INTERNATIONAL STANDARD ISO 1618

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

Aluminium oxide primarily used for the production of aluminium – Determination of vanadium content – *N*-Benzoyl-*N*-phenylhydroxylamine photometric method

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium – Dosage du vanadium – Méthode photométrique à la N*-benzoyl-*N*-phénylhydroxylamine*

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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 1618 and found it technically suitable for transformation. International Standard ISO 1618 therefore replaces ISO Recommendation R 1618-1970 to which it is technically identical.

ISO Recommendation R 1618 was approved by the Member Bodies of the following countries :

Australia
Austria
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France
Germany
Greece
Hungary
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Iran
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Italy
Korea, Rep. of
Netherlands
New Zealand
Norway
Peru
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Sweden
Switzerland
Thailand
Turkey
United Kingdom
U.S.S.R.
Yugoslavia

No Member Body expressed disapproval of the Recommendation.

The Member Bodies of the following countries disapproved the transformation of ISO/R 1618 into an International Standard :

Romania
United Kingdom

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Determination of vanadium content — *N*-Benzoyl-*N*-phenylhydroxylamine photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an *N*-benzoyl-*N*-phenylhydroxylamine photometric method for the determination of the vanadium content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products having a vanadium content, expressed as V_2O_5, between 0,000 3 and 0,016 % (*m/m*), provided that the Cr_2O_3 and the TiO_2 contents do not exceed 0,002 and 0,006 % (*m/m*) respectively.

1.1 Special cases (under study)

Cr_2O_3 contents greater than 0,002 % (*m/m*) and TiO_2 contents greater than 0,006 % (*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 2927, *Aluminium oxide primarily used for the production of aluminium — Sampling.*

3 PRINCIPLE

Preliminary oxidation of the vanadium present in a test portion to vanadium(V) using potassium permanganate in a 4,5 N sulphuric acid medium.

Formation of the vanadium *N*-benzoyl-*N*-phenylhydroxylamine complex. Extraction of the (violet) coloured complex, by means of chloroform in a 3,5 N hydrochloric acid medium.

Photometric measurement of the coloured complex at a wavelength of about 524 nm.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Sodium carbonate, anhydrous.

4.2 Boric acid (H_3BO_3), or

4.2.1 Boron trioxide (B_2O_3).

4.3 Sodium sulphate, anhydrous.

4.4 Chloroform, free from ethanol.

Purify the chloroform, ρ approximately 1,49 g/ml, by washing it five or six times with a volume of water equal to half the volume of the chloroform treated. Dry on anhydrous calcium chloride and distil, collecting the distillate in a dark glass container. Keep in a cool place (temperature below 25 °C) and away from the light.

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

4.6 Sulphuric acid, approximately 16 N solution.

Carefully pour 450 ml of sulphuric acid solution, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, into 500 ml of water, cool, dilute to 1 000 ml and mix.

4.7 Sulphuric acid, approximately 8 N solution.

Carefully pour 225 ml of sulphuric acid solution, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, into approximately 500 ml of water, cool, dilute to 1 000 ml and mix.

4.8 Potassium permanganate, 0,6 g/l solution.

4.9 *N*-Benzoyl-*N*-phenylhydroxylamine, 1 g/l solution in chloroform.

Dissolve 0,1 g of *N*-benzoyl-*N*-phenylhydroxylamine in 100 ml of purified chloroform (4.4).

4.10 Vanadium, standard solution corresponding to 1,000 g of V_2O_5 per litre.

Weigh, to the nearest 0,001 g, 1 g of V_2O_5, previously dried at 110 °C and cooled in a desiccator, transfer it to a beaker of suitable capacity (for example 250 ml) and add 20 ml of a 5 % (*m/m*) sodium hydroxide solution.

After dissolving, acidify with 12 ml of the sulphuric acid solution (4.6), transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 1,0 mg of V_2O_5.

4.11 Vanadium, standard solution corresponding to 0,10 g of V_2O_5 per litre.

Take 50,0 ml of the standard solution (4.10), transfer to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,10 mg of V_2O_5.

4.12 Vanadium, standard solution corresponding to 0,010 g of V_2O_5 per litre.

Take 50,0 ml of the standard solution (4.1), transfer to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,010 mg of V_2O_5.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Separating funnels, having a very short stem, and of capacity approximately 150 ml.

5.2 Spectrophotometer, or

5.3 Photoelectric absorptiometer, fitted with filters ensuring a maximum transmission between 520 and 530 nm.

6 PROCEDURE

6.1 Aliquot portion of principal solution (test portion)

Take 50,0 ml of the principal solution P (prepared in accordance with 6.1, 6.2 and 6.3 of ISO 804, but using the sulphuric acid solution (4.7) instead of nitric acid solution to take up the fused mass, and diluting to 250 or 500 ml, according to the V_2O_5 content).

6.2 Preparation of the calibration graph

6.2.1 *Preparation of standard colorimetric solutions* for photometric measurements with a cell of optical path length equal to or greater than 3 cm

Into a series of six separating funnels (5.1) which have been completely dried and the taps of which have previously been moistened with the chloroform (4.4), place the volumes of the standard vanadium solution (4.12) indicated in the following table :

Standard vanadium solution (4.12)	Corresponding mass of V_2O_5
ml	mg
0*	0
1,00	0,010
2,00	0,020
4,00	0,040
6,00	0,060
8,00	0,080

* Compensation solution.

Add to each funnel 20 ml of the sulphuric acid solution (4.6), dilute with water to approximately 70 ml, and cool. (The temperature must not exceed 25 °C.) Then add a few (3 or 4) drops of the potassium permanganate solution (4.8) until the solution shows a faintly pink coloration. Stir gently and wait 5 min for complete oxidation of the vanadium to take place.

6.2.2 *Colour development*

In order to avoid reduction of the vanadium, the addition of the cooled hydrochloric acid solution to the standard colorimetric solutions must be carried out immediately before extraction of the complex. It is therefore necessary to prepare only one point of the calibration graph at a time.

Add 10 ml of the *N*-benzoyl-*N*-phenylhydroxylamine solution (4.9) and, without stirring, 32 ml of the hydrochloric acid solution (4.5), which has previously been cooled to approximately 10 °C. Shake for 1 min and leave to stand until the two phases are completely separate.

Draw off the chloroform phase and collect it in a perfectly dry 50 ml one-mark volumetric flask in which approximately 1 g of the sodium sulphate (4.3) has already been placed. Wash the residual aqueous phase in the separating funnel once by shaking it with 10 ml of the chloroform (4.4) in order to extract the last traces of vanadium. Draw off the chloroform phase and collect it in the same flask. Dilute to the mark with the chloroform, mix, and leave to stand for 10 min.

6.2.3 *Photometric measurements*

Carry out the photometric measurements using either the spectrophotometer (5.2) at a wavelength of about 524 nm, or the photoelectric absorptiometer (5.3) with a suitable filter, after having adjusted the instrument to zero absorbance against the compensation solution.

6.2.4 *Plotting of the calibration graph*

Plot a graph having, for example, the V_2O_5 contents in milligrams per 50 ml of standard colorimetric solution as abscissae and the corresponding values of absorbance as ordinates.

6.3 Determination

6.3.1 *Preparation of the test solution*

Place the test portion (6.1) in a separating funnel (5.1). Add 20 ml of the sulphuric acid solution (4.6) and proceed as specified in 6.2.1, first paragraph after the table.

6.3.2 *Colour development*

Carry out the colour development as specified in 6.2.2.

6.3.3 *Photometric measurement*

Carry out the photometric measurement as specified in 6.2.3, after having adjusted the instrument to zero absorbance against the blank test solution (6.4.2).

6.4 Blank test

6.4.1 *Preparation of the solution*

Prepare the solution for the blank test, in the absence of extra-pure aluminium oxide, by directly dissolving 12 g of the sodium carbonate (4.1) and 4 g of the boric acid (4.2) or 2,25 g of the boron trioxide (4.2.1) in approximately 150 ml of hot water and 30 ml of the sulphuric acid solution (4.7). Boil for 10 min to eliminate the carbon dioxide; allow to cool and transfer the solution quantitatively to a volumetric flask of a capacity equal to that used for the preparation of the principal solution P (see 6.1). Place 36,7 ml of the sulphuric acid solution in a platinum dish of diameter approximately 70 mm, and evaporate until almost dry. Add to the dish a little hot water and 3,3 ml of the sulphuric acid solution. Heat, if necessary, and after cooling transfer the solution quantitatively to the flask containing the solution for the blank test. Cool, dilute to the mark and mix. Take 50,0 ml of the solution and proceed as specified in 6.3.1.

6.4.2 *Colour development*

Carry out the colour development as specified in 6.3.2.

7 EXPRESSION OF RESULTS

By means of the calibration graph (see 6.2.4), determine the quantity of V_2O_5 corresponding to the value of the photometric measurement of the test solution.

The vanadium content, expressed as a percentage by mass of V_2O_5, is given by the formula

$$m_1 \times \frac{D}{10 \times m_0}$$

where

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

m_1 is the mass, in milligrams, of V_2O_5 found in the aliquot portion of the principal solution P;

D is the ratio between the volume of the principal solution P and the volume of the aliquot portion 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 feature noted during the determination;

d) all operations 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.