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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1618

ALUMINIUM OXIDE

PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF VANADIUM CONTENT

PHOTOMETRIC METHOD USING *N*-BENZOYL-*N*-PHENYLHYDROXYLAMINE

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1618, *Aluminium oxide primarily used for the production of aluminium – Determination of vanadium content – Photometric method using N-benzoyl-N-phenylhydroxylamine*, 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 led to the adoption of Draft ISO Recommendation No. 1618 which was circulated to all the ISO Member Bodies for enquiry in March 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

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

ISO Recommendation | R 1618 | July 1970

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1. SCOPE

This ISO Recommendation describes a photometric method, using *N*-benzoyl-*N*-phenylhydroxylamine, for determining the vanadium content of aluminium oxide primarily used for production of aluminium.

2. FIELD OF APPLICATION

The method applies to the determination of vanadium contents between 0.0003 and 0.016 % of V_2O_5, in the industrial product, provided that the Cr_2O_3 and the TiO_2 impurities do not exceed 0.002 and 0.006 % respectively.

2.1 Special cases (under preparation)

Cr_2O_3 contents greater than 0.002 % and TiO_2 contents greater than 0.006 %.

3. PRINCIPLE

Formation of the vanadium-*N*-benzoyl-*N*-phenylhydroxylamine complex after oxidation of the vanadium present to vanadium (V) by means of permanganate in a 4.5 N sulphuric acid medium.

Extraction of the (violet) coloured complex, by means of chloroform in a 3.5 N hydrochloric acid medium. Photometric measurement at a wavelength of about 524 nm.

4. REAGENTS

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

4.1 *Anhydrous sodium carbonate.*

4.2 *Boric acid* (H_3BO_3) or

4.2.1 *Boron trioxide* (B_2O_3).

4.3 *Sulphuric acid,* approximately 8 N solution.

Carefully pour 225 ml of sulphuric acid solution, ρ 1.84 (g/ml), approximately 96 % (m/m), into approximately 500 ml of water, cool and dilute to 1000 ml.

4.4 *Sulphuric acid*, approximately 16 N solution.

Carefully pour 450 ml of sulphuric acid, ρ 1.84 (g/ml), approximately 96 % (m/m) into 500 ml of water, cool and dilute to 1000 ml.

4.5 *Potassium permanganate*, 0.6 g/l solution.

4.6 *Hydrochloric acid*, ρ 1.19 (g/ml), approximately 38 % (m/m) solution.

4.7 *Chloroform*, free from ethanol.

Purify the chloroform, approximately ρ 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.

4.8 *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 (4.7).

4.9 *Vanadium, standard solution* containing 1.000 g/l of V_2O_5.

Weigh, to the nearest 1 mg, 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 5 % (m/m) sodium hydroxide solution.

After dissolving, acidify with 12 ml of the sulphuric acid solution (4.4), transfer quantitatively into a 1000 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.

4.10 *Vanadium, standard solution* containing 0.10 g/l of V_2O_5.

Take 50.0 ml of the standard solution (4.9), 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.

4.11 *Vanadium, standard solution* containing 0.010 g/l of V_2O_5.

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 0.010 mg of V_2O_5.

4.12 *Anhydrous sodium sulphate.*

5. APPARATUS

Ordinary laboratory apparatus and

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

5.2 *Spectrophotometer*,

or

5.3 *Photoelectric absorptiometer*