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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 805

ALUMINIUM OXIDE

PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF IRON CONTENT

1,10-PHENANTHROLINE PHOTOMETRIC METHOD

1st EDITION

August 1968

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BRIEF HISTORY

The ISO Recommendation R 805, *Aluminium oxide primarily used for the production of aluminium – Determination of iron content – 1,10- phenanthroline photometric method,* was drawn up by Technical Committee ISO/TC 47, *Chemistry,* the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question by the Technical Committee began in 1961 and led, in 1964, to the adoption of a Draft ISO Recommendation.

In July 1966, this Draft ISO Recommendation (No. 1027) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in August 1968, to accept it as an ISO RECOMMENDATION.

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ISO Recommendation R 805 August 1968

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1. SCOPE

This ISO Recommendation describes a method for the photometric determination of iron content of aluminium oxide.

2. FIELD OF APPLICATION

The method is applicable to iron contents, expressed as iron (III) oxide (Fe_2O_3), greater than 0.005 %.

3. PRINCIPLE

Preliminary reduction of trivalent iron by means of hydroxylammonium chloride.

Formation of the bivalent iron - 1,10 - phenanthroline complex in a buffered medium (pH value between 3.5 and 4.2)*.

Photometric measurement at a wavelength of about 510 nm.

4. REAGENTS

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

4.1 *Hydroxylammonium chloride,* 10 g/l solution. Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in water and dilute to 1000 ml.

4.2 *1,10-phenanthroline hydrochloride,* 2.5 g/l solution. Dissolve 2.5 g of 1,10-phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2.HCl.H_2O$) in water and dilute to 1000 ml. (1,10-phenanthroline hydrochloride monohydrate may be replaced by 1,10-phenanthroline monohydrate.

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

4.3 *Buffer solution.* Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in approximately 500 ml of water. Add 240 ml of glacial acetic acid (CH_3COOH) approximately 17.4 N and dilute to 1000 ml.

4.4 *Sodium acetate,* 500 g/l solution. Dissolve 50 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in water and dilute to 100 ml.

4.5 *Acetic acid solution.* Dilute 500 ml of glacial acetic acid (CH_3COOH) approximately 17.4 N with water to 1000 ml.

4.6 *Iron, standard solution equivalent to 0.20 g/l of iron (III) oxide (Fe_2O_3).* This solution should be prepared by one of the two following methods :

4.6.1 Weigh to the nearest 1 mg, 0.982 g of ammonium-iron (II) sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2.6H_2O$] place in a beaker of suitable capacity and dissolve in water.

Add 20 ml of sulphuric acid $d = 1.83$, transfer quantitatively to a 1000 ml one-mark volumetric flask and dilute to the mark.

4.6.2 Weigh to the nearest 1 mg, 0.200 g of iron (III) oxide (Fe_2O_3) previously ignited at 600 °C and allowed to cool in a desiccator. Place it in an approximately 100 ml beaker, add 10 ml of hydrochloric acid solution $d = 1.18$ (approximately 37 % m/m solution) and heat gently until it is completely dissolved. Allow to cool, quantitatively transfer to a 1000 ml one-mark volumetric flask and dilute to the mark.

1 ml of this standard solution is equivalent to 0.2 mg of Fe_2O_3.

4.7 *Iron, standard solution containing 0.010 g/l of iron (III) oxide (Fe_2O_3).* Transfer 50.0 ml of the standard solution (4.6) to a 1000 ml one-mark volumetric flask and dilute to the mark.

1 ml of this standard solution is equivalent to 10 µg of Fe_2O_3.

The solution should be prepared just before use.

4.8 *pH paper,* covering the range 3.5 to 4.2 at intervals of 0.2 units.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *pH meter,* with a glass electrode.

5.2 *Spectrophotometer,* or

5.3 *Photoelectric absorptiometer.*

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6. PROCEDURE

6.1 Test portion

Depending on the iron content to be determined, place two aliquots of the principal solution (P)* in a beaker of suitable capacity and in a 100 ml one-mark volumetric flask respectively, according to the following table.

Fe_2O_3 content	Volume of principal solution (P)	Aliquots to be taken	
		volume	corresponding to a test portion
%	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

Together with the analysis and following the same procedure perform a blank test using the same quantities of all reagents used for the determination.

Use the blank test solution free from pure aluminium oxide ** for drawing the aliquots.

6.3 Preparation of calibration curve

6.3.1 *Preparation of the standard matching solutions* for photometric measurements with a 1 cm cell.

Into each of a series of seven 100 ml one-mark volumetric flasks place respectively :

Iron standard solution (4.7)	Corresponding to
ml	μg of Fe_2O_3
0 (1)	0
2.5	25
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250

(1) 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 with water and mix.

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* See ISO Recommendation R 804, *Aluminium oxide primarily used for the production of aluminium – Preparation of sample solution for analysis*, clauses 5.1, 5.2, 5.3

** See ISO Recommendation R 804, *Aluminium oxide primarily used for the production of aluminium – Preparation of sample solution for analysis*, clause 5.4.2.

6.3.2 *Photometric measurement.* After 10 minutes, carry out the photometric measurement using either the spectrophotometer (5.2) at a wavelength of about 510 nm or the photoelectric absorptiometer (5.3) with a suitable filter, adjusting the instrument to zero optical density using the compensation solution as reference.

6.3.3 *Preparation of calibration graph.* Prepare a calibration graph having for example the iron (III) oxide content in milligrammes per 100 ml of solution as abscissae and the corresponding values of optical density as ordinates.

6.4 Determination

6.4.1 *Preliminary check of the pH.* Add to the aliquot placed in the beaker 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 and stirring after each addition, the necessary amount either of the sodium acetate solution (4.4) or of acetic acid (4.5).

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

6.4.2 *Development of the colour.* To the aliquot of solution placed in the 100 ml one-mark volumetric flask, add the same quantities of all reagents used for the preliminary test (6.4.1). Dilute to the mark and mix thoroughly.

6.4.3 *Photometric measurement.* After 10 minutes carry out the photometric measurement following the same procedure as indicated in clause 6.3.2. Use water as the compensation solution (see Note, section 7).

7. EXPRESSION OF RESULTS

By reference to the calibration graph (see clause 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.

The iron content, expressed as iron (III) oxide (Fe_2O_3), is given as a percentage, by mass, by the following formula :

$$(A-B) \times \frac{D}{10 \times E}$$

where

A is the mass, in milligrammes, of iron (III) oxide (Fe_2O_3) determined in the aliquot of the principal solution (P);

B is the mass, in milligrammes, of iron (III) oxide (Fe_2O_3) determined in the aliquot corresponding to the blank test solution;

D is the ratio of volume of principal solution (P) to volume of aliquot taken;

E is the mass, in grammes, of the sample taken for the preparation of the principal solution (P).

NOTE. – The aliquot 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 :

$$A \times \frac{D}{10 \times E}$$

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

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