.3.1.2.

6.4.3 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements on the organic phase resulting from the test solution and on that originating from the blank test solution, according to the instructions in 6.3.2, after having adjusted the spectrophotometer (5.5) to zero absorbance against the 2-methylpropan-1-ol (4.3).

7 EXPRESSION OF RESULTS

By means of the calibration chart (6.3.3), determine the mass of P_2O_5 corresponding to the absorbance values.

The phosphorus pentoxide content (P_2O_5) is given, as a percentage by mass, by the formula

$$\frac{(m_1 - m_2) \times D \times 100}{1\,000 \times m_0} = \frac{(m_1 - m_2) \times D}{10 \times m_0}$$

where

m_0 is the mass, in grams, of the test portion used for the preparation of the principal solution P;

m_1 is the mass, in milligrams, of P_2O_5 found in the aliquot portion of the test solution;

m_2 is the mass, in milligrams, of P_2O_5 found in a corresponding aliquot portion of the blank test solution;

D is the ratio of the volume of the principal solution P to the volume of the aliquot portion of this solution taken for the determination.

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 the document to which reference is made, or regarded as optional.