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INTERNATIONAL STANDARD 805

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 iron content — 1,10-Phenanthroline photometric method

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Dosage du fer — Méthode photométrique à la phénanthroline-1,10

First edition — 1976-09-15

UDC 661.862.22 : 546.72 : 543.42 **Ref. No. ISO 805-1976 (E)**

Descriptors : aluminium oxide, chemical analysis, determination of content, iron, spectrophotometric analysis.

Price based on 4 pages

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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 805 and fount it technically suitable for transformation. International Standard ISO 805 therefore replaces ISO Recommendation R 805-1968 to which it is technically identical.

ISO Recommendation R 805 was approved by the Member Bodies of the following countries :

Austria	Hungary	Romania
Belgium	Korea, Rep. of	South Africa, Rep. of
Brazil	India	Spain
Bulgaria	Ireland	Sweden
Canada	Israel	Switzerland
Chile	Italy	Turkey
Czechoslovakia	Japan	United Kingdom
Egypt, Arab Rep. of	Netherlands	U.S.A.
France	Norway	U.S.S.R.
Germany	Poland	Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 805 into an International Standard.

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Aluminium oxide primarily used for the production of aluminium — Determination of iron content — 1,10-Phenanthroline photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a 1,10-phenanthroline photometric method for the determination of the iron content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products having an iron content, expressed as iron(III) oxide, equal to or greater than 0,005 % (*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 reduction of iron(III) by means of hydroxylammonium chloride.

Formation of the iron(II)-1,10-phenanthroline complex in a buffered medium (pH value between 3,5 and 4,2).

Photometric measurement of the coloured complex at a wavelength of about 510 nm.

NOTE — Aluminium, elements usually present in aluminium oxide (impurities) and flux do not cause interference.

4 REAGENTS

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

4.1 Hydroxylammonium chloride ($NH_2OH.HCl$), 10 g/l solution.

4.2 1,10-Phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2.HCl.H_2O$), 2,5 g/l solution.

This reagent may be replaced by a 2,5 g/l solution of 1,10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$).

4.3 Buffer solution, of pH 4,90.

Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in approximately 500 ml of water. Add 240 ml of glacial acetic acid, ρ approximately 1,05 g/ml, about 17,4 N. Dilute to 1 000 ml and mix.

4.4 Sodium acetate trihydrate ($CH_3COONa.3H_2O$), 500 g/l solution.

4.5 Acetic acid solution.

Dilute 500 ml of glacial acetic acid, ρ approximately 1,05 g/ml, about 17,4 N, with water to 1 000 ml.

4.6 Iron, standard solution corresponding to 0,200 g of iron(III) oxide per litre.

Prepare this solution by either of the following two methods :

4.6.1 Weigh, to the nearest 0,001 g, 0,982 g of ammonium iron(II) sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2.6H_2O$]. Place in a beaker of suitable capacity (for example 100 ml) and dissolve in water.

Add 20 ml of a solution of sulphuric acid, ρ approximately 1,84 g/ml, allow to cool, 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 0,200 mg of Fe_2O_3.

4.6.2 Weigh, to the nearest 0,001 g, 0,200 g of iron(III) oxide previously ignited at 600 °C and allowed to cool in a desiccator. Place it in a beaker of suitable capacity (for example 100 ml), add 10 ml of a hydrochloric acid solution, ρ approximately 1,19 g/ml, and heat gently until it is completely dissolved. Allow to cool, 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 0,200 mg of Fe_2O_3.

4.7 Iron, standard solution corresponding to 0,010 g of iron(III) oxide per litre.

Transfer 50,0 ml of the standard iron solution (4.6) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,010 mg of Fe_2O_3.

Prepare this solution just before use.

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4.8 pH paper, covering the pH range 3,5 to 4,2 at intervals of 0,2 unit.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 pH meter.

5.2 Spectrophotometer, or

5.3 Photoelectric absorptiometer, fitted with filters ensuring a maximum transmission between 500 and 520 nm.

6 PROCEDURE

6.1 Aliquot portion of principal solution (test portion)

Depending on the iron content to be determined, place two aliquot portions of the principal solution P (see 6.3 of ISO 804) in a beaker of suitable capacity and in a 100 ml one-mark volumetric flask respectively, according to table 1.

TABLE 1

Fe_2O_3 content	Principal solution P	Aliquot portions to be taken	
		Volume	Corresponding mass of test portion
% (*m/m*)	ml	ml	g
0,005 to 0,01	250	50,0	1
0,01 to 0,04	500	50,0	0,50
higher than 0,04	500	25,0	0,25

6.2 Blank test

Carry out at the same time as the determination and following the same procedure, a blank test using the same quantities of all reagents as used for the determination.

Use the blank test solution free from extra-pure aluminium oxide for taking the aliquot portions (see 6.4.2 of ISO 804).

6.3 Preparation of calibration graph

6.3.1 *Preparation of the standard colorimetric solutions,* for photometric measurements with a cell of optical path length 1 cm

Into each of a series of seven 100 ml one-mark volumetric flasks, place respectively the volumes of standard iron solution (4.7) shown in table 2.

TABLE 2

Standard iron solution (4.7)	Corresponding mass of Fe_2O_3
ml	mg
0*	0
2,5	0,025
5,0	0,050
10,0	0,100
15,0	0,150
20,0	0,200
25,0	0,250

* Compensation solution.

Add to each volumetric flask an amount of water sufficient to dilute to approximately 50 ml, then add 5 ml of the hydroxylammonium chloride solution (4.1), 5 ml of the 1,10-phenanthroline solution (4.2) and 25 ml of the buffer solution (4.3). Dilute to the mark and mix.

6.3.2 *Photometric measurements*

After 10 min, carry out the photometric measurements using either the spectrophotometer (5.2) adjusted to a wavelength of about 510 nm or the photoelectric absorptiometer (5.3) fitted with suitable filters, after having adjusted the instrument to zero absorbance against the compensation solution.

6.3.3 *Preparation of calibration graph*

Plot a graph having, for example, the iron(III) oxide content, in milligrams per 100 ml of standard colorimetric solution, as abscissae and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 *Preliminary test for control and adjustment of pH*

To the aliquot portion of the principal solution placed in the beaker (see 6.1), add an amount of water sufficient to dilute to approximately 60 ml. Then add 5 ml of the hydroxylammonium chloride solution (4.1), 5 ml of the 1,10-phenanthroline solution (4.2) and 25 ml of the buffer solution (4.3).

Check the pH value by means of the pH paper (4.8) or the pH meter (5.1). The value should be between 3,5 and 4,2; if necessary, adjust the pH value by adding slowly, stirring after each addition, the necessary amount either of the sodium acetate solution (4.4) or of the acetic acid solution (4.5).

Note the quantity of reagent added for the pH adjustment and discard the solution.

6.4.2 *Colour development*

To the other aliquot portion of the principal solution placed in the 100 ml one-mark volumetric flask (see 6.1), add the same quantities of all reagents as used for the preliminary test (6.4.1). Dilute to the mark and mix.

6.4.3 *Photometric measurements*

After 10 min, carry out the photometric measurements of the test solution (6.4.2) and the blank test solution (6.2) following the procedure specified in 6.3.2, after having adjusted the instrument to zero absorbance against water (see note in clause 7).

7 EXPRESSION OF RESULTS

By reference to the calibration graph (see 6.3.3), read the iron contents corresponding to the values of the photometric readings of the principal solution P, and the corresponding blank test solution.

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The iron content, expressed as a percentage by mass of iron(III) oxide, is given by the formula

$$(m_1 - m_2) \times \frac{D}{10 \times m_0}$$

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 iron(III) oxide determined in the aliquot portion of the principal solution P taken for the determination;

m_2 is the mass, in milligrams, of iron(III) oxide determined in the aliquot portion corresponding to the blank test solution;

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

NOTE — The aliquot portion of the blank test solution after preparation for the photometric determination generally shows a slight coloration. In this case, it is advisable to use it as compensation solution. The formula for expression of results then becomes

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

where the symbols have the same meaning as in the preceding formula.

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.

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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.

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