INTERNATIONAL STANDARD 804

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by alkaline fusion

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Mise en solution en vue de l'analyse — Méthode par fusion alcaline

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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 804 and found it technically suitable for transformation. International Standard ISO 804 therefore replaces ISO Recommendation R 804-1968 to which it is technically identical.

ISO Recommendation R 804 was approved by the Member Bodies of the following countries :

Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Ireland	Spain
Bulgaria	Israel	Sweden
Canada	Italy	Switzerland
Chile	Korea, Rep. of	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.

The Member Body of the following country disapproved the transformation of ISO/R 804 into an International Standard :

Egypt, Arab Rep. of

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by alkaline fusion

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the dissolution, by alkaline fusion, of aluminium oxide primarily used for the production of aluminium, in order to obtain a principal solution (solution P) for certain determinations.

2 REFERENCES

ISO 802, *Aluminium oxide primarily used for the production of aluminium — Preparation and storage of test samples.*

ISO 2927, *Aluminium oxide primarily used for the production of aluminium — Sampling.*

3 PRINCIPLE

Alkaline fusion of a test protion

- either with a mixture of sodium carbonate and boric acid,
- or with a mixture of sodium carbonate and sodium tetraborate.

Dissolution of the melt in an excess of nitric acid so that the final pH of the solution is approximately 1, after dilution to 500 ml, or approximately 0,4, after dilution to 250 ml.

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).

4.3 Sodium tetraborate, anhydrous ($Na_2B_4O_7$).

4.4 Aluminium oxide, extra pure.

4.5 Nitric acid, approximately 8 N solution.

Dilute 540 ml of nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution, with water and dilute to 1 000 ml.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum dish, flat bottomed, of diameter approximately 70 mm and depth approximately 35 mm, fitted with a platinum lid.

5.2 Electric furnace, capable of being controlled at 500 ± 50 °C.

5.3 Electric furnace, capable of being controlled between 1 000 and 1 025 °C.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,001 g, exactly 5 g of the test sample, dried at 300 °C (see 3.3 of ISO 802).

6.2 Fusion of test portion

Weigh into the platinum dish (5.1)

- either 12 g of the sodium carbonate (4.1) and 4 g of the boric acid (4.2),
- or 10,3 g of the sodium carbonate (4.1) and 3,3 g of the sodium tetraborate (4.3).

Mix thoroughly. Add the test portion (6.1) and carefully mix the whole, preferably with a platinum spatula. Cover the dish with its lid and place it in the electric furnace (5.2), controlled at 500 ± 50 °C, taking care to isolate it from the floor of the furnace by means of a support that cannot cause introduction of impurities. Maintain at 500 ± 50 °C until the reaction subsides. Then transfer the dish to the electric furnace (5.3), controlled between 1 000 and 1 025 °C, taking care to isolate it, as before, from the floor of the furnace.

Keep the dish in the furnace for 30 min. Ensure that a temperature of between 1 000 and 1 025 °C is maintained for a minimun of 20 min.

6.3 Preparation of the principal solution

Remove the dish from the furnace and allow to cool in air. Add boiling water to the dish, heating gently until dissolution of the melt.

After slight cooling, transfer the contents of the dish to a polyethylene beaker of suitable capacity containing 50 ml of the nitric acid solution (4.5).

NOTE — In the cases where dissolution of the melt in nitric acid prior to obtaining the principal solution is unsuitable for the determination of certain elements, the dissolution should be made in an alternative appropriate acid which should be stated in the method of test of the element.

Dissolve any residue still adhering to the walls of the dish (consisting essentially of iron(III) oxide, calcium oxide and titanium oxide) with 20 ml of the nitric acid solution and transfer the solution obtained to the polyethylene beaker. Carefully wash both the dish and the lid with hot water and transfer the washings to the polyethylene beaker.

By careful washing, transfer the contents of the polyethylene beaker to a glass beaker. Heat for a few minutes at a temperature close to the boiling point until any remaining aluminium hydroxide is completely dissolved. Allow to cool slightly. When the solution is lukewarm, transfer quantitatively to a 250 or 500 ml one-mark volumetric flask, depending on the content of the elements to be determined. Allow to cool, dilute to the mark and mix.

NOTE — When the solution obtained is opalescent, prepare a new solution, taking care to grind the aluminium oxide so as to obtain a particle size smaller than approximately 50 μm.

6.4 Preparation of the blank test solution

Prepare a blank test solution as indicated in 6.4.1 or 6.4.2, proceeding, in accordance with the instructions given in the International Standard relating to the determination to be carried out, in the presence or absence of extra-pure aluminium oxide (4.4).

6.4.1 *Blank test solution containing extra-pure aluminium oxide*

Follow the same procedure as used for the test portion (see 6.2 and 6.3), using exactly 5 g of extra-pure aluminium oxide (4.4), weighed to the nearest 0,001 g.

6.4.2 *Blank test solution free from extra-pure aluminium oxide*

Weigh into the same platinum dish (5.1)

— either 12 g of the sodium carbonate (4.1) and 4 g of the boric acid (4.2),

— or 10,3 g of the sodium carbonate (4.1) and 3,3 g of the sodium tetraborate (4.3).

Mix carefully. Cover the dish with its lid and place in the electric furnace (5.2), controlled at 500 ± 50 °C, taking care to isolate the dish from the floor of the furnace. Maintain at 500 ± 50 °C until the reaction subsides. Then transfer the dish to the electric furnace (5.3), controlled at a temperature of between 1 000 and 1 025 °C, taking care to isolate it, as before, from the floor of the furnace. Keep the dish in the furnace for a maximum of 5 min.

Remove the dish from the furnace and allow to cool in air. Add boiling water to the dish, heating gently until dissolution of the melt.

After slight cooling, transfer the contents of the dish to a polyethylene beaker of suitable capacity containing 30 ml of the nitric acid solution (4.5). Wash carefully both the dish and the lid with hot water and transfer the washings to the polyethylene beaker. Transfer carefully, by washing, the contents of the polyethylene beaker to a glass beaker. Heat for a few minutes at a temperature close to the boiling point. Allow to cool slightly and transfer quantitatively to either a 250 or a 500 ml one-mark volumetric flask, according to requirements.

Place 36,7 ml of the nitric acid solution (4.5) in the platinum dish and evaporate almost to dryness. Add a small amount of hot water to the dish. Add 3,3 ml of the nitric acid solution. Heat if necessary, and after cooling, transfer the solution quantitatively to the one-mark volumetric flask to which the contents of the glass beaker have previously been transferred. Allow to cool, dilute to the mark and mix.

NOTE — This technique of partial evaporation is used to ensure the same pH values, both in the blank test and in the principal solution, in the presence of the same quantities of impurities introduced.

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.