INTERNATIONAL STANDARD ISO 2073

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Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by hydrochloric acid attack under pressure

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Mise en solution en vue de l'analyse — Méthode par attaque à l'acide chlorhydrique sous pression

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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 2073 and found it technically suitable for transformation. International Standard ISO 2073 therefore replaces ISO Recommendation R 2073-1971 to which it is technically identical.

ISO Recommendation R 2073 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 2073 into an International Standard :

Romania

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by hydrochloric acid attack under pressure

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the dissolution of aluminium oxide primarily used for the production of aluminium, by means of attack by hydrochloric acid under pressure in a sealed borosilicate glass tube, in order to obtain a test solution for certain determinations.

The method is not applicable to the preparation of test solutions for the determination of silicon, sodium or boron, owing to the possibility of extraction of these elements from the glass.

NOTE — Hydrochloric acid may be replaced by another appropriate acid. In this case, the acid should be stated in the method of test of the impurity.

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

Attack of a test portion by hydrochloric acid under pressure in a sealed borosilicate glass tube heated in an electric oven controlled at 250 or 275 °C.

4 REAGENT

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

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Borosilicate glass tubing, having thick walls (of thickness about 2,4 mm) and an external diameter of about 16 mm.

5.2 Borosilicate glass rod, of diameter about 4 mm.

5.3 Glass-blowing equipment, comprising :

5.3.1 Gas burner, fed by a mixture of combustible gas and oxygen, with suitable heads.

5.3.2 Normal combustible gas and oxygen.

5.3.3 Glass cutter.

5.3.4 Protective goggles, tinted.

5.3.5 Blower, with rubber tube connected to gas burner.

5.4 Pencil, resistant to high temperatures, for marking glass.

5.5 Tube holder, to keep tubes vertical, or alternatively

5.5.1 Tube holder, to keep tubes at an inclination of about 30°.

5.6 Funnel, of diameter approximately 40 mm, with short stem.

5.7 Electric oven, capable of being controlled at 250 ± 5 °C and 275 ± 5 °C.

5.8 Protecting steel tubes, of internal diameter about 25 mm and length about 260 or 310 mm, according to the length of the glass tube (5.1) used, threaded and firmly fitted with screw caps at each end. A number of holes of diameter about 6 to 8 mm shall be made at random intervals along the length of the protective tube. This tube is necessary in order to avoid damage to the interior of the oven and to adjacent tubes in case a reaction tube should burst. The holes are necessary to allow the escape of gases in the event of breakage of a reaction tube and to avoid a rise of pressure inside the protective tube.

6 PROCEDURE

6.1 Test portion

According to the determination to be carried out, weigh, to the nearest 0,001 g, 1 or 2 g of the test sample, dried at 300 °C (see 3.3 of ISO 802).

6.2 Attack of the test portion

6.2.1 *Preparation of the tubes*

Cut the tubing (5.1) to a length of about 250 mm for a 1 g test portion, or to a length of about 300 mm for a 2 g test portion.

Clean the tubes in a cleaning solution, rinse thoroughly in distilled water, dry at approximately 125 °C and allow to cool.

Close one end of each tube in the flame so as to give it a hemispherical shape. Avoid an increase or reduction in the thickness of the tube and anneal to eliminate stresses. For this annealing, use a reducing flame (a yellow flame, obtained by reducing the flow of oxygen, without adjusting the flow of gas) until a thick coating of carbon black is deposited on the tube, and allow it to cool. A series of tubes can also be annealed by placing them in a furnace at approximately 550 °C and allowing the temperature of the furnace to drop slowly to approximately 300 °C.

Mark the prepared tubes with the pencil (5.4). Transfer the test portion (6.1) to a tube with the aid of the funnel (5.6).

Add exactly 7,20 ml of the hydrochloric acid solution (4.1) and 2 ml of water for a test portion of 1 g or 14,40 ml of the hydrochloric acid solution and 4 ml of water for a test portion of 2 g. Prepare two or three tubes under the same conditions, sealing them as follows. Using the glass-blowing equipment (5.3), join one end of a glass tube to the opening of the tube by simultaneous heating of the two glass parts until they are soft, in order to form a joint. Allow to cool just sufficiently to obtain a rigid joint.

Heat the top of the tube in the full flame at about 10 mm from the joint (turning the tube continually) until the walls are uniformly soft. Separate the two parts in the flame, with the minimum of pulling. After separation, heat the end of the tube in order to avoid a thick, protuberant joint. Do not overheat, or a slight swelling may be caused by a rise in the internal pressure of the gaseous atmosphere.

Anneal the second closure in a reducing flame until it is coated with carbon black.

6.2.2 *Dissolution of the test portion*

Shake the sealed tubes until the test portion is completely mixed with the acid. Place the sealed tubes in the protecting tubes (5.8) and screw up the caps. Place the assembly, using the tube holder (5.5 or 5.5.1), in the electric oven (5.7), controlled at 250 ± 5 °C, and leave them for about 16 h.

After this period, allow the furnace to cool to about 50 °C, then cautiously remove the tubes and leave them to cool at room temperature.

NOTE — The reaction time varies according to the type of aluminium oxide and its degree of calcination.

For aluminium oxides calcined at high temperature, the reaction temperature can be raised to 275 ± 5 °C but precautions shall then be taken as the increase in pressure resulting from the increase in temperature of the reaction may cause the tubes to burst.

6.2.3 *Opening the tube*

Once the protecting tubes have cooled to room temperature, open them and withdraw the sealed tubes. Carefully wipe the carbon black from the ends of the tubes, clean with cleaning solution, rinse with water and dry.

By means of the glass cutter (5.3.3), trace a line around the upper part of the tube, above the level of the liquid. In order to break the tube, moisten the line with water and touch with the glass rod (5.2) brought to red heat.

6.2.4 *Transfer of the solution*

Transfer the solution quantitatively from the tube into a beaker of suitable capacity (for example 400 ml), with hot water and using a "policeman", if necessary, to detach all salts which may have formed. Warm the solution to dissolve the salts and transfer the solution quantitatively to a one-mark volumetric flask of the capacity specified in the method for the determination of the impurity.

6.3 Preparation of the blank test solution

Prepare a blank test solution, following the same procedure as that specified in 6.2, using the same quantities of all the reagents as used for the dissolution of the test portion and 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.

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.