UDC 661.862.22 : 543.056

Ref. No. : ISO/R 2073-1971 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

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

1st EDITION

July 1971

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The 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,* 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. 2073, 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 2073 | July 1971

ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

PREPARATION OF SAMPLE SOLUTION FOR ANALYSIS BY MEANS OF ATTACK BY HYDROCHLORIC ACID UNDER PRESSURE

1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes 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.

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

2. PRINCIPLE

Attack of the test portion by hydrochloric acid under pressure in a sealed borosilicate glass tube heated in a natural ventilation electric furnace regulated at 250 °C.

3. REAGENT

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

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

4. APPARATUS

Ordinary laboratory apparatus and

4.1 *Borosilicate glass tubing*, having thick walls (about 2.4 mm in thickness) and an external diameter of about 16 mm.

4.2 *Borosilicate glass rod*, about 4 mm in diameter.

4.3 *Glass-blowing equipment,* comprising :

- *gas burner,* fed by a mixture of combustible gas and oxygen, with suitable heads;
- *normal combustible gas* and *oxygen;*
- *glass cutter;*
- *protective goggles,* tinted;
- *blower* with rubber tube connected to gas burner.

4.4 *Pencil,* resistant to high temperatures, for marking glass.

4.5 *Tube holder,* to keep tubes vertical, or alternatively

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

4.6 *Funnel,* 40 mm in diameter, with short stem.

4.7 *Electric furnace,* with natural ventilation, capable of being controlled at 250 ± 5 °C and 275 ± 5 °C.

4.8 *Protecting steel tubes,* of internal diameter about 25 mm and length about 260 or 310 mm, according to the length of the glass tube used, threaded and firmly fitted with screw caps at each end. A number of holes of about 6 to 8 mm diameter should 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 furnace 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.

5. PROCEDURE

5.1 Test portion

According to the determination to be carried out, weigh, to the nearest 0.001 g, 1 or 2 g of the sample, dried at 300 °C (see clause 2.3 of ISO Recommendation R 802, *Aluminium oxide primarily used for the production of aluminium – Preparation and storage of test samples*).

5.2 Attack of the test portion

5.2.1 *Preparation of the tubes.* Cut the tubing (4.1) 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 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 (4.4). Transfer the test portion (5.1) to a tube with the aid of the funnel (4.6).

Add exactly 7.20 ml of the hydrochloric acid solution (3.1) and 2 ml of water for a test portion of 1 g or 14.40 ml of the hydrochloric acid solution (3.1) 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 (4.3), 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.

5.2.2 *Dissolution of the test portion.* Shake the sealed tubes until the sample is completely mixed with the acid. Place the sealed tubes in the protecting tubes (4.8) and screw up the caps. Place the assembly, using the tube holder (4.5) or (4.5.1), in the electric furnace (4.7) controlled at 250 ± 5 °C and leave them for about 16 hours.

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 should then be taken as the increase in pressure resulting from the increase in temperature of the reaction may cause the tubes to burst.

5.2.3 *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 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 (4.2) brought to red heat.

5.2.4 *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.

5.3 Blank test

Carry out a blank test at the same time, using a sealed borosilicate glass tube with the same quantities of reagents as those used for the test. Carry out this test in the presence or absence of pure aluminium oxide according to the instructions given in the method for the determination of the impurity.

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 element.