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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 2071

ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF ZINC

ATOMIC ABSORPTION METHOD

1st EDITION

July 1971

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 2071, *Aluminium oxide primarily used for the production of aluminium – Determination of zinc – Atomic absorption 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 led to the adoption of Draft ISO Recommendation No. 2071, which was circulated to all the ISO Member Bodies for enquiry in July 1970. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Israel	Sweden
Belgium	Italy	Switzerland
Czechoslovakia	Korea, Rep. of	Thailand
France	Netherlands	U.A.R.
Germany	New Zealand	United Kingdom
Greece	Poland	U.S.A.
Hungary	Portugal	U.S.S.R.
India	South Africa, Rep. of	
Iran	Spain	

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 2071 | July 1971

ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF ZINC

ATOMIC ABSORPTION METHOD

1. SCOPE

This ISO Recommendation describes an atomic absorption method for the determination of zinc in aluminium oxide primarily used for the production of aluminium.

2. FIELD OF APPLICATION

The method is applicable to zinc contents, expressed as ZnO, greater than 0.002 %.

3. PRINCIPLE

Dissolution of a test portion by attack with hydrochloric acid under pressure.

Nebulization of the solution in the centre of an acetylene/air flame and determination of the zinc by photometric measurement of the absorption of the 213.8 nm line emitted by a zinc hollow cathode lamp.

NOTE. – The dissolution of the sample can also be effected by alkaline fusion (see Annex).

4. REAGENTS

For the analysis, use only water doubly distilled from a borosilicate glass apparatus with ground joints, or water of equivalent purity. Avoid the use of lead glass.

4.1 *Aluminium oxide,* purity greater than 99.95 %, containing less than 0.001 % ZnO.

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

4.3 *Aluminium,* acid solution (main solution).

Pickle 11 g of extra pure aluminium (99.999 % purity), in the form of shavings obtained by milling or drilling, in a little nitric acid, ρ 1.40 g/ml approximately (about 68 % m/m solution).

Wash the pickled shavings with water and then dry them by washing with acetone. Weigh, to the nearest milligramme, 10.588 g of these dried shavings and place them in a beaker of suitable capacity (for example 500 ml) and add 144 ml of the hydrochloric acid solution (4.2). Add one drop of pure mercury to aid the attack. Wait until the reaction quietens down, then place the beaker on a sand bath and maintain at a gentle heat until all the aluminium has dissolved. Allow to cool, transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

4.4 *Zinc* standard solution, containing 0.100 g/l of ZnO.

Weigh, to the nearest 0.1 mg, 0.100 g of zinc oxide, previously calcined at 1000 °C for 1 hour and cooled in a desiccator. Place this in a beaker of suitable capacity (for example 100 ml) and dissolve in 5.5 ml of the hydrochloric acid solution (4.2). Dilute the solution and transfer it quantitatively to a 1000 ml one-mark volumetric flask. Dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 100 µg of ZnO.

4.5 *Zinc* standard solution, containing 0.020 g/l of ZnO.

Take 100.0 ml of the standard zinc solution (4.4), place it in a 500 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 20 µg of ZnO.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Apparatus* as specified in ISO Recommendation R 2073, *Aluminium oxide primarily used for the production of aluminium – Preparation of sample solution for analysis by means of attack by hydrochloric acid under pressure.*

5.2 *Burette*, graduated in 0.05 ml conforming to ISO Recommendation R 385, *Burettes.*

5.3 *Spectrophotometer*, atomic absorption type, fitted with a burner fed from cylinders of acetylene and compressed air.

5.4 *Zinc hollow cathode lamp.*

NOTE. – All glassware, including reagent flasks, should be of borosilicate glass or glass of another type not releasing zinc, or as an alternative, plastics material. Rubber stoppers should not be used; use exclusively ground glass or plastics stoppers.

6. PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.001 g, 2 g of the sample dried at 300 °C, prepared according to clause 2.3 of ISO Recommendation R 802, *Aluminium oxide primarily used for the production of aluminium – Preparation and storage of test samples.*

6.2 Preparation of the calibration curve

6.2.1 *Preparation of the standard matching solutions.* Into each of a series of six 100 ml one-mark volumetric flasks, place 50 ml of the acid solution of aluminium (4.3) followed respectively by the volumes of standard zinc solution (4.5) shown in the following table, measured with the aid of the burette (5.2).

Standard solution of zinc (4.5)	Corresponding mass of zinc
ml	mg
0*	0
5.0	0.10
10.0	0.20
15.0	0.30
20.0	0.40
25.0	0.50

* Blank test on the reagents used for the preparation of the calibration curve.

Dilute to the mark and mix.