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Aluminium oxide primarily used for the production of aluminium — Determination of titanium content — Diantipyrylmethane photometric method

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Dosage du titane — Méthode photométrique au diantipyrylméthane

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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.

International Standard ISO 900 was developed by Technical Committee ISO/TC 47, *Chemistry,* and was circulated to the member bodies in September 1975.

It has been approved by the member bodies of the following countries :

Austria
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Germany
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Italy
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Netherlands
Poland
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Spain
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Thailand
Turkey
United Kingdom

No member body expressed disapproval of the document.

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

It cancels and replaces ISO Recommendation R 900-1968, of which it constitutes a technical revision.

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Aluminium oxide primarily used for the production of aluminium — Determination of titanium content — Diantipyrylmethane photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a diantipyrylmethane photometric method for the determination of the titanium content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products having a titanium content, expressed as titanium dioxide (TiO_2), equal to or greater than 0,001 % (*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 sample solution for analysis — Method by alkaline fusion.*

3 PRINCIPLE

Alkaline fusion of a test portion and extraction of the fused mass with sulphuric acid solution. Formation of the titanium-diantipyrylmethane complex in approximately 4,6 N sulphuric acid solution.

Photometric measurement of the coloured complex at a wavelength of approximately 420 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 **Sodium tetraborate,** anhydrous ($Na_2B_4O_7$).

4.3 **Sulphuric acid,** approximately 8 N solution.

4.4 **Sulphuric acid,** approximately 18 N solution.

4.5 **Ascorbic acid,** 30 g/l solution.

Use a freshly prepared solution.

4.6 **Copper(II) sulphate** pentahydrate ($CuSO_4.5H_2O$), 50 g/l solution.

4.7 **Diantipyrylmethane,** 50 g/l hydrochloric acid solution.

Dissolve 5 g of 4,4′-methylenediantipyrine (diantipyrylmethane) ($C_{23}H_{24}N_4O_2$) in 100 ml of approximately 1 N hydrochloric acid solution.

4.8 **Titanium,** standard solution corresponding to 0,400 g of titanium dioxide (TiO_2) per litre.

Prepare the solution by either of the following two methods :

4.8.1 In a platinum dish of suitable capacity, weigh, to the nearest 0,000 1 g, 0,601 5 g of *di*potassium hexafluorotitanate (K_2TiF_6) previously dried at about 50 °C and cooled in a desiccator. Moisten the product with a few drops of water and then add 15 ml of sulphuric acid solution, ρ approximately 1,84 g/ml. Evaporate carefully almost to dryness in a well-ventilated fume cupboard. Repeat the operation until the fluorine is completely expelled, using each time 5 to 6 ml of the same sulphuric acid solution. Finally, add 3 ml of the same sulphuric acid solution and heat until the residue is completely dissolved.

Allow to cool, immerse the dish and its contents in a beaker containing 95 ml of water and 5 ml of the same sulphuric acid solution and heat on a boiling water bath until the solution is completely clear.

Remove the platinum dish, wash it carefully with water, transfer the solution and the washings quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains the equivalent of 0,400 mg of TiO_2.

4.8.2 Weigh, to the nearest 0,000 1 g, 0,886 5 g of *di*potassium titanyl dioxalate dihydrate $[K_2TiO(C_2O_4)_2.2H_2O]$ and place in a Kjeldahl flask of capacity about 100 ml. Add 0,80 g of ammonium sulphate and 10 ml of sulphuric acid solution, ρ approximately 1,84 g/ml. Heat carefully until the reaction subsides and then boil for 10 min.

Cool and transfer the solution quantitatively to a beaker of suitable capacity containing 100 ml of water. Add, drop by drop, approximately 0,1 N potassium permanganate solution until the colour of the solution is permanently pink. Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains the equivalent of 0,400 mg of TiO_2.

NOTE — If *di*potassium hexafluorotitanate or *di*potassium titanyl dioxalate dihydrate is not available, the following alternative procedure may be adopted :

In a platinum crucible of suitable capacity, weigh, to the nearest 0,000 1 g, 0,200 0 g of titanium dioxide (TiO_2) previously calcined at 1 200 °C and cooled in a desiccator. Add 4 g of potassium pryosulphate ($K_2S_2O_7$) and fuse carefully at about 1 200 °C. Allow to cool and place the crucible and contents in a beaker of suitable capacity. Add 100 ml of the sulphuric acid solution (4.4) and heat gently to dissolve the melt. Remove the crucible and wash it with hot water, collecting the washings in the beaker. Transfer the solution and the washings quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

4.9 Titanium, standard solution corresponding to 0,040 g of titanium dioxide (TiO_2) per litre.

Transfer 50,0 ml of the standard titanium solution (4.8) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains the equivalent of 0,040 mg of TiO_2.

Prepare this solution at the time of use.

4.10 Titanium, standard solution corresponding to 0,010 g of titanium dioxide (TiO_2) per litre.

Transfer 25,0 ml of the standard titanium solution (4.9) to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains the equivalent of 0,010 mg of TiO_2.

Prepare this solution at the time of use.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Burette, graduated in 0,05 ml, complying with the requirements of ISO/R 385.

5.2 Spectrophotometer, or

5.3 Photoelectric absorptiometer, fitted with suitable filters, having maximum absorption at about 420 nm.

6 PROCEDURE

6.1 Preparation of the test solution (principal solution P)

Proceed in accordance with sub-clauses 6.1, 6.2 and 6.3 of ISO 804, replacing the nitric acid solution (4.5 of ISO 804) by the sulphuric acid solution (4.3) and diluting the principal solutions to 250 ml.

6.2 Test portion

Place 50,0 ml of the test solution (6.1), containing 1,000 g of the dried sample (see sub-clause 3.3 of ISO 802), in a 100 ml one-mark volumetric flask.

NOTE — If the TiO_2 content is greater than 0,01 % (*m/m*), reduce the test portion proportionately and take this into account in the calculation of the results.

6.3 Preparation of the calibration graph

6.3.1 *Preparation of the standard colorimetric solutions,* for photometric measurements using 4 cm cells

Into a series of seven 100 ml one-mark volumetric flasks transfer, by means of the burette (5.1), the volumes of the standard titanium solution (4.10) shown in the following table :

Standard titanium solution (4.10)	Corresponding mass of TiO_2
ml	mg
0*	0
1,00	0,010
2,00	0,020
3,00	0,030
4,00	0,040
6,00	0,060
10,00	0,100

* Compensation solution.

Add 25 ml of the sulphuric acid solution (4.4) to each flask. Dilute with water to about 70 ml, add 3 ml of the ascorbic acid solution (4.5) and 3 drops of the copper(II) sulphate solution (4.6) and mix. Allow to stand for 20 min, add 10 ml of the diantipyrylmethane solution (4.7), dilute to the mark and mix.

6.3.2 *Photometric measurements*

After at least 40 min, carry out the photometric measurements using the spectrophotometer (5.2) at a wavelength of about 420 nm or the photoelectric absorptiometer (5.3) fitted with suitable filters, after having adjusted the instrument to zero absorbance against the compensation solution.

NOTE — The absorbance of the solution is stable for about 24 h.

6.3.3 *Plotting of the calibration graph*

Plot a graph having, for example, the values of the masses, in milligrams, of titanium dioxide (TiO_2) contained in 100 ml of standard colorimetric solution, as abscissae and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 *Colour development*

To the test portion (6.2) placed in the 100 ml one-mark volumetric flask, add 20 ml of the sulphuric acid solution (4.4), 3 ml of the ascorbic acid solution (4.5) and 3 drops of the copper(II) sulphate solution (4.6) and mix. Allow to stand for 20 min, add 10 ml of the diantipyrylmethane solution (4.7), dilute to the mark and mix.

6.4.2 *Photometric measurement*

After at least 40 min, carry out the photometric measurement following the procedure specified in 6.3.2, after having adjusted the instrument to zero absorbance against water.

6.5 Blank test

6.5.1 *Preparation of the blank test solution*

Proceed in accordance with sub-clause 6.4.2 of ISO 804, replacing the nitric acid solution (4.5 of ISO 804) by the sulphuric acid solution (4.3) and diluting the solution to 250 ml.

6.5.2 *Colour development*

Proceed as specified in 6.4.1.

6.5.3 *Photometric measurement*

After at least 40 min, carry out the photometric measurement following the procedure specified in 6.3.2, after having adjusted the instrument to zero absorbance against water.

7 EXPRESSION OF RESULTS

From the calibration graph (see 6.3.3), determine the masses of titanium dioxide (TiO_2) corresponding to the absorbance of the test solution and to that of the blank test solution.

The titanium content, expressed as a percentage by mass of titanium dioxide (TiO_2), is given by the formula

$$\frac{m_1 - m_2}{1\,000} \times \frac{250}{50} \times \frac{100}{m_0}$$

$$= \frac{m_1 - m_2}{2\,m_0}$$

where

m_0 is the mass, in grams, of the test portion used for the preparation of the test solution (6.1);

m_1 is the mass, in milligrams, of titanium dioxide found in the aliquot portion of the test solution (6.1);

m_2 is the mass, in milligrams, of titanium dioxide found in a corresponding aliquot portion of the blank test solution.

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.